



Report No. 1067

Bitumen Proficiency Testing Program

Round 9

March 2018

Acknowledgments

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

© Copyright Proficiency Testing Australia, 2018

PO Box 7507 SILVERWATER NSW 2128, Australia

CONTENTS

1.	FOREWORD	1
2.	FEATURES OF THE PROGRAM	1
3.	FORMAT OF THE APPENDICES.....	2
4.	STATISTICAL DESIGN OF THE PROGRAM	2
5.	PTA AND TECHNICAL ADVISER'S COMMENTS.....	5
6.	OUTLIER RESULTS.....	12
7.	REFERENCE	12

APPENDIX A – Results and Data Analysis

Viscosity at 135.0°C.....	A2
Density at 15.0°C (Bottle).....	A3
Penetration at 25.0°C , 100g, 5s.....	A5
Softening Point.....	A6
Viscosity at 60.0°C.....	A8
Viscosity at 60.0°C after RTFO Treatment.....	A9
Viscosity at 60.0°C as % of Original After RTFO Treatment.....	A12

APPENDIX B – Homogeneity and Stability Testing

Homogeneity Testing.....	B1
Stability Testing.....	B2

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 ninth round of an ongoing series of programs.

The program was conducted in January 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 Emilia Cincu and the Technical Adviser was Dr Bill Chik, SAMI Bitumen Technologies Pty Ltd. This report was authorised by Mrs K. Cividin, Quality Manager - Senior Scientific Officer.

2. FEATURES OF THE PROGRAM

- (a) Participants were provided with two 700ml samples of bitumen Class 240 labelled PTA Sample A and PTA Sample B.
- (b) A total of 21 laboratories received samples, comprising:
 - 18 Australian participants; and
 - 3 overseas participants, including:
 - New Zealand;
 - Singapore; and
 - Trinidad and Tobago.

All participating laboratories submitted the 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 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 12.

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;
 - ordered z-score charts; and
- (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 no. 12 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).

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

$$\sqrt{\frac{\pi}{2}} \times \frac{\text{normIQR}}{\sqrt{n}} \quad n = \text{number of results}$$

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 tables summarise the results submitted by participants for the program.

TABLE A1: SUMMARY STATISTICS

Test	No. of Results	Median	Normalised IQR
Viscosity at 135.0°C (0.001 Pa.s)	22	0.3890	0.0133
Density at 15.0°C (bottle) (0.1 Kg/m ³)	16	1044.10	1.63
Penetration 25.0°C, 100g, 5s (0.1 mm)	25	64.00	3.04
Softening Point, (0.2°C)	23	48.00	0.74
Viscosity at 60.0°C (0.1 Pa.s)	21	198.30	3.41

TABLE A2: SUMMARY STATISTICS when an AS oven was used

Test	No. of Results	Median	Normalised IQR
Viscosity at 60.0°C After RTFO Treatment (0.1 Pa.s)	13	334.50	11.12
Viscosity at 60.0°C as % of Original After RTFO Treatment	13	169.00	4.52

TABLE A3: SUMMARY STATISTICS when an ASTM oven was used

Test	No. of Results	Median	Normalised IQR
Viscosity at 60.0°C After RTFO Treatment (0.1 Pa.s)	6	358.00	24.91
Viscosity at 60.0°C as % of Original After RTFO Treatment	6	187.00	14.57

5. PTA AND TECHNICAL ADVISER'S COMMENTS

All laboratories submitted their results on time for inclusion in the final report. The overall performance (considering the measurement uncertainty) meets our expectations.

In general, there are more statistical outliers in Round 9 when compared to previous PT rounds. From a total of 145 results, 16 outliers were identified, representing 11%. A total of 9 laboratories have been identified as having reported one or more outlier results, as depicted in Table D. All laboratories with outliers or an absolute z-score between 2.0 and 3.0 are encouraged to review their procedures.

Results were separated into two groups for analysis of Viscosity at 60.0°C after RTFO treatment and Viscosity at 60.0°C as percentage of original after RTFO treatment, according to the oven which was used during testing. The type of oven can influence the results, due to a major difference in their technical design: the ASTM RTFO-type of oven has a big fan on top of the oven, while the Australian design RTFO-type of oven does not. We encourage our participants to report their results from both types of ovens if available. It might be beneficial for Australian laboratories to be equipped with both types of ovens.

Variation exists between laboratories and often within the same laboratory when two technicians analyse the same sample. In general, people can make mistakes in laboratory testing, but by participating in a Proficiency Testing Program, these mistakes can hopefully be identified and corrected.

A possible source of error in the determination of viscosity at 60°C is the application of a capillary viscometer tube that is not clean enough. A possible error in density measurement is that the water in the 25°C bath is unclean. The water in the 25°C bath should be kept clean at all times.

During the program, a participant requested additional information regarding the class of bitumen. After evaluating the necessity of this information, the class of bitumen was made available to all participants. This information will be made available in the future rounds.

We will continue to provide two test samples each time so that two technicians from the same laboratory can participate in the program.

The following table gives a comparison of the robust CVs and percentage of outliers for tests common to previous programs. Comparison of robust CVs and percentage of outliers with the previous rounds demonstrates a higher number of outliers for this round, especially for Viscosity at 135.0°C, Density at 15°C, Viscosity at 60.0°C after RTFO treatment and Viscosity at 60.0°C as percentage of original after RTFO treatment.

TABLE B: COMPARISON OF ROBUST CVs AND PERCENTAGE OF OUTLIERS

Test	Round 7		Round 8		Round 9	
	CV	% Outliers	CV	% Outliers	CV	% Outliers
Viscosity at 135.0°C (0.001 Pa.s)	4.7%	-	4.0%	-	3.4%	9.1%
Density at 15.0°C (Bottle) (0.1 Kg/m ³)	0.1%	-	0.1%	17.7%	0.2%	25%
Penetration 25.0°C, 100g, 5s (0.1 mm)	5.1%	5.6%	2.7%	8.7%	4.7%	8%
Softening Point (0.2°C)	3.5%	5.6%	0.9%	20.0%	1.5%	8.7%
Viscosity at 60.0°C (0.1 Pa.s)	3.0%	-	2.6%	15.8%	1.7%	13%
Viscosity at 60.0°C After RTFO Treatment (0.1 Pa.s)	AS: 7.6% ASTM: 5.1%	-	6.3%	-	AS: 3.3% ASTM: 7.0%	AS: 7.7% ASTM: -
Viscosity at 60.0°C as % of Original After RTFO Treatment	AS: 4.5% ASTM: 7.0%	-	5.0%	5.9%*	AS: 2.7% ASTM: 7.8%	AS: 15.4% ASTM: -

Notes:

“-“ indicates that no outliers were detected

*please refer to PTA and Technical Adviser's comments on page 5 of Report No.1004

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 from this round were provided by SAMI Bitumen Technologies, Camellia Sydney. Samples came from their bitumen storage tank and were prepared using AS2008. Certified reference materials used included 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. Results for Viscosity at 135°C, Density, Penetration, Softening point and Viscosity at 60°C each returned at least 11 sets of results from the same method group, therefore results generated using each of these methods have been pooled for analysis. For Viscosity at 60°C after RTFO, and Viscosity at 60°C as percentage of original after RTFO, the test method is a combination of two methods, namely AS 2341.2 and AS 2341.10. However, some participants just wrote either one of these two methods in the results sheet. As a result, the statistical analysis by method groups could not be performed.

TABLE C1: VISCOSITY AT 135°C (METHOD AS 2341.4)

Laboratory Code	Result	MU (\pm)	Z-Score	
2	0.403	#	0.25	
3	0.439	0.0003	3.19	§
13	0.4	#	0.00	
21	0.397	#	-0.25	
28A	0.392	#	-0.65	
28B	0.394	#	-0.49	
29	0.405	0.01	0.41	
30A	0.428	#	2.29	?
30B	0.425	#	2.04	?
31	0.4	0.02	0.00	
32A	0.3791	#	-1.71	
32B	0.386	#	-1.14	

No. of Results	12
Median	0.4000
Norm IQR	0.0122
Uncertainty (Median)	0.0044
Robust CV	3.1%
Minimum	0.379
Maximum	0.439
Range	0.060

Notes:

"#" indicates no response was provided by the laboratory

"?" indicates an absolute z-score greater than 2.0 but less than 3.0, i.e. $2.0 < |z\text{-score}| < 3.0$

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

TABLE C2: DENSITY AT 15.0°C (METHOD AS 2341.7)

Laboratory Code	Result	MU (\pm)	Z-Score	
2	1039	#	-2.70	?
13	1044.6	#	0.13	
14	1043.4	#	-0.48	
19	1043.9	#	-0.23	
22	1043.9	#	-0.23	
23	1046.5	0.4	1.08	
24	1044.5	#	0.08	
25	1052.7	#	4.21	§
28B	1055.1	#	5.42	§
29	1043.4	1	-0.48	
32A	1049.8	#	2.75	?
32B	1045.3	#	0.48	
34	1043.3	#	-0.53	
35	1044.2	#	-0.08	

No. of Results	14
Median	1044.35
Norm IQR	1.98
Uncertainty (Median)	0.66
Robust CV	0.2%
Minimum	1039.0
Maximum	1055.1
Range	16.1

Notes:

"#" indicates no response was provided by the laboratory

"?" indicates an absolute z-score greater than 2.0 but less than 3.0, i.e. $2.0 < |z\text{-score}| < 3.0$

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

TABLE C3: PENETRATION AT 25.0°C (METHOD AS 2341.12)

Laboratory Code	Result	MU (\pm)	Z-Score	
2	64	#	0.30	
3	56.3	2.6	-2.00	
8A	62	#	-0.30	
8B	61	#	-0.60	
8C	62	#	-0.30	
13	65	#	0.60	
14	62	#	-0.30	
19	65	#	0.60	
22	67	#	1.19	
23	61	1	-0.60	
24	67	#	1.19	
25	65	#	0.60	
28A	60	#	-0.89	
28B	61	#	-0.60	
29	62	2	-0.30	
30A	57.6	#	-1.61	
30B	61.9	#	-0.33	
31	66	3.3	0.89	
32A	75	#	3.58	§
32B	76	#	3.88	§
34	71	#	2.38	?
35	65	#	0.60	

No. of Results	22
Median	63.00
Norm IQR	3.35
Uncertainty (Median)	0.90
Robust CV	5.3%
Minimum	56.3
Maximum	76.0
Range	19.7

Notes:

"#" indicates no response was provided by the laboratory

"?" indicates an absolute z-score greater than 2.0 but less than 3.0, i.e. $2.0 < |z\text{-score}| < 3.0$

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

TABLE C4: SOFTENING POINT (METHOD AS 2341.18 and AGPT/T131*)

Laboratory Code	Result	MU (\pm)	Z-Score		Method
2	48.6	#	0.69		AS2341.18
3	50.2	0.5	2.53	?	AS2341.18
8A	47.5	#	-0.57		AS2341.18
8B	48.5	#	0.57		AS2341.18
8C	48	#	0.00		AS2341.18
13	48.2	#	0.23		AS2341.18
14	49	#	1.15		AS2341.18
21	45.5	#	-2.87	?	AS2341.18
22	47.8	#	-0.23		AS2341.18
24	48	#	0.00		AS2341.18
25	46.8	#	-1.38		AS2341.18, AGPT/T131
28A	48.5	#	0.57		AS2341.18
28B	47.5	#	-0.57		AS2341.18
29	46.5	2	-1.72		AS2341.18
30A	48.6	#	0.69		AS2341.18
30B	48	#	0.00		AS2341.18
31	48	2.4	0.00		AS2341.18
32A	45	#	-3.44	§	AGPT/T131
32B	46.6	#	-1.61		AGPT/T131
35	48.2	#	0.23		AS2341.18

No. of Results	20
Median	48.00
Norm IQR	0.87
Uncertainty (Median)	0.24
Robust CV	1.8%
Minimum	45.0
Maximum	50.2
Range	5.2

Notes:

"#" indicates no response was provided by the laboratory

"?" indicates an absolute z-score greater than 2.0 but less than 3.0, i.e. $2.0 < |z\text{-score}| < 3.0$

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

"**" AGPT/T131 is based on AS 2341.18

TABLE C5: VISCOSITY AT 60°C (METHOD AS 2341.2)

Laboratory Code	Result	MU (\pm)	Z-Score	
2	198.3	#	0.91	
3	188.2	#	0.86	
8A	203	#	0.93	
8B	206	#	0.95	
8C	200	#	0.92	
13	197	#	0.90	
14	201	#	0.92	
19	194	#	0.89	
22	194.7	#	0.89	
23	197.6	1.4	0.90	
24	201	#	0.92	
28A	198	#	0.91	
28B	200	#	0.92	
29	210	12	0.97	
32A	188	#	0.85	
32B	196.2	#	0.90	
34	200.6	#	0.92	
35	199.4	#	0.91	

No. of Results	18
Median	198.85
Norm IQR	3.34
Uncertainty (Median)	0.99
Robust CV	1.7%
Minimum	188.0
Maximum	210
Range	22.0

Notes:

"#" indicates no response was provided by the laboratory

All other tests returning results from the same method group were less than 11; therefore reliable conclusions cannot be drawn from the analysing grouped methods on this occasion. For these tests, 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	Laboratory Code No.
Viscosity at 135.0°C (0.001 Pa.s)	3, 38
Density at 15.0°C (Bottle) (0.1 Kg/m ³)	2, 25, 28B, 32A
Penetration 25.0°C, 100g, 5s (0.1 mm)	32A, 32B
Softening Point (0.2°C)	21, 32A
Viscosity at 60.0°C (0.1 Pa.s)	29, 32A, 38
Viscosity at 60.0°C after RTFO Treatment (0.1 Pa.s)	3
Viscosity at 60.0°C as % of original after RTFO Treatment	3, 32A

7. REFERENCE

- [1] *Guide to Proficiency Testing Australia*, 2016 (This document can be found on the PTA website, www.pta.asn.au)

APPENDIX A

Results and Data Analysis

Viscosity at 135.0°C	A2
Density at 15.0°C (Bottle)	A3
Penetration 25.0°C, 100g, 5s	A5
Softening Point	A6
Viscosity at 60.0°C	A8
Viscosity at 60.0°C after RTFO Treatment.....	A9
Viscosity at 60.0°C as % of original after RFTO Treatment.....	A12

Viscosity at 135.0°C (0.001 Pa.s)					
Laboratory Code	Result	MU (±)	Z-Score		Method
2	0.403	#	1.05		AS2341.4
3	0.439	0.0003	3.75	§	AS2341.4
9	0.385	#	-0.30		ASTM D2171
13	0.4	#	0.82		AS2341.4
14	0.382	#	-0.52		AS2341.2
19	0.382	#	-0.52		AS2341.2
21	0.397	#	0.60		AS2341.4
22	0.37	#	-1.42		AGPT/T111
23	0.387	0.005	-0.15		AS2341.3
24	0.367	#	-1.65		AS/NZS2341.2
25	0.391	#	0.15		AGPT/T111
28A	0.392	#	0.22		AS2341.4
28B	0.394	#	0.37		AS2341.4
29	0.405	0.01	1.20		AS2341.4
30A	0.428	#	2.92	?	AS2341.4
30B	0.425	#	2.70	?	AS2341.4
31	0.4	0.02	0.82		AS2341.4
32A	0.3791	#	-0.74		AS2341.4
32B	0.386	#	-0.22		AS2341.4
34	0.38	#	-0.67		AS2341.3
35	0.386	#	-0.22		AS2341.2
38	0.319	13.8	-5.25	§	ASTM D4402/D4402M-15

No. of Results	22
Median	0.3890
Norm IQR	0.0133
Uncertainty (Median)	0.0036
Robust CV	3.4%
Minimum	0.319
Maximum	0.439
Range	0.120

Notes:

"#" indicates no response was provided by the laboratory

"?" indicates an absolute z-score greater than 2.0 but less than 3.0, i.e. $2.0 < |z\text{-score}| < 3.0$

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

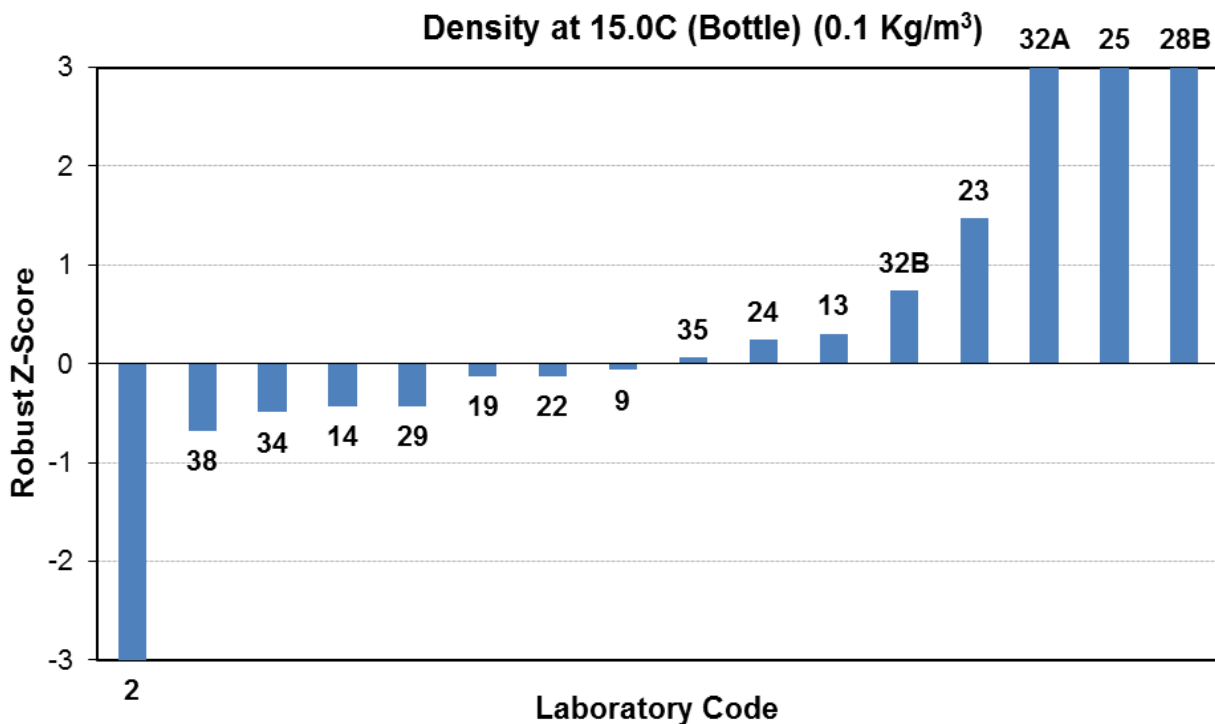
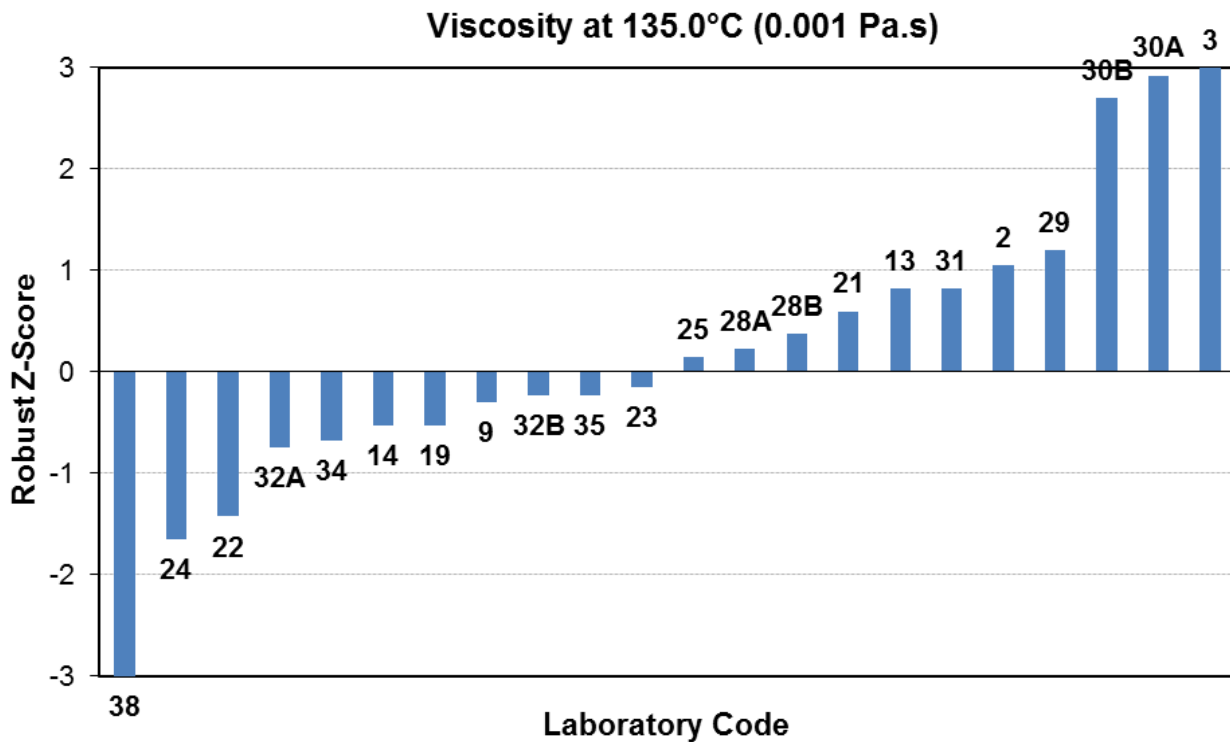
Density at 15.0°C (Bottle) (0.1 Kg/m ³)					
Laboratory Code	Result	MU (±)	Z-Score		Method
2	1039	#	-3.13	§	AS2341.7
9	1044	#	-0.06		ASTM D70
13	1044.6	#	0.31		AS2341.7
14	1043.4	#	-0.43		AS2341.7
19	1043.9	#	-0.12		AS2341.7
22	1043.9	#	-0.12		AS2341.7
23	1046.5	0.4	1.47		AS2341.7
24	1044.5	#	0.25		AS2341.7
25	1052.7	#	5.27	§	AS2341.7
28B	1055.1	#	6.74	§	AS2341.7
29	1043.4	1	-0.43		AS2341.7
32A	1049.8	#	3.50	§	AS2341.7/ASTM D4311
32B	1045.3	#	0.74		AS2341.7
34	1043.3	#	-0.49		AS2341.07
35	1044.2	#	0.06		AS2341.7
38	1043	#	-0.67		ASTM D70-2009

No. of Results	16
Median	1044.10
Norm IQR	1.63
Uncertainty (Median)	0.51
Robust CV	0.2%
Minimum	1039.0
Maximum	1055.1
Range	16.1

Notes:

"#" indicates no response was provided by the laboratory

"§" indicates an outlier, i.e. |z-score| ≥ 3.0



Penetration at 25.0°C, 100g, 5s (0.1 mm)					
Laboratory Code	Result	MU (±)	Z-Score		Method
2	64	#	0.00		AS2341.12
3	56.3	2.6	-2.53	?	AS2341.12
8A	62	#	-0.66		AS2341.12
8B	61	#	-0.99		AS2341.12
8C	62	#	-0.66		AS2341.12
9	65	#	0.33		ASTM D5
13	65	#	0.33		AS2341.12
14	62	#	-0.66		AS2341.12
16	63.2	1.2	-0.26		ASTM D5-13
19	65	#	0.33		AS2341.12
22	67	#	0.99		AS2341.12
23	61	1	-0.99		AS2341.12
24	67	#	0.99		AS2341.12
25	65	#	0.33		AS2341.12
28A	60	#	-1.32		AS2341.12
28B	61	#	-0.99		AS2341.12
29	62	2	-0.66		AS2341.12
30A	57.6	#	-2.11	?	AS2341.12
30B	61.9	#	-0.69		AS2341.12
31	66	3.3	0.66		AS2341.12
32A	75	#	3.62	§	AS2341.12
32B	76	#	3.95	§	AS2341.12
34	71	#	2.30	?	AS2341.12
35	65	#	0.33		AS2341.12
38	69	0.9	1.65		ASTM D5/D5M-13

No. of Results	25
Median	64.00
Norm IQR	3.04
Uncertainty (Median)	0.76
Robust CV	4.7%
Minimum	56.3
Maximum	76.0
Range	19.7

Notes:

"#" indicates no response was provided by the laboratory
 "?" indicates an absolute z-score greater than 2.0 but less than 3.0, i.e. $2.0 < |z\text{-score}| < 3.0$

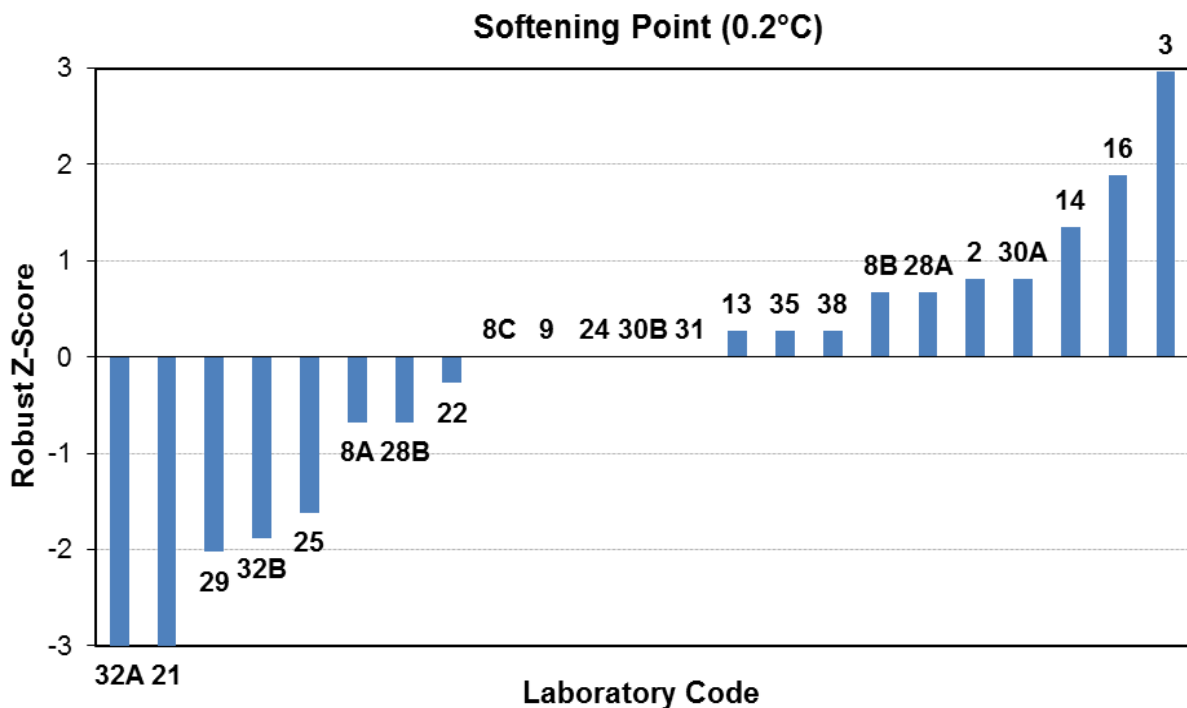
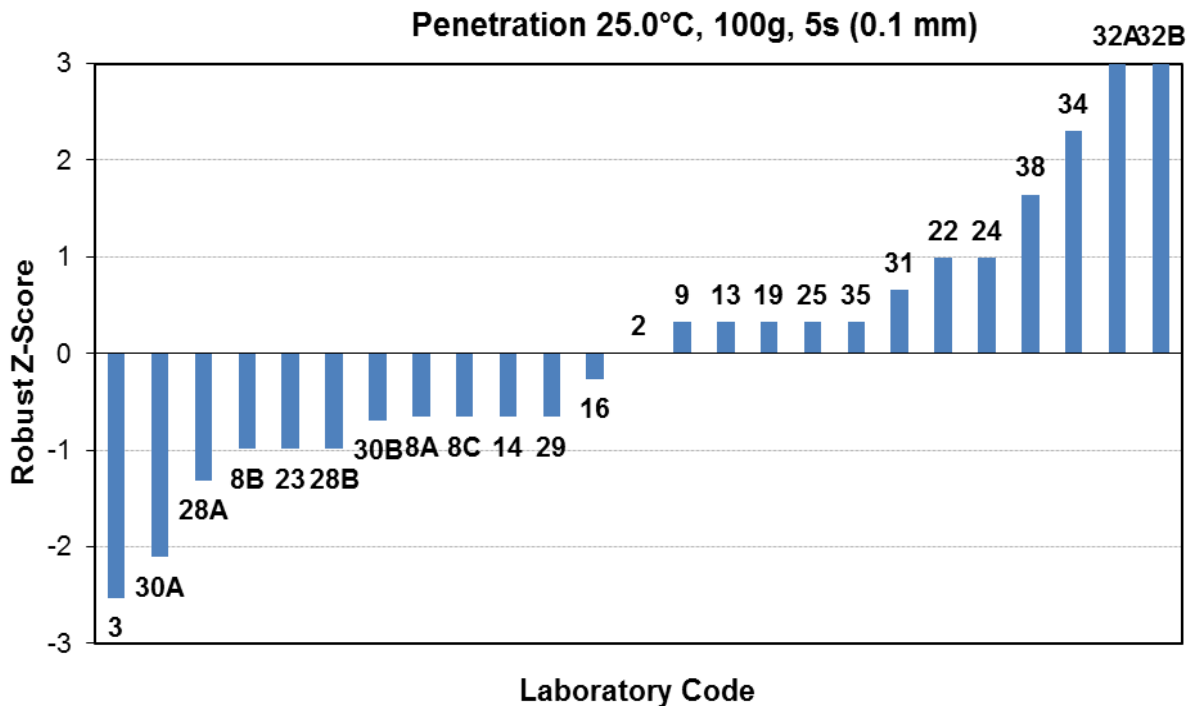
"§" indicates an outlier, i.e. $|z\text{-score}| \geq 3.0$

Softening point (0.2°C)					
Laboratory Code	Result	MU (±)	Z-Score		Method
2	48.6	#	0.81		AS2341.18
3	50.2	0.5	2.97	?	AS2341.18
8A	47.5	#	-0.67		AS2341.18
8B	48.5	#	0.67		AS2341.18
8C	48	#	0.00		AS2341.18
9	48	#	0.00		ASTM D36
13	48.2	#	0.27		AS2341.18
14	49	#	1.35		AS2341.18
16	49.4	0.8	1.89		ASTM D36-14
21	45.5	#	-3.37	§	AS2341.18
22	47.8	#	-0.27		AS2341.18
24	48	#	0.00		AS2341.18
25	46.8	#	-1.62		AS2341.18, AGPT/T131
28A	48.5	#	0.67		AS2341.18
28B	47.5	#	-0.67		AS2341.18
29	46.5	2	-2.02	?	AS2341.18
30A	48.6	#	0.81		AS2341.18
30B	48	#	0.00		AS2341.18
31	48	2.4	0.00		AS2341.18
32A	45	#	-4.05	§	AGPT/T131
32B	46.6	#	-1.89		AGPT/T131
35	48.2	#	0.27		AS2341.18
38	48.2	0.9	0.27		ASTM D36/D36M-12

No. of Results	23
Median	48.00
Norm IQR	0.74
Uncertainty (Median)	0.19
Robust CV	1.5%
Minimum	45.0
Maximum	50.2
Range	5.2

Notes:

"#" indicates no response was provided by the laboratory
 "?" indicates an absolute z-score greater than 2.0 but less than 3.0, i.e. $2.0 < |z\text{-score}| < 3.0$
 "§" indicates an outlier, i.e. $|z\text{-score}| \geq 3.0$



Viscosity at 60.0°C (0.1 Pa.s)					
Laboratory Code	Result	MU (±)	Z-Score		Method
2	198.3	#	0.00		AS2341.2
3	188.2	#	-2.96	?	AS2341.2
8A	203	#	1.38		AS2341.2
8B	206	#	2.26	?	AS2341.2
8C	200	#	0.50		AS2341.2
9	196	#	-0.67		ASTM D2171
13	197	#	-0.38		AS2341.2
14	201	#	0.79		AS2341.2
19	194	#	-1.26		AS2341.2
22	194.7	#	-1.06		AS2341.2
23	197.6	1.4	-0.21		AS2341.2
24	201	#	0.79		AS/NZS2341.2
28A	198	#	-0.09		AS2341.2
28B	200.0	#	0.50		AS2341.2.2015
29	210	12	3.43	§	AS2341.2
31	199	10	0.21		AS2341.4
32A	188	#	-3.02	§	AS2341.2
32B	196.2	#	-0.62		AS2341.2
34	200.6	#	0.67		AS2341.2
35	199.4	#	0.32		AS2341.2
38	184	13.8	-4.19	§	ASTM D4402/D4402M-15

No. of Results	21
Median	198.30
Norm IQR	3.41
Uncertainty (Median)	0.93
Robust CV	1.7%
Minimum	184.0
Maximum	210.0
Range	26.0

Notes:

"#" indicates no response was provided by the laboratory
 "?" indicates an absolute z-score greater than 2.0 but less than 3.0, i.e. $2.0 < |z\text{-score}| < 3.0$

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

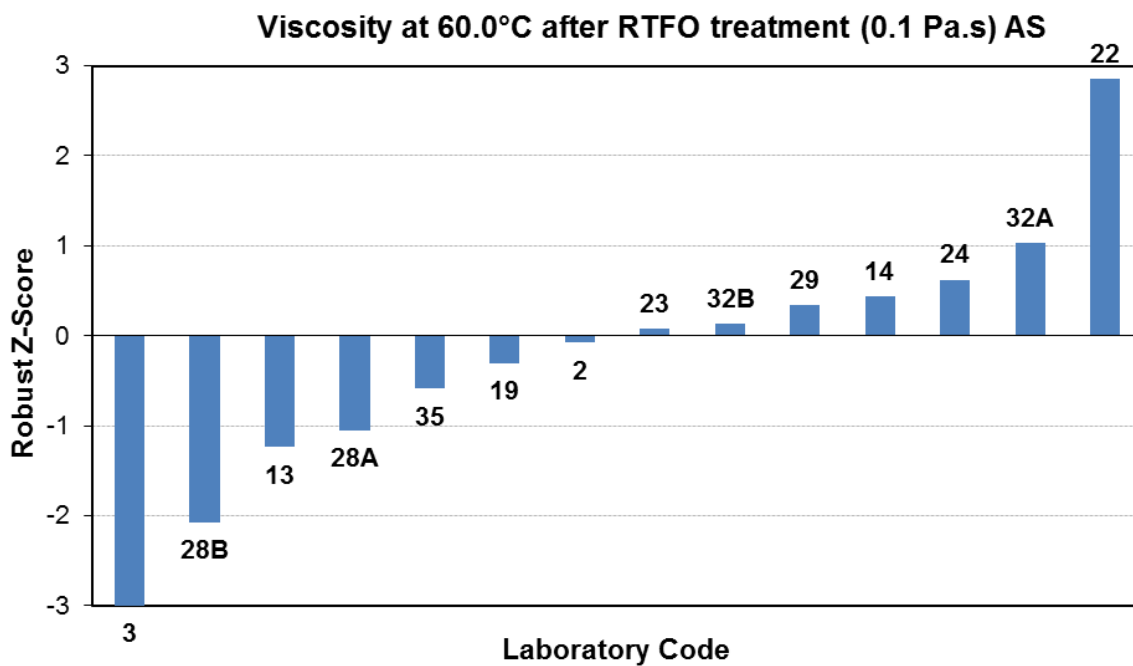
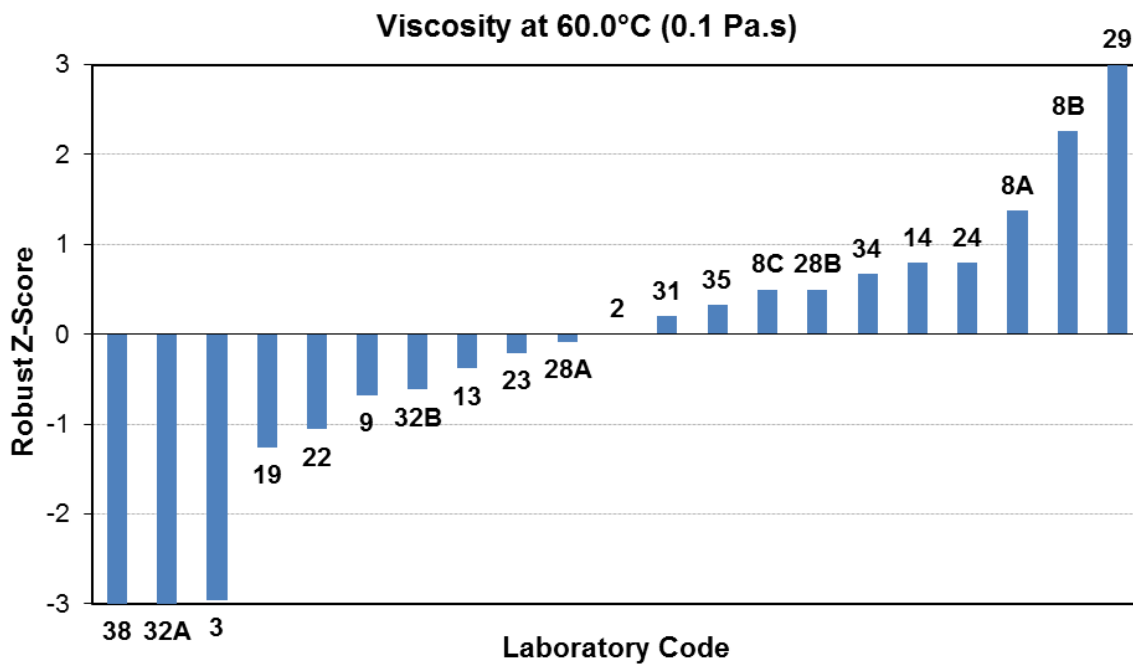
Viscosity at 60.0°C After RTFO Treatment (0.1 Pa.s) AS Oven					
Laboratory Code	Result	MU (±)	Z-Score		Method
2	334.5	#	0.00		AS2341.2, AS2341.10
3	279.9	#	-4.91	§	AS2341.2, AS2341.10
9	#	#			D2872/D2171
13	322	#	-1.12		AS2341.10
14	340	#	0.49		AS2341.2
19	332	#	-0.22		AS 2341.2
22	#	#			AS2341.2
23	336.1	2.1	0.14		AS2341.2, AS2341.10
24	342	#	0.67		AS2341.2
28A	324	#	-0.94		AS2341.2, AS2341.10
28B	313.0	#	-1.93		AS2341.2, AS2341.10
29	339	8	0.40		AS2341.10
31	#	#			AS2341.10
32A	346.4	#	1.07		AS2341.2
32B	336.7	#	0.20		AS2341.2
35	329	#	-0.49		AS2341.2
38	#	#			ASTM D4402/D4402M-15

No. of Results	13
Median	334.50
Norm IQR	11.12
Uncertainty (Median)	3.87
Robust CV	3.3%
Minimum	279.9
Maximum	346.4
Range	66.5

Notes:

"#" indicates no response was provided by the laboratory

"§" indicates an outlier, i.e. |z-score| ≥ 3.0

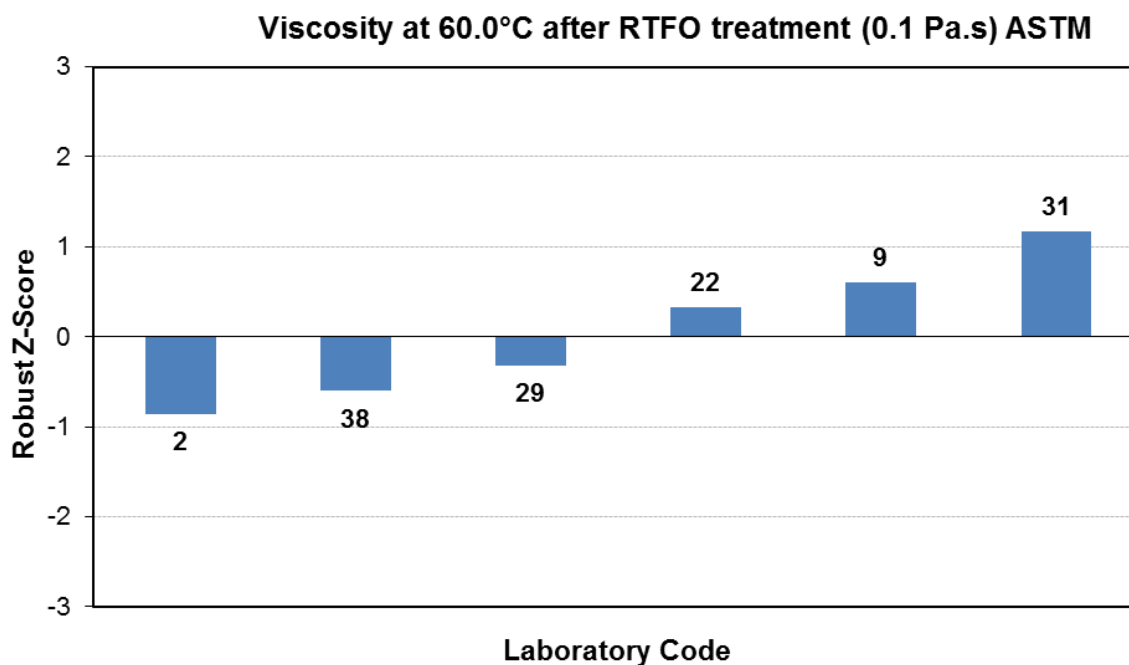


Viscosity at 60.0°C After RTFO Treatment (0.1 Pa.s) ASTM Oven					
Laboratory Code	Result	MU (±)	Z-Score		Method
2	336.7	#	-0.86		AS2341.2, AS2341.10
9	373	#	0.60		D2872/D2171
22	366	#	0.32		AS2341.2
29	350	8	-0.32		AS2341.10
31	387	19.4	1.16		AS2341.10
38	343	13.8	-0.60		ASTM D4402/D4402M-15

No. of Results	6
Median	358.00
Norm IQR	24.91
Uncertainty (Median)	12.74
Robust CV	7.0%
Minimum	336.7
Maximum	387.0
Range	50.3

Notes:

"#" indicates no response was provided by the laboratory



Viscosity at 60.0°C as % of Original After RTFO Treatment AS Oven					
Laboratory Code	Result	MU (±)	Z-Score		Method
2	169	#	0.00		AS2341.2, AS2341.10
3	148.9	#	-4.44	§	AS2341.2, AS2341.10
13	164	#	-1.11		AS2341.10
14	169	#	0.00		AS2341.10
19	171	#	0.44		AS2341.10
23	170.1	1.1	0.24		AS2341.2, AS2341.10
24	170	#	0.22		AS2341.10
28A	164	#	-1.11		AS2341.2, AS2341.10
28B	157	#	-2.65	?	AS2341.2, AS2341.10
29	161	3	-1.77		AS2341.10
32A	184	#	3.32	§	AS2341.2
32B	172	#	0.66		AS2341.2
35	165	#	-0.88		AS2341.10

No. of Results	13
Median	169.00
Norm IQR	4.52
Uncertainty (Median)	1.57
Robust CV	2.7%
Minimum	148.9
Maximum	184.0
Range	35.1

Notes:

"#" indicates no response was provided by the laboratory

"?" indicates an absolute z-score greater than 2.0 but less than 3.0, i.e. $2.0 < |z\text{-score}| < 3.0$

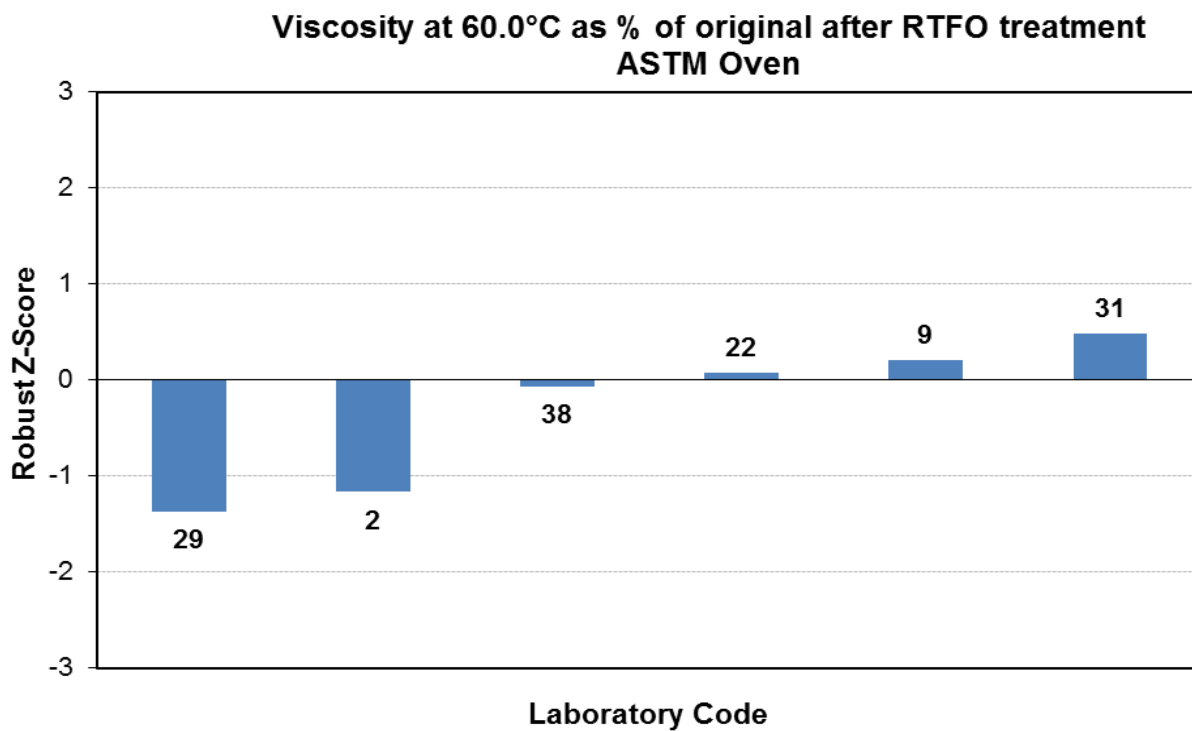
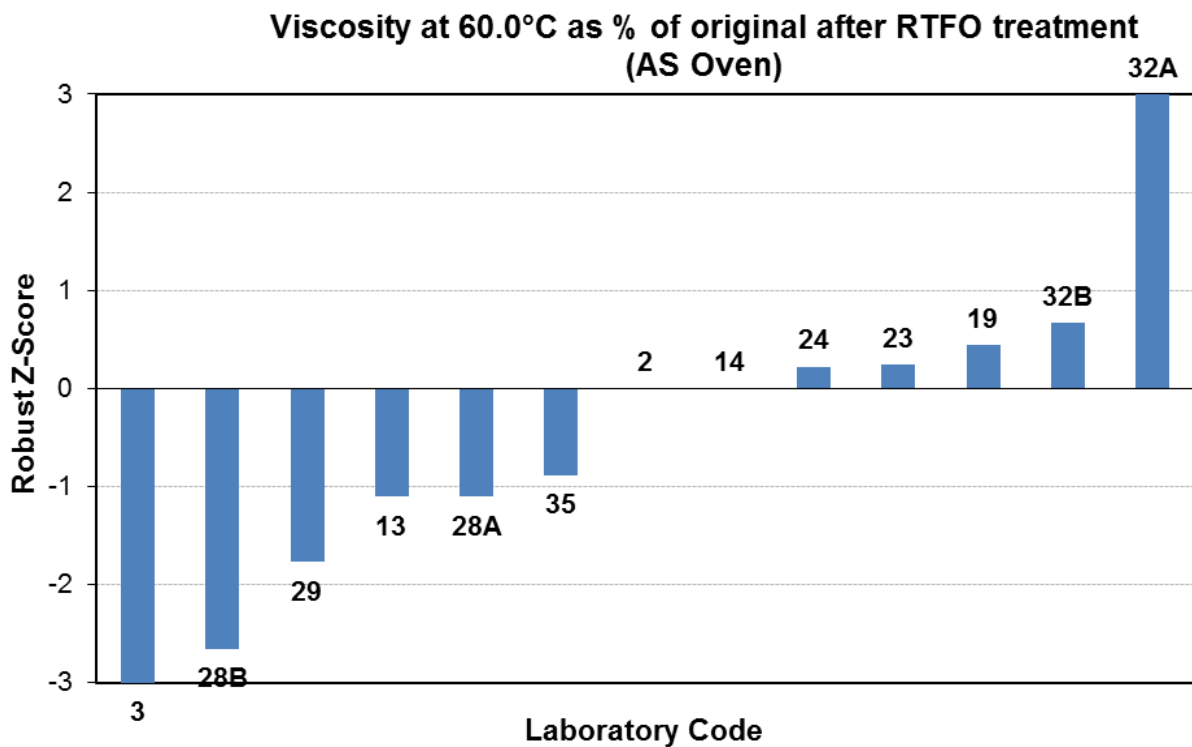
"§" indicates an outlier, i.e. $|z\text{-score}| \geq 3.0$

Viscosity at 60.0°C as % of Original After RTFO Treatment ASTM Oven					
Laboratory Code	Result	MU (±)	Z-Score		Method
2	170	#	-1.17		AS2341.2, AS2341.10
9	190	#	0.21		Calculation
22	188	#	0.07		ASTM D2872
29	167	3	-1.37		AS2341.10
31	194	9.7	0.48		#
38	186	#	-0.07		#

No. of Results	6
Median	187.00
Norm IQR	14.57
Uncertainty (Median)	7.45
Robust CV	7.8%
Minimum	167.0
Maximum	194.0
Range	27.0

Notes:

"#" indicates no response was provided by the laboratory



Additional Information							
Laboratory Code	Measured RTFOT temperature (0.1°C)		Make and Model of Rotational Viscometer	Mean Shear Rates for All Viscosities Tested		Filling Method in Determination of Density at 15°C	Cannon-Manning or Asphalt Institute Viscometer Used?
	AS	ASTM		135°C	60°C		
2	#	#	Brookfield LVDV-II+	77.4	1.10	Partial	Asphalt Institute
3	163.0	#	Brookfield LVDV-I Prime	204 (D 60 rpm, SF 0.34, Torque 87%)	#	#	Cannon-Manning AI:100
8A	#	#	#	#	#	#	Asphalt Institute
8B	#	#	#	#	#	#	Asphalt Institute
8C	#	#	#	#	#	#	Asphalt Institute
9	#	163.2	#	#	#	#	#
13	163.0	#	RVDV-II+PX	93.0 sec-1	1.28 sec-1	Partial	Asphalt Institute
14	163.0	#	#	1065.8	1.30	Partial	Cannon-Manning at 135°C, Asphalt Institute at 60°C
16	#	#	#	#	#	#	#
19	163.0	#	#	#	#	Partial	Cannon-Manning
21	#	#	Brookfield DVII+Pro Extra	20.40 sec-1	#	#	#
22	#	163.0	Brookfield DVII+	126	#	Partial	Asphalt Institute
23	161.0	#	#	#	1.92 sec-1	Partial	Asphalt Institute
24	163.0	#	#	1143.4	2.42, post RTFO 0.984	Partial	Cannon-Manning at 135°C, Asphalt Institute at 60°C
25	#	#	Brookfield RDVD-II+P	#	#	Partial	#
28A	163.0	#	Brookfield RVDV-I+	#	#	#	Asphalt Institute

Additional Information (continuation)							
Laboratory Code	Measured RTFOT temperature (0.1°C)		Make and Model of Rotational Viscometer	Mean Shear Rates for All Viscosities Tested		Filling Method in Determination of Density at 15°C	Cannon-Manning or Asphalt Institute Viscometer Used?
	AS	ASTM		135°C	60°C		
28B	163.0	#	Brookfield RVDV-I+	#	#	Partial	Asphalt Institute
29	163.0	163.0	Brookfield LV	20.40 sec-1	1.3 sec-1	Partial	Asphalt Institute
30A	#	#	DV2TLVTJO	20.4 sec-1	#	#	#
30B	#	#	DV2TLVTJO	20.4 sec-1	#	3	#
31	#	163	Brookfield DVI+DVII	34.0	0.34	#	#
32A	163.0	#	Brookfield DVII+PRO	28.0	8.16	Partial	Cannon-Manning
32B	163.0	#	Brookfield DVII+PRO	28.0	5.05	Partial	Cannon-Manning
34	#	#	#	#	#	Partial	Cannon-Manning
35	163.3	#	#	1099	1.261 (pre), 1.559 (post)	Partial	Asphalt Institute 100 for 60°C, Cannon-Manning CM-8 for 135°C
38	#	163.0	Brookfield V-II+	93	0.82	Partial	No

APPENDIX B

Homogeneity Testing

Homogeneity Testing

Sample no.	Penetration at 25.0°C AS 2341.12		Viscosity at 135.0°C AS 2341.4	
	Results p.u.	Deviation	Results Pa.s.	Deviation
77	65	-0.4637	0.401	-0.09985
11	64	1.0819	0.400	0.14978
57	64	1.0819	0.400	0.14978
16	65	-0.4637	0.399	0.39940
88	65	-0.4637	0.400	0.14978
96	65	-0.4637	0.402	-0.34948
43	65	-0.4637	0.404	-0.84873
104	65	-0.4637	0.401	-0.09985
39	65	-0.4637	0.399	0.39940
67	64	1.0819	0.400	0.14978
Average	64.7		0.4006	

Water bath temperature checked = 25°C
Thermosel temperature checked = 135°C

Notes:

Deviation lies within test method requirements
Sample numbers are randomly chosen by PTA
Samples are homogeneous

Repeatability

for AS 2341.12 <3%
for AS 2341.4 <7%

APPENDIX C

Documentation

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

**PROFICIENCY TESTING AUSTRALIA
BITUMEN PROFICIENCY TESTING PROGRAM
ROUND 9
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 700 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 at 135⁰C
Density at 15C (bottle)
Penetration 25⁰C, 100g, 5s
Softening Point, ⁰C

PTA Sample B:

Viscosity at 60⁰C
Viscosity at 60⁰C after RTFO treatment
Viscosity at 60⁰C as percentage of original after RTFO treatment

3. Participants can perform any or all of the above mentioned tests. Participants are to treat the proficiency testing samples as they would a routine sample tested in the laboratory
4. If performing both 'Sample B' tests it is recommended to perform the Dynamic Viscosity at 60⁰C test first.
5. If possible, participants are encouraged to perform 'Sample B' tests using both AS and ASTM ovens.
6. Please report oven (RTFO) temperature applied during RTFOT. Measure RTFOT temperature using a calibrated thermometer because some ovens have misleading temperature readings.
7. Participants should use the routine test methods which would normally be used to test samples. Please identify the method used on the Results Sheet.
8. 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.
9. The results for all determinations are to be recorded on the results sheet to the accuracy and reporting basis indicated.
10. Testing may commence as soon as the sample is received. All laboratories must return the results sheet no later than **26 January 2018** to:

Emilia Cincu

Proficiency Testing Australia

Phone: 02 9736 8397 Fax: 02 9743 6664 Email: emilia.cincu@pta.asn.au

**PROFICIENCY TESTING AUSTRALIA
BITUMEN PROFICIENCY TESTING PROGRAM
ROUND 9**

RESULTS SHEET

Lab Code

PTA
SAMPLE A

TEST (report to)	Result	MU ±	Method
Viscosity at 135.0°C (0.001 Pa.s)			
Density at 15.0°C (bottle) (0.1 Kg/m ³)			
Penetration 25.0°C, 100g, 5s (0.1 mm)			
Softening Point, (0.2°C)			

PTA
SAMPLE B

TEST (report to)	Result		MU ±	Method
	AS	ASTM		
Viscosity at 60.0°C, (0.1 Pa.s) <input type="checkbox"/>				
Viscosity at 60.0°C after RTFO treatment, (0.1 Pa.s)				
Viscosity at 60.0°C as % of original after RTFO treatment				
Measured RTFOT temperature, (0.1°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: at 135.0°C _____ at 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 **26 January 2018** to:

Emilia Cincu

Fax: 02 9743 6664

Proficiency Testing Australia

Email: emilia.cincu@pta.asn.au

- *End of Report* -