

Report No. 1123

Bitumen Proficiency Testing Program

Round 10

January 2019

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.

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1. FOREWORD

This report summarises the results of a proficiency testing program on the determination of selected chemical tests of bitumen. It constitutes the tenth 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 November 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 Adviser was Dr B Chik, SAMI Bitumen Technologies Pty Ltd. This report was authorised by Mrs K. Cividin, PTA Quality Manager.

2. FEATURES OF THE PROGRAM

- (a) Participants were provided with two 700ml samples labelled PTA Sample A and PTA Sample B containing bitumen C170.
- (b) A total of 30 laboratories received samples, comprising:
 - 23 Australian participants; and
 - 7 overseas participants, including:
 - New Zealand (3);
 - Qatar (1);
 - Serbia (1);
 - Singapore (1); and
 - Trinidad and Tobago (1).

Of these 30 laboratories, two 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 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 9.

3. FORMAT OF THE APPENDICES

- (a) Appendix A contains the analysis of results reported by laboratories for the samples. This section contains the following for each determinant, where appropriate:
 - a table of results and calculated z-scores;
 - a list of summary statistics; and
 - ordered z-score charts.
- (b) Appendix B contains details of the homogeneity testing.
- (c) Appendix C contains copies of the *Instructions to Participants and Results Sheet*.

4. STATISTICAL DESIGN OF THE PROGRAM

- (a) Outlier Results and Z-scores

In order to assess laboratories' testing performance, a robust statistical approach, using z-scores, was utilised. Z-scores give a measure of how far a

result is from the consensus value (i.e. the median), and gives a "score" to each result relative to the other results in the group.

A z-score close to zero indicates that the result agrees well with those from other laboratories, whereas a z-score with an absolute value greater than or equal to 3.0 is considered to be an outlier and is marked by the symbol "§".

The table on page 9 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)	29	0.4091	0.0259
Density at 15.0°C (bottle) (0.1 Kg/m ³)	19	1036.50	1.78
Penetration 25.0°C, 100g, 5s (0.1 mm)	31	71.00	4.82
Softening Point, (0.2°C)	27	47.60	0.52
Viscosity at 60.0°C (0.1 Pa.s)	24	180.00	6.38
Penetration Index	18	-1.0000	0.1964

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)	12	332.70	20.52
Viscosity at 60.0°C as % of Original After RTFO Treatment	12	185.00	11.12

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	359.60	12.43
Viscosity at 60.0°C as % of Original After RTFO Treatment	6	200.00	4.70

5. PTA AND TECHNICAL ADVISER'S COMMENTS

Out of 30 participating laboratories (23 Australian and 7 overseas), two laboratories did not submit results in time to be included in the final report.

Overall performance against prior expectations

A total of six laboratories have been identified as having reported one or more outlier results, as depicted in table D. From a total of 184 results, 14 outliers have been identified, representing 7.6%. In general, there are less statistical outliers in Round 10 when compared to previous PT rounds (8.6% in Round 8 and 11.03% in Round 9). This indicates a good overall performance taking into consideration the measurement uncertainty.

All laboratories with outliers or an absolute z-score between 2.0 and 3.0 are encouraged to review their procedures.

The overall performance of the laboratories was good and generally in line with previous rounds of proficiency testing.

Results analysis

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. Participants are encouraged to report their results from both types of ovens if available. It might also be beneficial for Australian laboratories to be equipped with both types of ovens.

Results for viscosity at 60.0°C were pooled as the type of oven used does not influence the results.

Variations within laboratories and between participants

The performance of different operators in the same laboratory was very similar. For example laboratory code 45 participated in this program with three operators (45A, 45B and 45C). A similar performance was observed across various tests, with outlier results obtained by all three operators for Penetration at 25°C. The values were unusually high, and could be a result of a higher load used for the test (greater than 100 g).

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.

In this round PTA Sample A and PTA Sample B were identical and the Penetration Index should have similar values if calculated based on results from Sample A or Sample B. The median value obtained by the participants was -1.00. This value is in line with the calculated value of -1.10 obtained by the Technical Advisor/Supplier for these samples. The Penetration Index was recalculated based on the submitted results for laboratory codes 49, 78 and 100. The following values were obtained: laboratory code 49: -0.4, laboratory code 78: +0.04 and laboratory code 100: -1.10.

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

TABLE B: COMPARISON OF ROBUST CVs AND PERCENTAGE OF OUTLIERS

Test	Round 8		Round 9		Round 10	
	CV	% Outliers	CV	% Outliers	CV	% Outliers
Viscosity at 135.0°C (0.001 Pa.s)	4.0%	-	3.4%	9.1%	6.3%	3.4%
Density at 15.0°C (Bottle) (0.1 Kg/m ³)	0.1%	17.7%	0.2%	25%	0.2%	5.2%
Penetration 25.0°C, 100g, 5s (0.1 mm)	2.7%	8.7%	4.7%	8%	6.8%	9.6%
Softening Point (0.2°C)	0.9%	20.0%	1.5%	8.7%	1.1%	7.4%
Viscosity at 60.0°C (0.1 Pa.s)	2.6%	15.8%	1.7%	13%	3.5%	8.3%
Viscosity at 60.0°C After RTFO Treatment (0.1 Pa.s)	6.3%	-	AS: 3.3% ASTM: 7.0%	AS: 7.7% ASTM: -	AS: 6.2% ASTM: 3.5%	AS: - ASTM: 16.6%
Viscosity at 60.0°C as % of Original After RTFO Treatment	5.0%	5.9%*	AS: 2.7% ASTM: 7.8%	AS: 15.4% ASTM: -	AS: 6.0% ASTM: 2.3%	AS: - ASTM: 16.6%
Penetration Index	N/A	N/A	N/A	N/A	-19.6%	16.6%
Total % of outliers	8.6%		11.03%		7.6%	

Notes:

“-“ indicates that no outliers were detected

“N/A” indicates not applicable

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 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 130°C, Density at 15°C, Penetration at 25°C, softening Point and Viscosity at 60°C each returned at least 11 sets of results from the same method group. The results are tabulated below.

TABLE C: ANALYSIS BY GROUPED METHODS

Test	Method	No. of Results	Median	Uncertainty of the Median
Viscosity at 135.0°C	AS 2341.4	16	0.4280	0.0100
Density at 15.0°C	AS 2341.7	15	1036.90	0.73
Penetration at 25.0°C	AS 2341.12	25	71.00	1.30
Softening Point	AS 2341.18	20	47.60	0.18
Viscosity at 60.0°C	AS 2341.2	21	180.00	1.32

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	49
Density at 15.0°C (Bottle)	67
Penetration 25.0°C, 100g, 5s	45A, 45B and 45C
Softening Point	78 and 100
Viscosity at 60.0°C	53 and 64
Viscosity at 60.0°C after RTFO Treatment	78
Viscosity at 60.0°C as % of original after RTFO Treatment	64
Penetration Index	49, 78 and 100

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	A1
Density at 15.0°C (Bottle)	A3
Penetration 25.0°C, 100g, 5s	A5
Softening Point	A8
Viscosity at 60.0°C	A10
Viscosity at 60.0°C after RTFO Treatment.....	A12
Viscosity at 60.0°C as % of original after RTFO Treatment.....	A16
Penetration Index.....	A20
Additional Information.....	A22

Viscosity at 135.0°C (0.001 Pa.s)					
Lab Code	Result	MU	Z-Score		Method
1	0.396	#	-0.50		AS 2341.4
5	0.40	#	-0.35		AS 2341.3
6	0.404	#	-0.20		AS 2341.2
9	#	#	na		#
12	0.451	#	1.61		AS 2341.4
21	0.403	#	-0.24		AS 2341.2
24	0.4091	#	0.00		D2170
32	0.428	4%	0.73		AS/NZS 2341.4
34	0.413	#	0.15		SRPS EN 12595:2015
35	0.394	#	-0.58		AS 2341.2
36	0.431	#	0.84		AG:PT/T111
39A	0.371	#	-1.47		AS 2341.4
39B	0.387	#	-0.85		AS 2341.4
45A	0.442	#	1.27		AS 2341.4
45B	0.454	#	1.73		AS 2341.4
45C	0.443	#	1.31		AS 2341.4
49	0.194	#	-8.29	§	AS 2341.4
53	0.41	4%	0.03		AS 2341.4
59A	0.400	#	-0.35		AS 2341.4
59B	0.400	#	-0.35		AS 2341.4
64	0.343	0.01	-2.55	?	ASTM D 4402-2013
67	#	#	na		#
68	0.375	0.01	-1.31		AS 2341.3
71	0.3881	#	-0.81		AS 2341.2
73	0.430	4%	0.81		AS/NZS 2341.4
77	0.396	#	-0.50		ASTM D4402
78	#	#	na		#
95	0.429	#	0.77		AS 2341.4
96	0.436	#	1.04		AG:PT/T111
98	0.428	#	0.73		AS 2341.4
99	0.461	#	2.00		#
100	0.454	#	1.73		AS 2341.4

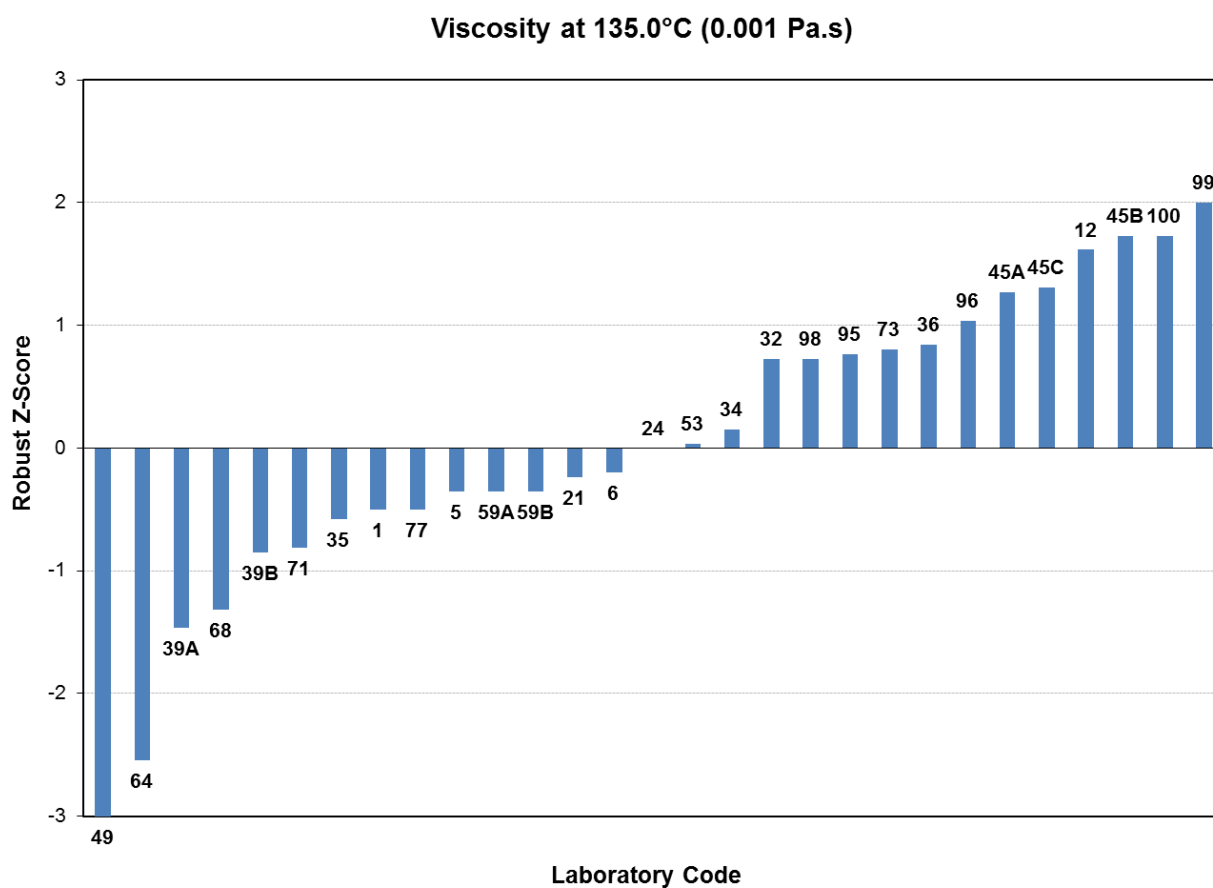
No of Results	29
Median	0.4091
Norm IQR	0.0259
Uncertainty (Median)	0.0060
Robust CV	6.3%
Minimum	0.194
Maximum	0.461
Range	0.267

"?" 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

"#" indicates no response was provided by the laboratory

"na" indicates not applicable



Density at 15.0°C (bottle) (0.1 kg/m ³)				
Lab Code	Result	MU	Z-Score	Method
1	1038.6	#	1.18	AS 2341.7
5	1036.5	#	0.00	AS 2341.07
6	1035.2	#	-0.73	AS 2341.7
9	#	#	na	#
12	#	#	na	#
21	1036.9	#	0.22	AS 2341.7
24	1035.2	#	-0.73	D70
32	1035.1	0.9	-0.79	AS 2341.7
34	#	#	na	#
35	1037.1	#	0.34	AS 2341.7
36	#	#	na	#
39A	1034.1	#	-1.35	AS 2341.7
39B	1038.8	#	1.29	AS 2341.7
45A	#	#	na	#
45B	#	#	na	#
45C	#	#	na	#
49	#	#	na	#
53	#	#	na	#
59A	#	#	na	#
59B	#	#	na	#
64	1034.0	#	-1.41	ASTM D70-2009
67	1029.5	#	-3.93	§ ASTM D70
68	1038.7	0.2	1.24	AS 2341.7
71	1037.1	#	0.34	AS 2341.7
73	1035.6	0.9	-0.51	AS 2341.7
77	#	#	na	#
78	#	#	na	#
95	1035.0	#	-0.84	AS 2341.7
96	1040.5	#	2.25	? AS 2341.7
98	1035.0	#	-0.84	AS 2341.7
99	1037.0	#	0.28	#
100	1037.8	#	0.73	AS 2341.7, ASTM D4311

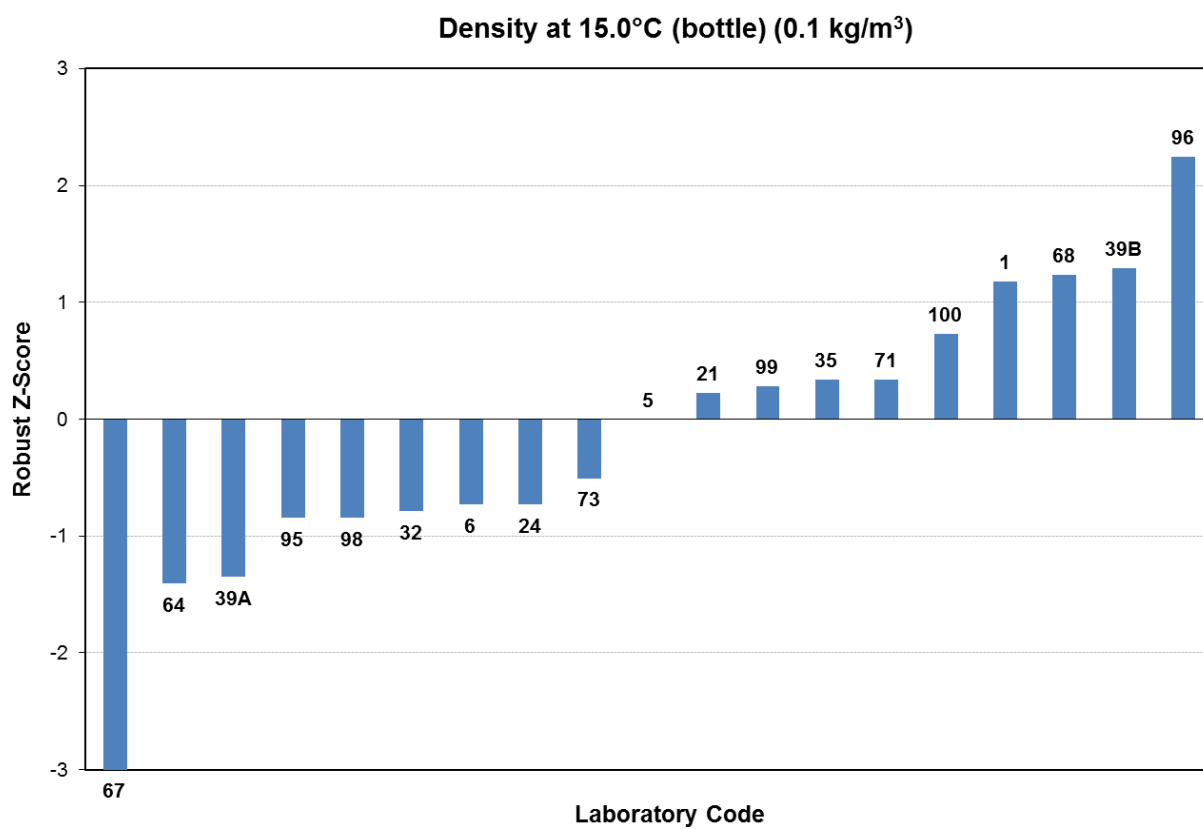
No of Results	19
Median	1036.50
Norm IQR	1.78
Uncertainty (Median)	0.51
Robust CV	0.2%
Minimum	1029.5
Maximum	1040.5
Range	11.0

"?" 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

"#" indicates no response was provided by the laboratory

"na" indicates not applicable



Penetration 25.0°C, 100g, 5s (0.1 mm)					
Lab Code	Result	MU	Z-Score		Method
1	71	#	0.00		AS 2341.12
5	72	#	0.21		AS 2341.12
6	69	#	-0.42		AS 2341.12
9	71.2	1.5	0.04		ASTM D5
12	65.1	#	-1.22		AS 2341.12
21	71.3	#	0.06		AS 2341.12
24	68	#	-0.62		D5
32	68	8%	-0.62		AS 2341.12
34	#	#	na		#
35	75	#	0.83		AS 2341.12
36	65.5	#	-1.14		AS 2341.12
39A	64	#	-1.45		AS 2341.12
39B	65	#	-1.25		AS 2341.12
45A	110.8	#	8.26	§	AS 2341.12
45B	112.6	#	8.63	§	AS 2341.12
45C	111.6	#	8.43	§	AS 2341.12
49	82	#	2.28	?	AS 2341.12
53	68	8%	-0.62		AS 2341.12
59A	65	#	-1.25		AS 2341.12
59B	65	#	-1.25		AS 2341.12
64	75	1.2	0.83		ASTM D5-2012
67	65.3	#	-1.18		ASTM D5
68	71	1	0.00		AS 2341.12
71	70.0	#	-0.21		AS 2341.12
73	70	8%	-0.21		AS 2341.12
77	74	#	0.62		ASTM D5
78	85	#	2.91	?	AS 2341.12
95	70	#	-0.21		AS 2341.12
96	72	#	0.21		AS 2341.12
98	72	#	0.21		AS 2341.12
99	70	#	-0.21		#
100	79	#	1.66		AS 2341.12

No of Results	31
Median	71.00
Norm IQR	4.82
Uncertainty (Median)	1.08
Robust CV	6.8%
Minimum	64.0
Maximum	112.6
Range	48.6

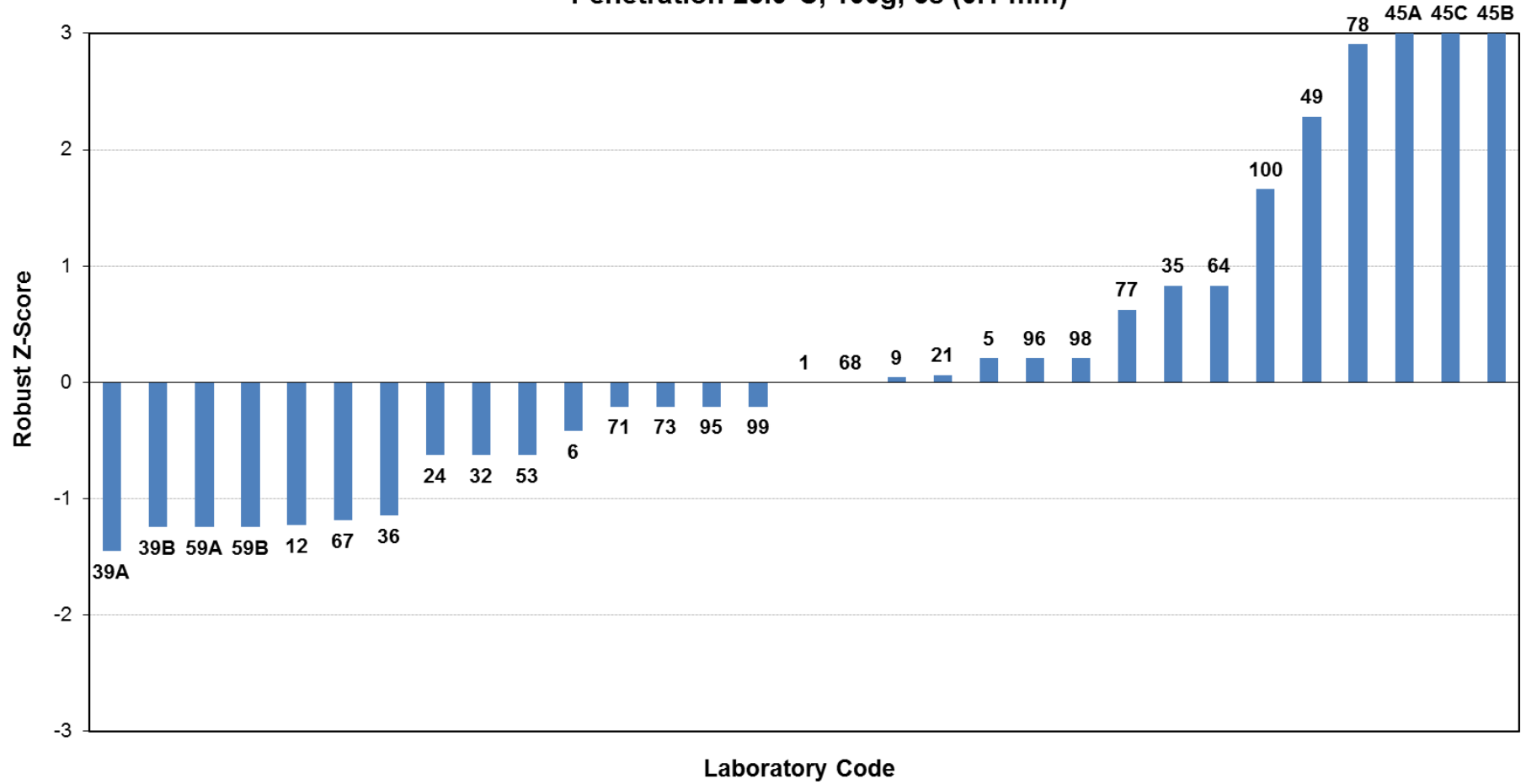
"?" indicates an absolute z-score greater than 2.0 but less than 3.0,
i.e. $2.0 < |z\text{-score}| < 3.0$

"§" indicates an outlier

"#" indicates no response was provided by the laboratory

"na" indicates not applicable

Penetration 25.0°C, 100g, 5s (0.1 mm)



Softening Point (0.2°C)					
Lab Code	Result	MU	Z-Score		Method
1	47.6	#	0.00		AS 2341.18
5	#	#	na		#
6	47.4	#	-0.39		ASTM D36
9	48.8	1.1	2.31	?	ASTM D36
12	47.6	#	0.00		AS 2341.18
21	47.2	#	-0.77		AS 2341.18
24	48.0	#	0.77		D36
32	48.0	2.0	0.77		AS 2341.18
34	#	#	na		#
35	47.0	#	-1.16		AS 2341.18
36	47.6	#	0.00		AG:PT/T131/AS 2341.18
39A	48.0	#	0.77		AS 2341.18
39B	47.5	#	-0.19		AS 2341.18
45A	47.0	#	-1.16		AS 2341.18
45B	48.5	#	1.73		AS 2341.18
45C	48.0	#	0.77		AS 2341.18
49	48.2	#	1.16		AS 2341.18
53	48.0	2.0	0.77		AGPT/T131
59A	48.0	#	0.77		AS 2341.18
59B	47.5	#	-0.19		AS 2341.18
64	47.8	1.4	0.39		ASTM D36-2099
67	46.2	#	-2.70	?	ASTM D36
68	#	#	na		#
71	#	#	na		#
73	47.0	2.0	-1.16		AS 2341.18
77	47.6	#	0.00		ASTM D36
78	49.5	#	3.66	§	AS 2341.18
95	48.0	#	0.77		AS 2341.18
96	46.4	#	-2.31	?	AS 2341.18, AG:PT/T131
98	48.0	#	0.77		AS 2341.18
99	#	#	na		#
100	46.0	#	-3.08	§	AS 2341.18

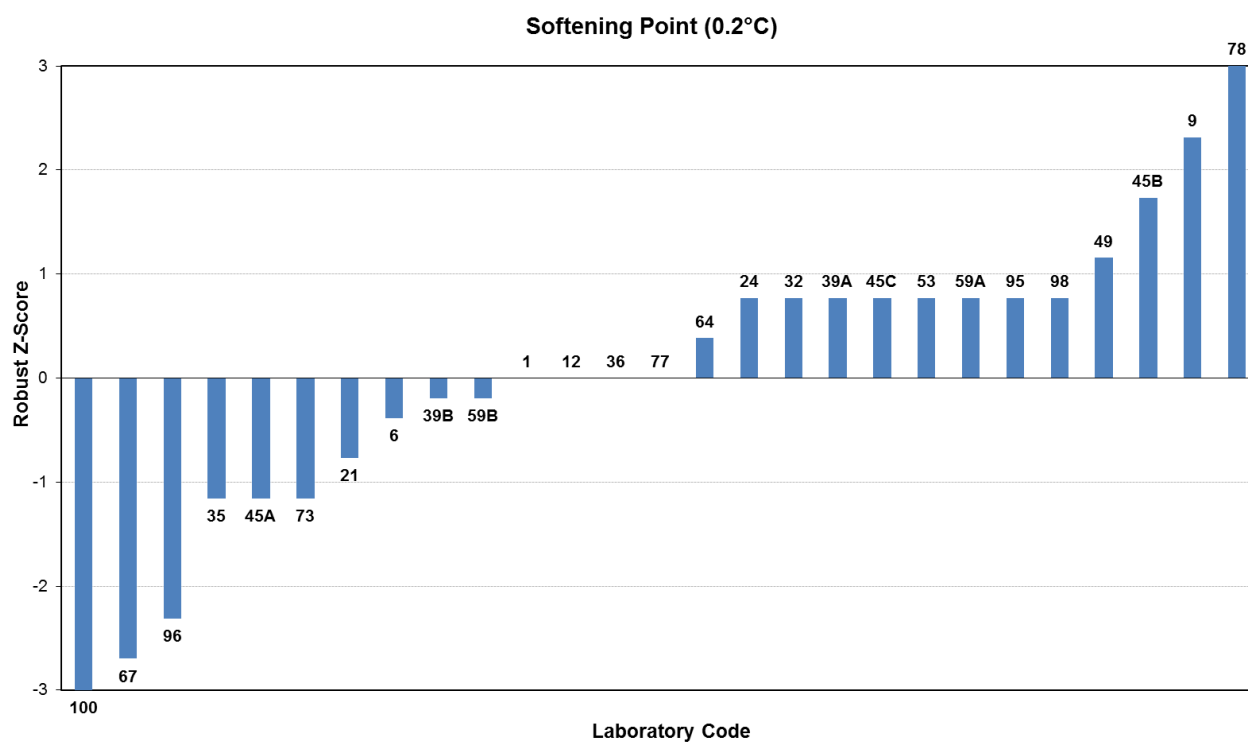
No of Results	27
Median	47.60
Norm IQR	0.52
Uncertainty (Median)	0.13
Robust CV	1.1%
Minimum	46.0
Maximum	49.5
Range	3.5

"?" 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

"#" indicates no response was provided by the laboratory

"na" indicates not applicable



Viscosity at 60.0°C (0.1 Pa.s) AS+ASTM					
Lab Code	Result	MU	Z-Score		Method
1	177.9	#	-0.33		AS 2341.2
5	182.9	#	0.45		AS 2341.2
6	177.9	#	-0.33		AS/NZ 2341.2
9	#	#	na		#
12	#	#	na		#
21	182.6	#	0.41		AS 2341.2
24	#	#	na		#
32	185	7%	0.78		AS/NZS 2341.2
34	#	#	na		#
35	179.5	#	-0.08		AS 2341.2
36	#	#	na		#
39A	180.0	#	0.00		AS 2341.2
39B	180.4	#	0.06		AS 2341.2
45A	184.0	#	0.63		AS 2341.2
45B	184	#	0.63		AS 2341.4
45C	184	#	0.63		AS 2341.2
49	175.7	#	-0.67		AS 2341.2
53	202	7%	3.45	§	AS 2341.2
59A	171.3	#	-1.36		AS 2341.2
59B	173.2	#	-1.07		AS 2341.2
64	154.2	0.012	-4.05	§	ASTM D 4402-2013
67	#	#	na		#
68	180.0	1.6	0.00		AS 2341.2
71	174.5	#	-0.86		AS 2341.2
73	184	7%	0.63		AS/NZS 2341.2
77	#	#	na		#
78	162.3	#	-2.78	?	AS 2341.2
95	184	#	0.63		AS 2341.4
96	#	#	na		#
98	184	#	0.63		AS 2341.2
99	170	#	-1.57		#
100	177.5	#	-0.39		AS 2341.2

No of Results	24
Median	180.00
Norm IQR	6.38
Uncertainty (Median)	1.63
Robust CV	3.5%
Minimum	154.2
Maximum	202.0
Range	47.8

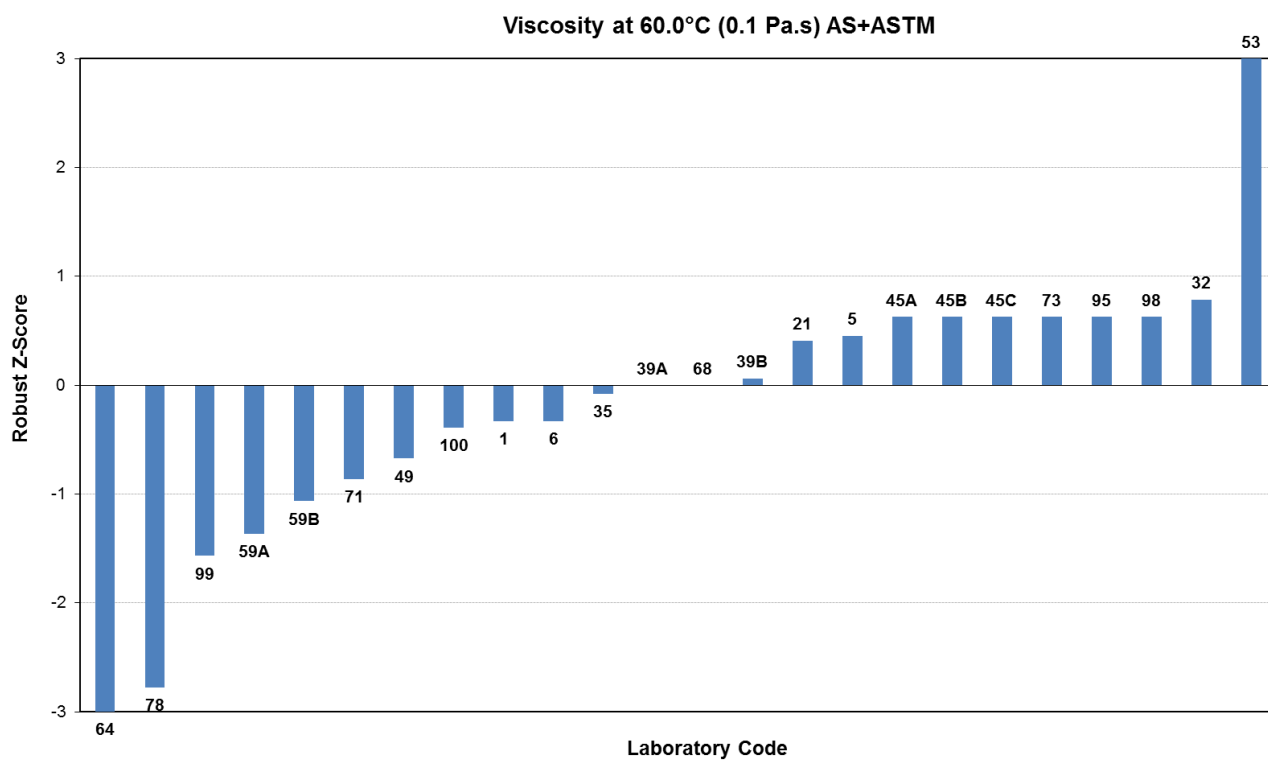
"?" indicates an absolute z-score greater than 2.0 but less than 3.0, i.e.

$2.0 < |z\text{-score}| < 3.0$

"§" indicates an outlier

"#" indicates no response was provided by the laboratory

"na" indicates not applicable



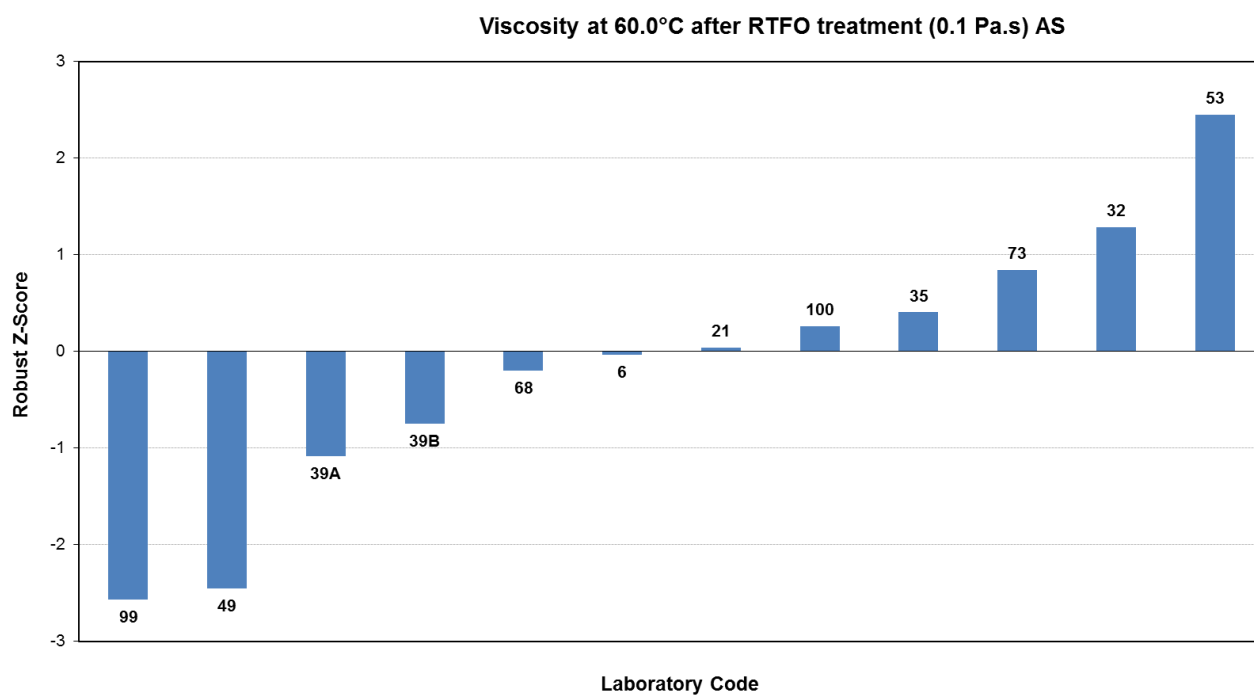
Viscosity at 60.0°C after RTFO treatment (0.1 Pa.s) AS					
Lab Code	Result	MU	Z-Score		Method
1	#	#	na		#
5	#	#	na		#
6	331.9	#	-0.04		AS/NZ 2341.10 / AS/NZ 2341.2
9	#	#	na		#
12	#	#	na		#
21	333.5	#	0.04		AS 2341.2
24	#	#	na		#
32	359	7%	1.28		AS/NZS 2341.10
34	#	#	na		#
35	341.0	#	0.40		AS 2341.2
36	#	#	na		#
39A	310.4	#	-1.09		AS 2341.10
39B	317.3	#	-0.75		AS 2341.2
45A	#	#	na		#
45B	#	#	na		#
45C	#	#	na		#
49	282.4	#	-2.45	?	AS 2341.2 and AS 2341.10
53	383	7%	2.45	?	AS 2341.10 and AS 2341.2
59A	#	#	na		#
59B	#	#	na		#
64	#	#	na		#
67	#	#	na		#
68	328.6	6.3	-0.20		AS 2341.2
71	#	#	na		#
73	350	7%	0.84		AS/NZS 2341.10
77	#	#	na		#
78	#	#	na		#
95	#	#	na		#
96	#	#	na		#
98	#	#	na		#
99	280	#	-2.57	?	#
100	338.1	#	0.26		AS 2341.10, AS 2341.2

No of Results	12
Median	332.70
Norm IQR	20.52
Uncertainty (Median)	7.42
Robust CV	6.2%
Minimum	280.0
Maximum	383.0
Range	103.0

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

"#" indicates no response was provided by the laboratory

"na" indicates not applicable



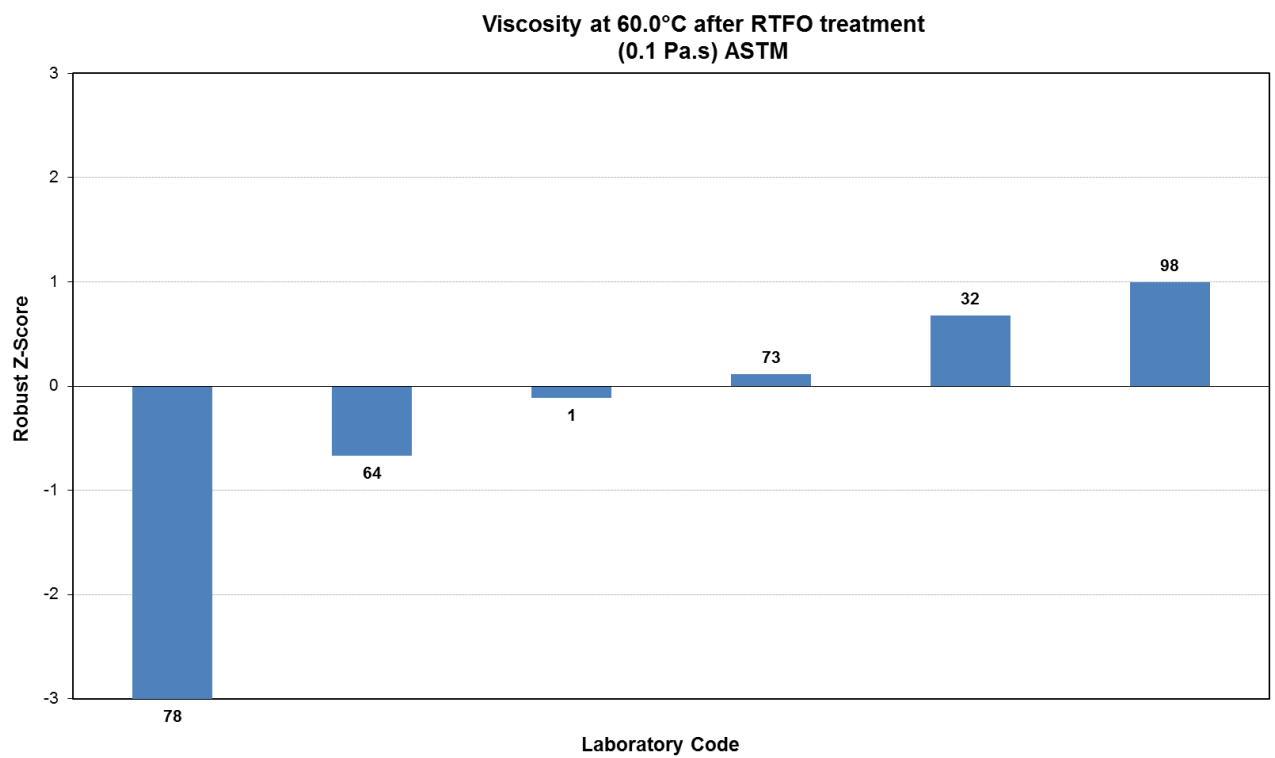
Viscosity at 60.0°C after RTFO treatment (0.1 Pa.s) ASTM					
Lab Code	Result	MU	Z-Score		Method
1	358.2	#	-0.11		AS 2341.2, ASTM D2872-04
5	#	#	na		#
6	#	#	na		#
9	#	#	na		#
12	#	#	na		#
21	#	#	na		#
24	#	#	na		#
32	368	7%	0.68		AS/NZS 2341.10
34	#	#	na		#
35	#	#	na		#
36	#	#	na		#
39A	#	#	na		#
39B	#	#	na		#
45A	#	#	na		#
45B	#	#	na		#
45C	#	#	na		#
49	#	#	na		#
53	#	#	na		#
59A	#	#	na		#
59B	#	#	na		#
64	351.3	0.012	-0.67		ASTM D4402-2013
67	#	#	na		#
68	#	#	na		#
71	#	#	na		#
73	361	7%	0.11		AS/NZS 2341.10
77	#	#	na		#
78	314.6	#	-3.62	§	AS 2341.2
95	#	#	na		#
96	#	#	na		#
98	372	#	1.00		AS 2341.2/.10
99	#	#	na		#
100	#	#	na		#

No of Results	6
Median	359.60
Norm IQR	12.43
Uncertainty (Median)	6.36
Robust CV	3.5%
Minimum	314.6
Maximum	372.0
Range	57.4

"§" indicates an outlier

"#" indicates no response was provided by the laboratory

"na" indicates not applicable



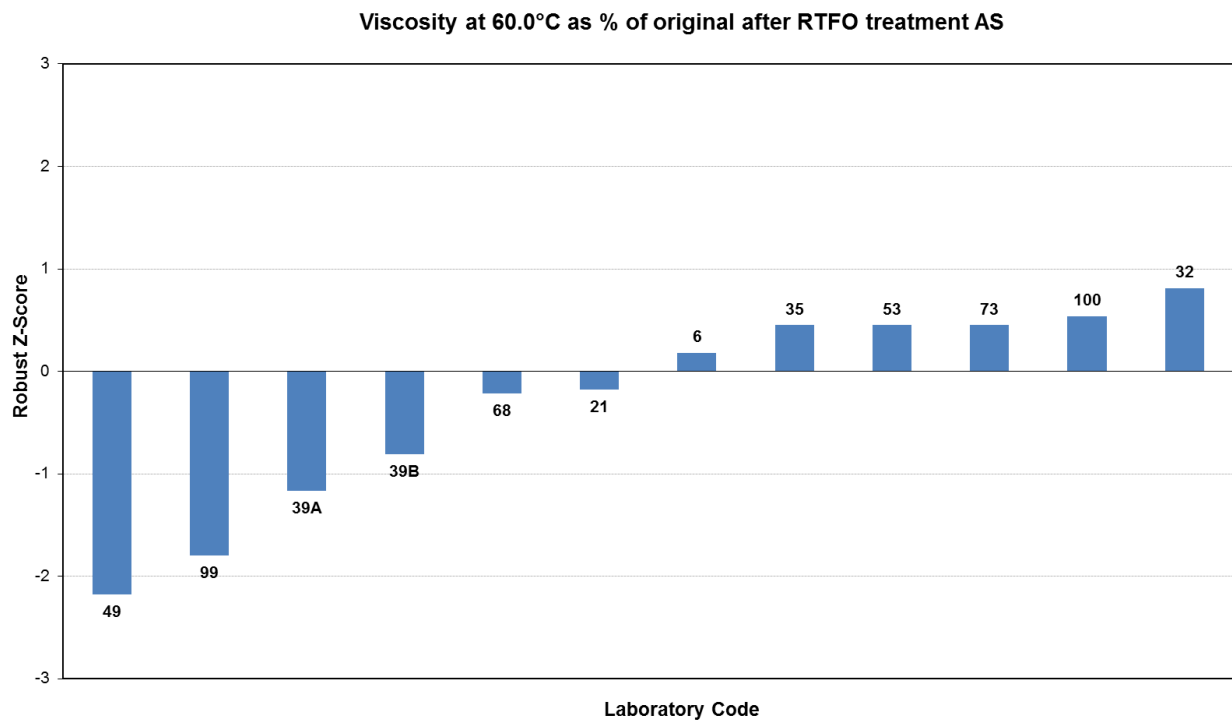
Viscosity at 60.0°C as % of original after RTFO treatment AS				
Lab Code	Result	MU	Z-Score	Method
1	#	#	na	#
5	#	#	na	#
6	187	#	0.18	AS/NZ 2341.10 / AS/NZ 2341.2
9	#	#	na	#
12	#	#	na	#
21	183	#	-0.18	AS 2341.10
24	#	#	na	#
32	194	12%	0.81	AS/NZS 2341.10
34	#	#	na	#
35	190	#	0.45	AS 2341.10
36	#	#	na	#
39A	172	#	-1.17	AS 2341.2 / AS 2341.10
39B	176	#	-0.81	AS 2341.10
45A	#	#	na	#
45B	#	#	na	#
45C	#	#	na	#
49	160.8	#	-2.18	? AS 2341.2 and AS 2341.10
53	190	12%	0.45	AS 2341.10
59A	#	#	na	#
59B	#	#	na	#
64	#	#	na	#
67	#	#	na	#
68	182.6	1.6	-0.22	Calculation
71	#	#	na	#
73	190	12%	0.45	AS/NZS 2341.10
77	#	#	na	#
78	#	#	na	#
95	#	#	na	#
96	#	#	na	#
98	#	#	na	#
99	165	#	-1.80	#
100	191	#	0.54	AS 2341.2

No of Results	12
Median	185.0
Norm IQR	11.1
Uncertainty (Median)	4.0
Robust CV	6.0%
Minimum	161
Maximum	194
Range	33

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

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"na" indicates not applicable

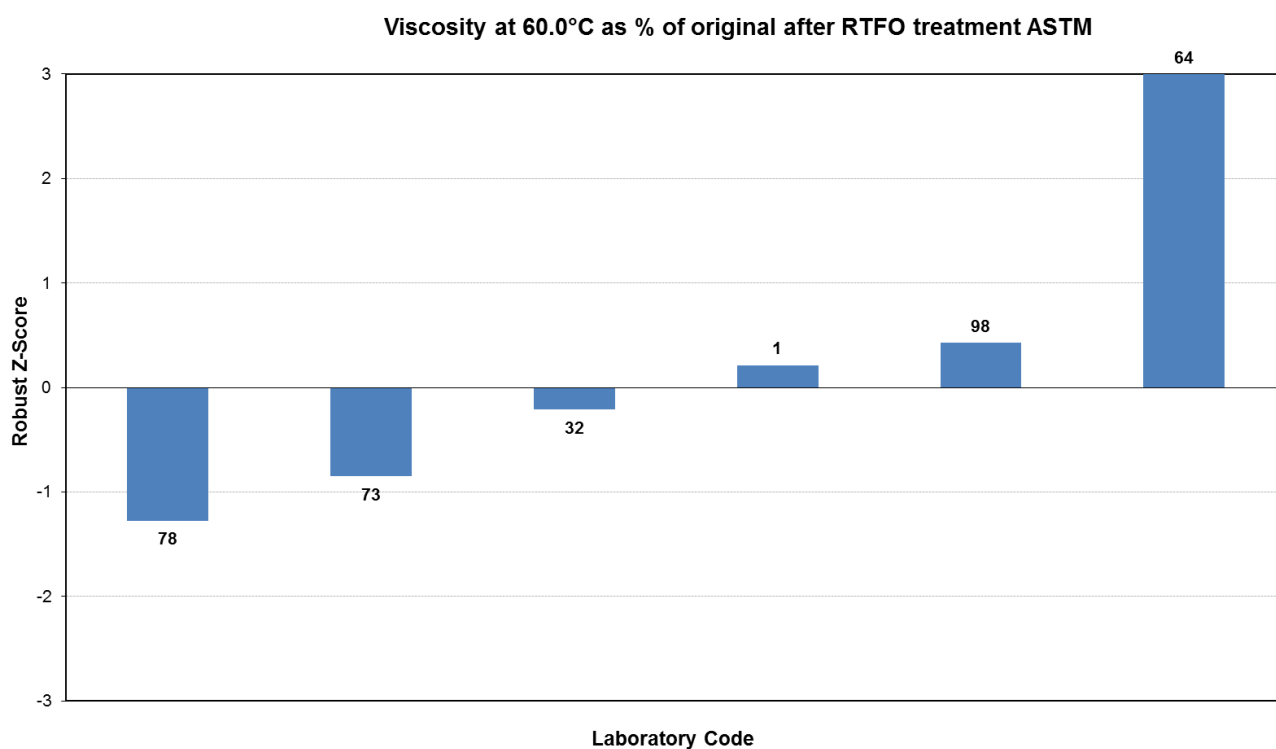


Viscosity at 60.0°C as % of original after RTFO treatment ASTM					
Lab Code	Result	MU	Z-Score		Method
1	201	#	0.21		ASTM D2872-04
5	#	#	na		#
6	#	#	na		#
9	#	#	na		#
12	#	#	na		#
21	#	#	na		#
24	#	#	na		#
32	199	12%	-0.21		AS/NZS 2341.10
34	#	#	na		#
35	#	#	na		#
36	#	#	na		#
39A	#	#	na		#
39B	#	#	na		#
45A	#	#	na		#
45B	#	#	na		#
45C	#	#	na		#
49	#	#	na		#
53	#	#	na		#
59A	#	#	na		#
59B	#	#	na		#
64	227.8	#	5.92	§	#
67	#	#	na		#
68	#	#	na		#
71	#	#	na		#
73	196	12%	-0.85		AS/NZS 2341.10
77	#	#	na		#
78	194.0	#	-1.28		ASTM D2872
95	#	#	na		#
96	#	#	na		#
98	202	#	0.43		AS 2341.2/.10
99	#	#	na		#
100	#	#	na		#

No of Results	6
Median	200.0
Norm IQR	4.7
Uncertainty (Median)	2.4
Robust CV	2.3%
Minimum	194
Maximum	228
Range	34

“§” indicates an outlier

"#" indicates no response was provided by the laboratory
"na" indicates not applicable



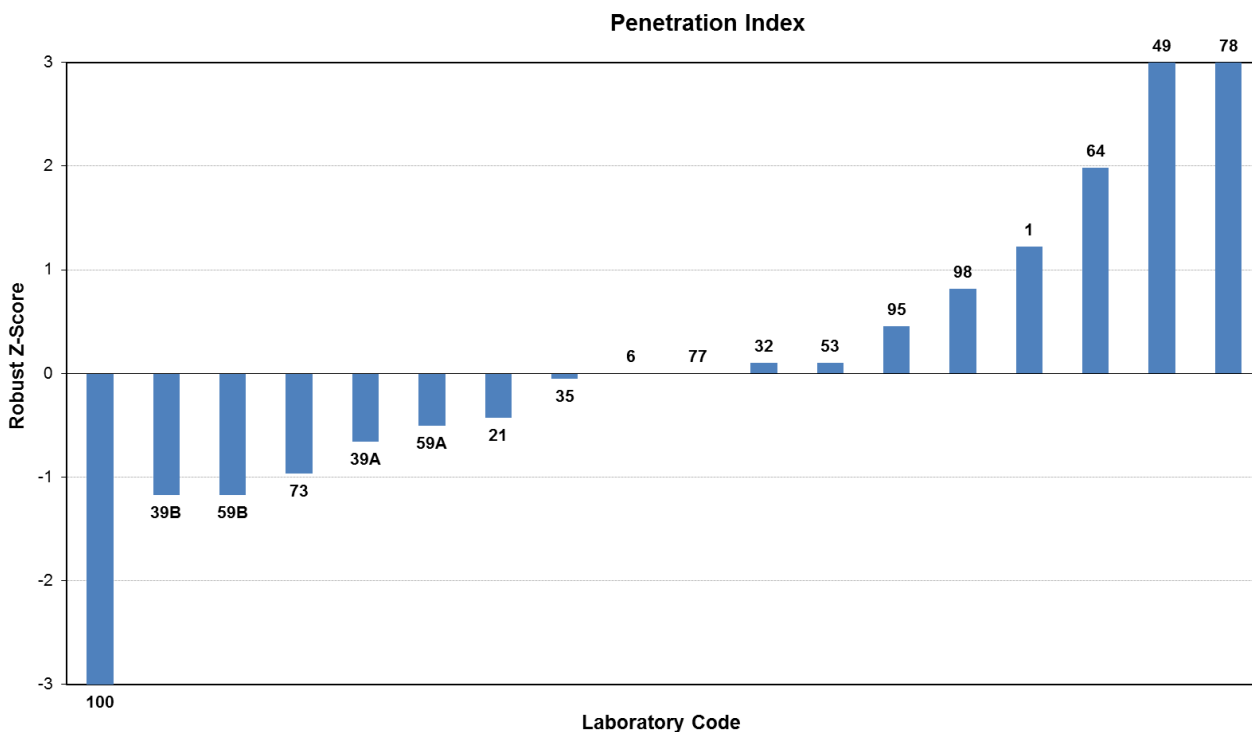
Penetration Index			
Lab Code	Result	Z-Score	
1	-0.76	1.22	
5	#	na	
6	-1.0	0.00	
9	#	na	
12	#	na	
21	-1.084	-0.43	
24	#	na	
32	-0.98	0.10	
34	#	na	
35	-1.01	-0.05	
36	#	na	
39A	-1.13	-0.66	
39B	-1.23	-1.17	
45A	#	na	
45B	#	na	
45C	#	na	
49	-0.387	3.12	§
53	-0.98	0.10	
59A	-1.10	-0.51	
59B	-1.23	-1.17	
64	-0.61	1.99	
67	#	na	
68	#	na	
71	#	na	
73	-1.19	-0.97	
77	-1.0	0.00	
78	-0.166	4.25	§
95	-0.91	0.46	
96	#	na	
98	-0.84	0.81	
99	#	na	
100	-4.805	-19.37	§

No of Results	18
Median	-1.0000
Norm IQR	0.1964
Uncertainty (Median)	0.0580
Robust CV	-19.6%
Minimum	-4.805
Maximum	-0.166
Range	4.639

“\$” indicates an outlier

"#" indicates no response was provided by the laboratory

"na" indicates not applicable



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		
1	#	163.0	DV7+ Brookfield	112	#	Partial filling	Cannon-Manning
5	#	#	#	#	#	Partial	Cannon-Manning
6	163.0	#	#	#	#	Partial	Cannon-Manning
9	#	#	#	#	#	#	#
12	#	#	Brookfield DV2TLVT JO	20.4	#	#	#
21	162.5	#	#	1053	at 60°C pre 1.411, at 60°C post 1.546, average 1.478	Partial	60°C AI100, 135°C CM8
24	#	#	#	#	#	#	#
32	163.0	163.0	Brookfield LV at 135°C	20.4 sec ⁻¹	1.40 sec ⁻¹	Partial	Asphalt Institute
34	#	#	#	#	#	Partial filling	#
35	162.6	#	#	1020	1.42 Pre, 1.50 Post RTFO	Partial	Cannon-Manning
36	#	#	Brookfield LVDV II+	23.8	#	#	#
39A	163.0	#	Brookfield DV-I +	34.00	1.405	Partial	Asphalt Institute
39B	163	#	RVDV-I+	34.00	1.383	Partial	Asphalt Institute
45A	#	#	Brookfield DV-II+PRO	17.0	0.17	#	#
45B	#	#	Brookfield DV-II+PRO	17%	0.17%	#	#
45C	#	#	Brookfield DV-II+PRO	17.0	0.17	#	#

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		
49	163.1	#	Brookfield LV DV-I Prime	#	#	#	Asphalt Institute
53	162.6	#	Brookfield RV at 135°C	34.0	#	#	Asphalt Institute
59A	#	#	RV DV 2-T	34.00	1.499	#	AI
59B	#	#	RV DV 2-T	34.00	1.483	#	AI
64	#	163.0	Brookfield RVDVII+	19.16 N/m ²	115.75 N/m ²	Partial filling	#
67	#	#	#	#	#	#	#
68	162.0	#	#	#	1.41	Partial filling	Asphalt Institute
71	#	#	#	#	6.8120	Partial	A.I at 60°C, C.M at 135°C
73	163.0	163.0	Brookfield LV at 135°C	20.4 sec ⁻¹	1.35 sec ⁻¹	Partial filling	Asphalt Institute
77	#	#	Brookfield HADV-I+, spindle SC4-21, 100 rpm speed	#	#	#	#
78	#	163.0	#	#	#	#	Asphalt Institute
95	#	#	RVDV-II+PX	93 sec ⁻¹	1.25 sec ⁻¹	Partial filling method	#
96	#	#	Brookfield	0.436	#	Partial	#
98	#	163.0	Brookfield LVDVII+	20.40 sec ⁻¹	1.38 sec ⁻¹	Partial	Asphalt Institute
99	163.0	#	#	#	#	Partial	Cannon-Manning
100	163.0	#	Brookfield DV-II+ PRO	28	#	Partial	Cannon-Manning

APPENDIX B

Homogeneity Testing

Homogeneity Testing

The samples utilised in this program were supplied by SAMI Bitumen Technologies Pty Ltd, Camellia Laboratory. For this program, ten samples were randomly selected and tested for homogeneity. Statistical analysis showed that the samples were sufficiently homogeneous so that any results identified as outliers could not be attributed to sample variability.

Penetration at 25°C AS 2341.12

Sample no.	Results p.u.	Deviation
1	69	0.8977
6	70	-0.5386
24	70	-0.5386
34	70	-0.5386
67	69	0.8977
68	70	-0.5386
92	70	-0.5386
100	69	0.8977
Average	69.625	

Viscosity at 135°C AS 2341.4

Results Pa.s.	Deviation
0.430	1.71429
0.440	-0.57143
0.440	-0.57143
0.440	-0.57143
0.440	-0.57143
0.440	-0.57143
0.430	1.71429
0.440	-0.57143
0.440	-0.57143
0.4375	

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

Deviation lies within test method requirements

APPENDIX C

Documentation

Instructions to Participants	C1
Results Sheet	C3

**PROFICIENCY TESTING AUSTRALIA
BITUMEN PROFICIENCY TESTING PROGRAM
ROUND 10
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 containing bitumen C170 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
Penetration Index

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. For calculation of the Penetration Index, the following formula has to be used:

$$PI = (20 - 500A) / (1 + 50A)$$

where $A = ((\text{Log pen}_{25\text{C}} - \text{log}800) / (25 - \text{ASTM softening point}))$, assuming "AS" and "ASTM" softening points are the same.

Determine the softening point and penetration at 25⁰C and apply the computer programme to establish the PI.

8. Participants should use the routine test methods which would normally be used to test samples. Please identify the method used on the Results Sheet.

9. 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.
10. The results for all determinations are to be recorded on the results sheet to the accuracy and reporting basis indicated.
11. Testing may commence as soon as the sample is received. All laboratories must return the results sheet no later than **16 November 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 10**

RESULTS SHEET

Lab Code

PTA
SAMPLE A

TEST (report to)	Result	MU ±	Method
Viscosity at 135.0°C (0.001 Pa.s)			
Density at 15.0C (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) □				
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)				
Penetration Index				

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: _____

- End of Report -