

# FINAL REPORT

Project Title: P74: Better characterising bituminous binders to manage risk and aid development of performance-based binder specifications (Years 1 and 2)

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# 1 INTRODUCTION

This document provides a summary report on binder testing work conducted during Years 1 and 2 of NACOE Project P74 (2017-19). The main goal of the project was to develop a database of binder test properties that can be used to:

1. identify the changes and variability in quality-control and performance-related properties of various grades of bitumen and polymer modified binders (PMBs) supplied in Queensland
2. investigate the relationships between Australian specified test properties and rheological properties measured using the dynamic shear rheometer (DSR).

The database of binder properties obtained in the project included the test results obtained for various grades of bitumen and PMBs which were obtained from six different Queensland binder suppliers during 2017-19. A total of 59 binder samples were tested. These included 42 samples of PMBs, 16 samples of bitumen and 1 sample of a 10% crumb rubber binder. It was initially hoped that samples of each binder grade would be able to be obtained from at least two different suppliers, but some grades were only available from a single supplier. Information about the binder grades, the number of samples tested and the number of suppliers which submitted samples of particular binder grades is included in the relevant sections in this document.

Each of the binder samples were subjected to a range of selected specification tests in accordance with either the Australian bitumen specification (AS 2008) or Australian PMB specification (AGPT/T190) at the TMR laboratory. The same samples were also tested using a DSR at the same laboratory. DSR tests that were conducted included complex viscosity at 60 °C tests at 12% strain, temperature-frequency sweep tests (which were used to generate DSR complex modulus master curves), and stress ratio tests at 10 °C. Table 1.1 provides a summary of the tests conducted on each of the binders and gives an indication of whether the tests were conducted once or in duplicate on each sample.

Elastometer consistency 6% at 60 °C tests have been shown to provide information on the relative rutting performance of binders in asphalt at high road surface temperatures (60 °C). This test parameter currently has a 'to be reported' specification requirement in the current version of AGPT/T190 (published in February 2014). Minimum specification requirements for consistency 6% at 60 °C were agreed at the May 2018 Austroads Bituminous Surfacing Working Group (BSWG) meeting after results obtained from industry were analysed. These agreed limits for consistency 6% at 60 °C have been included in relevant parts of this document as they have been proposed to be included in the next update to AGPT/T190.

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Table 1.1: Test methods used during the project

	Test property	Method of test	Number of tests on each sample
AS 2008-related properties	Viscosity at 60 °C (Pa.s)	AS/NZS 2341.2	Duplicate
	Viscosity at 60 °C after RTFO treatment (Pa.s), and: Percentage of original after RTFO (%)	AS/NZS 2341.2 AS/NZS 2341.10	Viscosity tests in duplicate: Single result of percentage increase was calculated using duplicate viscosity results.
	Viscosity at 135 °C (Pa.s)	AS/NZS 2341.2	Duplicate
	Penetration at 25 °C (0.1 mm)	AS 2341.12	Duplicate
	Penetration at 25 °C after RTFO treatment (0.1 mm)	AS 2341.12 AS/NZS 2341.10	Duplicate
AGPT/T190-related properties	Viscosity at 165 °C (Pa.s)	AGPT/T111	Duplicate
	Torsional recovery at 25 °C (%)	AGPT/T122	Duplicate
	Softening point (°C)	AGPT/T131	Duplicate
	Consistency at 60 °C (Pa.s)	AGPT/T121	Duplicate
	Consistency 6% at 60 °C (Pa.s)	AGPT/T121	Duplicate
	Segregation (%)	AGPT/T108	Single tests on each sample The individual softening point tests that were conducted as part of this test were performed in duplicate.
	<i>DSR stress ratio at 10 °C (proposed to be included in the next update to AGPT/T190)</i>	AGPT/T125	Duplicate
DSR test properties	Complex viscosity ( $\eta^*$ ) 12% at 60 °C (Pa.s)	Modified version of AGPT/T192	Duplicate
	Temperature-frequency sweep tests	Modified version of EN 14770	Duplicate

The results of DSR stress ratio at 10 °C tests (AGPT/T125) have also been found to provide information about the relative resistance of different binders to low temperature (10 °C) fatigue cracking on the road. Due to this, it has been proposed by the Austroads BSWG to include DSR stress ratio at 10 °C tests in the next update to AGPT/T190 as a 'to be reported' test parameter. It is expected that a dataset of industry results will be obtained over time due to this 'report' requirement. Minimum specification requirements for this test can then be set based on analysis of the industry results which have been obtained for different binder grades. DSR stress ratio at 10 °C tests were conducted on the PMBs included in the study to provide an initial TMR dataset of test results for different PMB grades.

DSR complex viscosity at 60 °C and 12% strain (DSR  $\eta^*$  12% at 60 °C) tests were conducted on binder samples to determine whether this DSR test parameter would give similar results to AGPT/T190 consistency 6% at 60 °C tests. These tests were conducted by subjecting each sample tested sequentially to oscillation frequencies of 1.67 and 5.00 rad/s. This testing protocol allowed DSR  $\eta^*$  12% at 60 °C results to be obtained at both oscillation frequencies.

DSR complex viscosity and DSR temperature-frequency sweep tests were conducted on each binder sample without removing it from the DSR. Complex viscosity tests were performed first, followed by temperature-frequency sweep tests. Temperature-frequency sweep tests were

performed by measuring the complex modulus ( $G^*$ , Pa) and phase angle ( $\delta$ , degree) of the samples using test temperatures of between 20 and 60 °C (at 10 °C intervals) and oscillation frequencies of between 0.631 and 63.1 rad/s (at five logarithmic intervals per decade). Samples were tested at 60 °C first and then the test temperature was lowered progressively during each test. Master curves of complex modulus ( $G^*$ ) results were generated from the results obtained in temperature-frequency sweep tests by using the equations and methods described in AGPT/T274. These were converted into complex viscosity results ( $\eta^*$ , Pa.s) by dividing the  $G^*$  result obtained for a binder sample by the relevant oscillation frequency (e.g. 1 rad/s).

## 2 OBJECTIVE 1-1: VARIABILITY IN QUALITY/PERFORMANCE PROPERTIES OF BITUMENS

### 2.1 Test results obtained for C170 and C320 bitumen samples

Four samples of C170 bitumen and four samples of C320 bitumen were obtained from three different suppliers in the study. Each individual sample conformed to relevant specification requirements in terms of the properties that were tested. Table 2.1 and Table 2.2 summarise the results of a simple statistical analysis (i.e. mean, standard deviation and coefficient of variance (CoV)) which were obtained from samples which represented each bitumen grade. The statistical results were obtained in order to provide information on product conformance and variability. Relevant AS 2008-specified limits for the two bitumen grades are also included in the tables. Some of the mean test results shown in Sections 2 and 3 of this document have been listed with a greater level of precision than required by the relevant test method. Mean test results have been presented in this way so that statistical differences between the samples can be more easily observed.

Table 2.1: AS 2008-related test results obtained for C170 bitumen samples

Test property	Test method	Mean of test results	AS 2008 requirement	Standard deviation of test results	Coefficient of Variation of test results (%)
Viscosity at 60 °C (Pa.s)	AS/NZS 2341.2	182.0	140–200	8.37	4.6
Viscosity at 60 °C, percentage of original after RTFO (%)	AS/NZS 2341.2 AS/NZS 2341.10	178.3	300 max	16.94	9.5
Viscosity at 135 °C (Pa.s)	AS/NZS 2341.2	0.386	0.25–0.45	0.028	7.3
Penetration at 25 °C (0.1 mm)	AS 2341.12	70.3	62 min	2.63	3.7

Table 2.2: AS 2008-related test results obtained for C320 bitumen samples

Test property	Test method	Mean of test results	AS 2008 requirement	Standard deviation of test results	Coefficient of Variation of test results (%)
Viscosity at 60 °C (Pa.s)	AS/NZS 2341.2	312.0	260–380	30.22	9.7
Viscosity at 60 °C, percentage of original after RTFO (%)	AS/NZS 2341.2 AS/NZS 2341.10	214.8	300 max	17.91	8.3
Viscosity at 135 °C (Pa.s)	AS/NZS 2341.2	0.498	0.40–0.65	0.032	6.4
Penetration at 25 °C (0.1 mm)	AS 2341.12	54.3	40 min	3.50	6.5

Table 2.1 and Table 2.2 show that all tested properties had low CoV values (less than 10%) indicating that the sample-to-sample variation observed for these bitumens was quite small. An insight into the probability that C170 and C320 bitumens (which were sourced from the three different suppliers) would meet AS 2008 specification requirements was gained by comparing the lower and upper 95% confidence limits of results obtained from further statistical analysis of the data. These confidence limits were calculated using the mean and standard deviation values shown in the tables. It was also assumed that the test results obtained for batches produced by these suppliers followed a normal distribution.

Table 2.3 and Table 2.4 compare the AS 2008 specification requirements for C170 and C320 bitumen grades with the lower and upper 95% confidence limits obtained for each test parameter. A lower 95% confidence limit has not been included in the tables for the post-RTFO viscosity at 60 °C test parameter as AS 2008 requirements for this test do not specify a minimum limit in this test. In the same way, a higher 95% confidence limit has not been included for the penetration at 25 °C test parameter as AS 2008 only includes a minimum limit for penetration at 25 °C test results. The mean results obtained for each tested property as well as the range of values obtained during testing have also been included in the tables for reference.

All relevant 95% confidence limits were found to be well within the specified limits listed in Table 2.3 and Table 2.4. This implies that C170 and C320 bitumens obtained from the suppliers included in this study would be expected to comply with tested AS 2008 requirements with a high level of probability (if batch results obtained from the suppliers are normally distributed).

**Table 2.3: Conformance-probability of C170 bitumen samples**

Test property	AS 2008 requirement (min)	Lower 95% confidence limit	Mean (& range of test results)	Upper 95% confidence limit	AS 2008 requirement (max)
Viscosity at 60 °C (Pa.s)	140	168.3	182.0 (175.0–194.0)	195.7	200
Viscosity at 60 °C, percentage of original after RTFO (%)	–	–	178.3 (160.0–201.0)	206.0	300
Viscosity at 135 °C (Pa.s)	0.25	0.339	0.386 (0.344–0.407)	0.432	0.45
Penetration at 25 °C (0.1 mm)	62	65.9	70.3 (68.0–73.0)	–	–

**Table 2.4: Conformance-probability of C320 bitumen samples**

Test property	AS 2008 requirement (min)	Lower 95% confidence limit	Mean (& range of test results)	Upper 95% confidence limit	AS 2008 requirement (max)
Viscosity at 60 °C (Pa.s)	260	262.4	312.0 (281.0–349.0)	361.6	380
Viscosity at 60 °C, percentage of original after RTFO (%)	–	–	214.8 (199.0–240.0)	244.1	300
Viscosity at 135 °C (Pa.s)	0.40	0.446	0.498 (0.461–0.538)	0.550	0.65
Penetration at 25 °C (0.1 mm)	40	48.5	54.3 (50.0–58.0)	–	–

## 2.2 Test results obtained for C600 bitumen samples

Four different samples of C600 bitumen were obtained from two different suppliers during the study. All individual samples conformed to specification requirements for the properties tested. Table 2.5 summarises the results of a simple statistical analysis of the AS 2008-related results obtained for this binder grade. The results are presented in the same way as in Table 2.1 and

Table 2.2. An insight into the probability that C600 bitumens (which were sourced from different suppliers) would meet AS 2008 specification requirements is included in Table 2.6. This table contains the equivalent information to Table 2.3 and Table 2.4.

**Table 2.5: AS 2008-related test results for C600 bitumen samples**

Test property	Test method	Mean of test results	AS 2008 requirement	Standard deviation of test results	Coefficient of Variation of test results (%)
Viscosity at 60 °C (Pa.s)	AS/NZS 2341.2	607.8	500–700	41.15	6.8
Viscosity at 60 °C, percentage of original after RTFO (%)	AS/NZS 2341.2 AS/NZS 2341.10	251.0	300 max	31.70	12.6
Viscosity at 135 °C (Pa.s)	AS/NZS 2341.2	0.690	0.60–0.85	0.009	1.3
Penetration at 25 °C (0.1 mm)	AS 2341.12	38.3	20 min	2.63	6.9

**Table 2.6: Conformance-probability of C600 bitumen samples**

Test property	AS 2008 requirement (min)	Lower 95% confidence limit	Mean (& range of test results)	Upper 95% confidence limit	AS 2008 requirement (max)
Viscosity at 60 °C (Pa.s)	500	540.3	607.8 (567.0–648.0)	675.2	700
Viscosity at 60 °C, percentage of original after RTFO (%)	–	–	251.0 (208.0–281.0)	303.0	300
Viscosity at 135 °C (Pa.s)	0.60	0.675	0.690 (0.683–0.703)	0.705	0.85
Penetration at 25 °C (0.1 mm)	20	33.9	38.3 (36.0–42.0)	–	–

The results shown in Table 2.5 indicate that the sample-to-sample variation for C600 bitumen samples was quite small for three of the tested properties (CoV values less than 10%). The CoV value obtained for the percentage increase in post-RTFO viscosity at 60 °C test was higher than that observed in other tests (13%). This may have caused the upper 95% confidence limit obtained for this property in Table 2.6 to be slightly higher than the AS 2008-specified limit of 300% maximum. Even though this was the case, the upper 95% confidence limit was only 3% higher than the specified maximum limit for this test parameter and all four individual results were well below this limit. Based on these observations, it appears that C600 samples obtained from the three suppliers included in the study would be expected to meet AS 2008 requirements for the percentage increase in post-RTFO viscosity at 60 °C test with a fairly high level of probability. As the upper and lower 95% confidence limits for the other test parameters in Table 2.6 were all well within relevant AS 2008 specification limits, the results obtained in the study appear to indicate that C600 bitumen obtained from these suppliers would be expected to meet these test requirements with a high level of probability.

## 2.3 Test results obtained for M1000 bitumen samples

Four different samples of M1000 bitumen were obtained from one supplier in this study. All individual samples conformed to specification requirements for the properties tested. Table 2.7

provides basic statistical information presented in the same way as Table 2.5, regarding the AS 2008-related test results applicable to these bitumen samples. M1000 bitumen is specified in AS 2008 in terms of viscosity at 60 °C and penetration at 25 °C results which are obtained after binder samples have been subjected to rolling thin film oven (RTFO) treatment. This differs from the specification requirements for C170, C320 and C600 bitumens where these properties are specified on binder samples prior to them being subjected to RTFO treatment.

**Table 2.7: AS 2008-related test results for M1000 bitumen samples**

Test property	Test method	Mean of test results	AS 2008 requirement	Standard deviation of test results	Coefficient of Variation of test results (%)
Viscosity at 60 °C after RTFO treatment (Pa.s)	AS/NZS 2341.2 AS/NZS 2341.10	5170.3	3500–6500	695.7	13.5
Viscosity at 135 °C (Pa.s)	AS/NZS 2341.2	1.047	1.5 max	0.059	5.7
Penetration at 25 °C after RTFO treatment (0.1 mm)	AS 2341.12 AS/NZS 2341.10	27.8	26 min	1.71	6.2

An insight into the probability that M1000 bitumens (which were sourced from the same supplier) would meet AS 2008 specification requirements is given in Table 2.8. This table shows the analogous conformance probability results to that shown in Table 2.6.

**Table 2.8: Conformance-probability of M1000 bitumen samples**

Test property	AS 2008 requirement (min)	Lower 95% confidence limit	Mean (& range of test results)	Upper 95% confidence limit	AS 2008 requirement (max)
Viscosity at 60 °C after RTFO treatment (Pa.s)	3500	4029.4	5170.3 (4713.0–6204.0)	6311.1	6500
Viscosity at 135 °C (Pa.s)	–	–	1.047 (0.994–1.128)	1.144	1.50
Penetration at 25 °C after RTFO treatment (0.1 mm)	26	25.0	27.8 (26.0–30.0)	–	–

The results shown in Table 2.7 indicate that the post-RTFO viscosity at 60 °C property had a higher sample-to-sample variation (CoV = 13.5%) than other tested properties which showed quite small variations (CoV < 10%). Even though this was the case, the 95% confidence limits calculated for this test property were well within AS 2008 specified limits (Table 2.8). The upper 95% confidence limit for viscosity at 135 °C in Table 2.8 was also well with the maximum specified requirement for this test property. Based on these results, M1000 bitumen obtained from the single supplier used in this study would be expected meet the requirements for these two tests with a high level of probability (if batch results obtained from the supplier were normally distributed).

The lower 95% confidence limit obtained for the post-RTFO penetration at 25 °C property was found to be slightly lower than the specified limit of 26 (0.1 mm) minimum, and also one individual result was found to be at this limit. Based on these results, M1000 bitumen obtained from this supplier may fail to meet the requirements of the post-RTFO penetration at 25 °C property on some occasions.



## 3 OBJECTIVE 1-2: VARIABILITY IN QUALITY/PERFORMANCE PROPERTIES OF PMBS

### 3.1 Test results obtained for A15E grade binders

Fourteen different A15E grade PMB samples were obtained from one supplier in the study. Table 3.1 summarises the AGPT/T190-related results obtained for the A15E binder samples that were tested. The table includes the results obtained in AGPT/T190 tests which currently have specified limits, as well as the results obtained in consistency 6% at 60 °C and DSR stress ratio at 10 °C tests which have been identified as being able to rank binder performance on the road. Consistency-related tests were conducted using mould A test conditions as per AGPT/T190. Current AGPT/T190 specified limits and basic statistical parameters (i.e. mean, standard deviation, CoV) are included in the table to provide information on product conformance and variability. The recently agreed consistency 6% at 60 °C specification limit for an A15E grade PMB and the proposed 'report' requirement for DSR stress ratio at 10 °C tests are also included in Table 3.1.

Individual binder samples conformed to all the pertinent property requirements in most cases, except for one sample that failed the minimum consistency at 60 °C requirement as noted in the table. The test results obtained for this binder sample were included in the analysis along with the other binder samples.

**Table 3.1: AGPT/T190-related test results for A15E grade binders**

Test property	Test method	Mean of test results	AGPT/T190 requirement	Standard deviation of test results	Coefficient of Variation of test results (%)
Viscosity at 165 °C (Pa.s)	AGPT/T111	0.796	0.90 max	0.062	7.8
Torsional recovery at 25 °C (%)	AGPT/T122	68.6	55–80	1.83	2.7
Softening point (°C)	AGPT/T131	97.2	82–105	1.85	1.9
Consistency at 60 °C (Pa.s)	AGPT/T121 (Mould A)	10838.4	5000 min	3157.3	32.5
Consistency 6% at 60 °C (Pa.s)	AGPT/T121 (Mould A)	1457.0	900 min <sup>(1)</sup>	163.2	11.2
Segregation (%)	AGPT/T108	1.3	8 max	0.93	70.6
DSR stress ratio at 10 °C <sup>(2)</sup>	AGPT/T125	2.63	Report <sup>(2)</sup>	0.22	8.5
Conformance	One sample failed the minimum consistency at 60 °C limit for an A15E grade binder (i.e. 5000 Pa.s) in AGPT/T190. All of the other 13 samples met AGPT/T190 limits for the properties that were tested.				

Note 1: The listed consistency 6% at 60 °C limit for A15E was agreed by Austroads BSWG in May 2018.

Note 2: This property has been proposed by Austroads BSWG to be included in AGPT/T190 as a 'report' property when it is next revised.

Table 3.1 shows that four of the test properties (i.e. viscosity at 165 °C, torsional recovery at 25 °C, softening point and DSR stress ratio at 10 °C) had CoV values of less than 10% which indicates that the sample-to-sample variation for the A15E binder was generally small with regard to these test properties. Consistency at 60 °C results showed a noticeably high CoV of 32.5% (partly due to one sample that had a particularly lower consistency value than the specified minimum), but this property is proposed by the Austroads BSWG to be replaced by the consistency 6% at 60 °C property in the next update to AGPT/T190 (as the latter property appears to be able to better rank the relative rutting performance of different binders in asphalt). The results obtained in consistency 6% at 60 °C tests showed a much lower CoV value of 11% than consistency at 60 °C results.

The results obtained in segregation tests showed the highest CoV (70.6%), however, this is most likely due to the consistently low results (0.0 to 3.0%) obtained (i.e. minor deviations from a very small mean value can cause a high CoV to be determined). All individual segregation results comfortably met the specified limit of 8% maximum indicating that the binder appeared stable in terms of polymer segregation during hot storage.

An insight into the probability that A15E binders (which were sourced from a single supplier) would meet AGPT/T190 specification requirements was gained by comparing the lower and/or upper 95% confidence limits obtained from the data shown in Table 3.1. These confidence limits were determined using the same method as was used in the case of the bitumen samples that were tested (Section 2). It was again assumed that the test results obtained for A15E binders followed a normal distribution.

Table 3.2 compares the lower and/or upper AGPT/T190 specified limits for a A15E grade PMB with the relevant 95% confidence limits that were calculated. In a similar way to the analysis of the bitumen sample test results, only lower 95% confidence limits were calculated if AGPT/T190 requirements only included a minimum specified value for a particular test. Only higher 95% confidence limits were determined if AGPT/T190 requirements only included a maximum specified value for a particular test. The associated mean test results and range of values obtained during testing have also been included in Table 3.2 for reference. Consistency 6% at 60 °C, rather than consistency at 60 °C results, and DSR stress ratio at 10 °C results have been included in the table to reflect proposed future changes to AGPT/T190.

All relevant 95% confidence limits were found to be well within the specified limits listed in Table 3.2 even though one non-conforming sample was included in the analysis. This implies that A15E binders, which were obtained from a single supplier in this study, would be expected to comply with AGPT/T190 requirements in terms of the tests performed (if batch results obtained from this supplier were normally distributed).

**Table 3.2: Conformance-probability of A15E binder samples**

Test property	AGPT/T190 requirement (min)	Lower 95% confidence limit	Mean (& range of test results)	Upper 95% confidence limit	AGPT/T190 requirement (max)
Viscosity at 165 °C (Pa.s)	–	–	0.796 (0.673–0.878)	0.898	0.90
Torsional recovery at 25 °C (%)	55	65.57	68.6 (64.0–71.0)	71.57	80
Softening point (°C)	82	94.2	97.2 (94.5–101.0)	100.2	105
Consistency 6% at 60 °C (Pa.s)	900 min <sup>(1)</sup>	1189.3	1457.0 (1066.0–1730.0)	–	–
Segregation (%)	–	–	1.3 (0.0–3.0)	2.9	8.0
DSR stress ratio at 10 °C <sup>(2)</sup>	Report <sup>(2)</sup>	2.26	2.63 (2.20–3.06)	2.99	Report <sup>(2)</sup>

Note 1: The listed Consistency 6% at 60 °C limit for A15E was agreed by BSWG in May 2018.

Note 2: This property has been proposed by Austroads BSWG to be included in AGPT/T190 as a 'report' property when it is next revised.

### 3.2 Test results obtained for A10E binders

Two different samples of an A10E grade PMB were obtained from one supplier for this study. Table 3.3 provides the AGPT/T190-related results obtained for each of the individual samples as well as the current AGPT/T190 specification limits for this PMB grade. The Austroads BSWG proposed specification requirements for consistency 6% at 60 °C and DSR stress ratio at 10 °C tests are also included in the table. Consistency-related tests were conducted on A10E binder samples using mould A test conditions as per AGPT/T190. The results shown in Table 3.3 indicate that the two samples comfortably satisfied specification limits for the properties tested.

**Table 3.3: AGPT/T190-related test results for A10E grade binders**

Test property	Test method	Test results		AGPT/T190 requirement
		Sample A	Sample B	
Viscosity at 165 °C (Pa.s)	AGPT/T111	0.755	0.722	1.10 max
Torsional recovery at 25 °C (%)	AGPT/T122	70.0	63.0	60–86
Softening point (°C)	AGPT/T131	97.5	95.0	88–110
Consistency at 60 °C (Pa.s)	AGPT/T121 (Mould A)	13716.0	10956.0	6000 min
Consistency 6% at 60 °C (Pa.s)	AGPT/T121 (Mould A)	1494.0	1304.0	1000 min <sup>(1)</sup>
Segregation (%)	AGPT/T108	0.0	2.5	8 max
DSR stress ratio at 10 °C <sup>(2)</sup>	AGPT/T125	2.74	2.57	Report <sup>(2)</sup>
Conformance	Two samples satisfied the AGPT/T190 limits for tested properties.			

Note 1: The listed consistency 6% at 60 °C limit for A10E as agreed by the BSWG in May 2018.

Note 2: This property has been proposed by Austroads BSWG to be included in AGPT/T190 as a 'report' property when it is next revised.

The results obtained for the A10E binders were not subjected to statistical analysis as only two binder samples were tested. The statistical parameters obtained from the analysis of a set of two test results are unlikely to reflect the general trends that would be obtained if more samples were tested.

### 3.3 Test results obtained for A35P binders

Four different samples of an A35P grade PMB were obtained from two different suppliers in this study. Table 3.4 summarises the AGPT/T190-related results and basic statistical results obtained for the A35P binder samples in the study. These are presented in the same way as that shown for the A15E grade binder in Table 3.1. Consistency-related tests were conducted on A35P binder samples using mould A test conditions as per AGPT/T190.

It is noted in Table 3.4 that all four samples failed to meet the segregation requirement of 8% maximum. The results obtained for the individual samples were all close to 15% indicating that these binders were quite susceptible to polymer segregation during hot storage. A35P-grade binders are typically manufactured using a plastomeric ethylene vinyl acetate (EVA) polymer which is known to be completely miscible with bitumen (i.e. segregation results should be close to 0% if a typical EVA polymer is used). The high segregation results obtained in the study suggest that the two suppliers which provided A35P binder samples in this study could produce these binders using a different type of polymer additive than EVA which is not particularly miscible with bitumen. Alternatively, these types of PMBs could contain more than one polymer type (i.e. an EVA polymer and another type of polymer which is not particularly miscible with bitumen). Although the A35P grade binder samples showed high segregation test results, no polymer lumps were present in the samples when they were inspected immediately prior to testing.

**Table 3.4: AGPT/T190-related test results for A35P grade binders**

Test property	Test method	Mean of test results	AGPT/T190 requirement	Standard deviation of test results	Coefficient of Variation (%) of test results
Viscosity at 165 °C (Pa.s)	AGPT/T111	0.461	0.60 max	0.046	10.0
Torsional recovery at 25 °C (%)	AGPT/T122	20.3	6–21	4.27	21.1
Softening point (°C)	AGPT/T131	65.9	62–74	1.65	2.5
Consistency at 60 °C (Pa.s)	AGPT/T121 (Mould A)	3204.5	2000 min	1110.3	34.7
Consistency 6% at 60 °C (Pa.s)	AGPT/T121 (Mould A)	1302.3	1000 min <sup>(1)</sup>	432.3	33.2
Segregation (%)	AGPT/T108	15.3	8 max	1.26	8.3
DSR stress ratio at 10 °C <sup>(2)</sup>	AGPT/T125	1.21	Report <sup>(2)</sup>	0.29	24.2
Conformance	All four samples failed the segregation test. Two samples failed to meet the AGPT/T190 maximum torsional recovery limit. One sample failed to meet the proposed AGPT/T190 minimum consistency 6% at 60 °C limit.				

Note 1: The listed consistency 6% at 60 °C limit for A35P as agreed by the BSWG in May 2018.

Note 2: This property has been proposed by Austroads BSWG to be included in AGPT/T190 as a 'report' property when it is next revised.

The results shown in Table 3.4 indicate that many test properties had high CoV values (up to around 30%) which were noticeably higher than the CoV results obtained for other binders used for this study. These high results most likely reflect the instability of the polymer in the PMB samples tested. If a polymer in a PMB is extremely prone to segregate during hot storage, then standard laboratory mixing processes (which are conducted prior to testing) are not always sufficient to redisperse the polymer in a PMB. If the polymer in the PMB is somewhat prone to segregation (rather than extremely prone to segregation), then the polymer in the binder can be dispersed during laboratory mixing. The sample obtained by the laboratory, however, may not be representative of PMB production batch if the whole batch is not sufficiently mixed when the sample is taken (as the level of polymer in the binder will be different in different parts of the production batch).

Based on the results obtained for the A35P binder samples, it appears that A35P grade binders which were obtained from the two suppliers used in the study would not be expected to meet all AGPT/T190-specified requirements for this binder grade. As all tested binder samples failed the requirements of the segregation test, the results obtained for the binders were not statistically analysed to determine 95% confidence limits for each specified test property.

### 3.4 Test results obtained for S35E grade binders

Fourteen different samples of an S35E grade PMB were obtained from one supplier for this study. All individual samples conformed to specification requirements for the properties tested. Table 3.5 summarises the AGPT/T190-related results which were obtained for the S35E binder samples. These are presented in the same way as Table 3.1. Consistency-related tests were conducted on S35E binder samples using mould B test conditions as per AGPT/T190.

Table 3.5 shows that most properties had low CoV values (less than 10%) indicating that the sample-to-sample variation of the S35E binder was quite small. Viscosity at 165 °C test results showed a higher CoV of 14%, but individual viscosity at 165 °C results were all well below the maximum specification limit. As in the case of tests conducted on the A15E binder samples, the

results of segregation tests showed the highest CoV result (110.8%). This high CoV result is again likely due to the consistently low segregation values (0.0 to 1.0%) obtained in these tests. All individual segregation test results obtained for S35E binder samples comfortably met the specified limit of 8% maximum indicating that the binder was stable in terms of polymer segregation during hot storage.

**Table 3.5: AGPT/T190-related test results for S35E grade binders**

Test property	Test method	Mean of test results	AGPT/T190 requirement	Standard deviation of test results	Coefficient of Variation (%) of test results
Viscosity at 165 °C (Pa.s)	AGPT/T111	0.280	0.55 max	0.039	14.0
Torsional recovery at 25 °C (%)	AGPT/T122	23.0	16–32	1.96	8.5
Softening point (°C)	AGPT/T131	51.9	48–56	1.20	2.3
Consistency at 60 °C (Pa.s)	AGPT/T121 (Mould B)	461.9	300 min	40.26	8.7
Consistency 6% at 60 °C (Pa.s)	AGPT/T121 (Mould B)	325.2	250 min <sup>(1)</sup>	27.26	8.4
Segregation (%)	AGPT/T108	0.4	8 max	0.48	110.8
DSR stress ratio at 10 °C <sup>(2)</sup>	AGPT/T125	1.77	Report <sup>(2)</sup>	0.08	4.6
Conformance	All 14 samples satisfied the AGPT/T190 limits for tested properties.				

Note 1: The listed consistency 6% at 60 °C limit for S35E as agreed by the BSWG in May 2018.

Note 2: This property has been proposed by Austroads BSWG to be included in AGPT/T190 as a 'report' property when it is next revised.

An insight into the probability that S35E binders (which were sourced from a single supplier) would meet AGPT/T190 specification requirements is given in Table 3.6, where statistical analysis results and specification limits are presented the same way as Table 3.2 for the S35E binder samples.

**Table 3.6: Conformance-probability of S35E binder samples**

Test property	AGPT/T190 requirement (min)	Lower 95% confidence	Mean (& range of test results)	Upper 95% confidence	AGPT/T190 requirement (max)
Viscosity at 165 °C (Pa.s)	–	–	0.280 (0.185–0.349)	0.344	0.55
Torsional recovery at 25 °C (%)	16	19.8	23.0 (19.0–26.0)	26.2	32
Softening point (°C)	48	49.9	51.9 (50.0–54.0)	53.9	56
Consistency 6% at 60 °C (Pa.s)	250 <sup>(1)</sup>	280.5	325.2 (294.0–403.0)	–	–
Segregation (%)	–	–	0.4 (0.0–1.0)	1.2	8.0
DSR stress ratio at 10 °C <sup>(2)</sup>	Report <sup>(2)</sup>	1.63	1.77 (1.65–1.93)	1.90	Report <sup>(2)</sup>

Note 1: The listed consistency 6% at 60 °C limit for S35E was agreed by BSWG in May 2018.

Note 2: This property has been proposed by Austroads BSWG to be included in AGPT/T190 as a 'report' property when it is next revised.

All 95% confidence limits were found to be well within the relevant specified limits as shown in Table 3.6. This implies that S35E binders which were obtained from the supplier used in this study would be expected to comply with AGPT/T190 requirements in terms of the tests performed (if batch results obtained from this supplier were normally distributed).

### 3.5 Test results obtained for S25E binders

Four different samples of an S25E grade PMB were obtained from two different suppliers in this study. All individual samples conformed to specification requirements for the properties tested. Table 3.7 summarises the AGPT/T190-related results obtained for the S25E binder samples which are presented in the same way as Table 3.1. Consistency-related tests were conducted on S25E binder samples using mould A test conditions as per AGPT/T190.

**Table 3.7: AGPT/T190-related test results for S25E grade binders**

Test property	Test method	Mean of test results	AGPT/T190 requirement	Standard deviation of test results	Coefficient of Variation (%) of test results
Viscosity at 165 °C (Pa.s)	AGPT/T111	0.610	0.80 max	0.034	5.6
Torsional recovery at 25 °C (%)	AGPT/T122	69.5	54–85	6.03	8.7
Softening point (°C)	AGPT/T131	93.4	82–100	5.39	5.8
Consistency at 60 °C (Pa.s)	AGPT/T121 (Mould A)	14251.3	5000 min	6318.1	44.3
Consistency 6% at 60 °C (Pa.s)	AGPT/T121 (Mould A)	1437.0	900 min <sup>(1)</sup>	481.2	33.5
Segregation (%)	AGPT/T108	0.0	8 max	0.0	0.0
DSR stress ratio at 10 °C <sup>(2)</sup>	AGPT/T125	2.19	Report <sup>(2)</sup>	0.35	16.1
Conformance	All four samples satisfied the AGPT/T190 limits for tested properties.				

Note 1: The listed consistency 6% at 60 °C limit for S25E as agreed by the BSWG in May 2018.

Note 2: This property has been proposed by Austroads BSWG to be included in AGPT/T190 as a 'report' property when it is next revised.

The results shown in Table 3.7 indicate that the consistency at 60 °C, consistency 6% at 60 °C and DSR stress ratio at 10 °C test properties showed high CoV values indicating that the sample-to-sample variation of the S25E binder in terms of these test properties was quite high. All other test properties showed low CoV values (i.e. less than 10%).

An insight into the probability that S25E binders (which were sourced from two different suppliers) would meet AGPT/T190 specification requirements is given in Table 3.8 where statistical analysis results and specification limits are presented the same way as Table 3.2. The results shown in the table indicate that the upper 95% confidence limit result obtained for the softening point property (102.2 °C) was slightly higher than the relevant specification limit of 100 °C maximum. The lower 95% confidence limit obtained for the consistency 6% at 60 °C property (647.8 Pa.s) was also noticeably lower than its proposed specification limit of 900 Pa.s minimum.

Even though all individual S25E binder samples met AGPT/T190-specified requirements in terms of the tests performed, the results of the CoV analysis suggest that S25E binder batches (which were obtained from two different binder supplies) may fail current AGPT/T190 requirements for softening point on some occasions. Batches obtained from these two suppliers may also fail proposed consistency 6% at 60 °C test requirements on some occasions.



**Table 3.8: Conformance-probability of S25E binder samples**

Test property	AGPT/T190 requirement (min)	Lower 95% confidence	Mean (& range of test results)	Upper 95% confidence	AGPT/T190 requirement (max)
Viscosity at 165 °C (Pa.s)	–	–	0.610 (0.560–0.635)	0.665	0.80
Torsional recovery at 25 °C (%)	54	59.6	69.5 (61.0–75.0)	79.4	85
Softening point (°C)	82	84.5	93.4 (85.5–97.5)	102.2	100
Consistency 6% at 60 °C (Pa.s)	900 <sup>(1)</sup>	647.8	1437.0 (1124.0–2154.0)	–	–
Segregation (%)	–	–	0.0 (0.0–0.0)	0.0	8.0
DSR stress ratio at 10 °C <sup>(2)</sup>	Report <sup>(2)</sup>	1.61	2.19 (1.77–2.48)	2.76	Report <sup>(2)</sup>

Note 1: The listed consistency 6% at 60 °C limit for S15E was agreed by BSWG in May 2018.

Note 2: This property has been proposed by Austroads BSWG to be included in AGPT/T190 as a 'report' property when it is next revised.

### 3.6 Test results obtained for S15E binders

Four different samples of an S15E grade PMB were obtained from one supplier for this study. All individual samples conformed to specification requirements for the properties tested. Table 3.9 summarises the AGPT/T190-related results obtained for the S15E binder samples presented in the same way as Table 3.1. Consistency-related tests were conducted on S15E binder samples using mould A test conditions as per AGPT/T190.

**Table 3.9: AGPT/T190-related test results for S15E grade binders**

Test property	Test method	Mean of test results	AGPT/T190 requirement	Standard deviation of test results	Coefficient of Variation (%) of test results
Viscosity at 165 °C (Pa.s)	AGPT/T111	0.446	0.55 max	0.055	12.4
Torsional recovery at 25 °C (%)	AGPT/T122	43.5	32–62	6.86	15.8
Softening point (°C)	AGPT/T131	64.0	55–75	5.72	8.9
Consistency at 60 °C (Pa.s)	AGPT/T121 (Mould A)	1581.0	700 min	613.9	38.8
Consistency 6% at 60 °C (Pa.s)	AGPT/T121 (Mould A)	700.0	400 min <sup>(1)</sup>	180.8	25.8
Segregation (%)	AGPT/T108	0.8	8 max	0.87	115.5
DSR stress ratio at 10 °C <sup>(2)</sup>	AGPT/T125	2.33	Report <sup>(2)</sup>	0.15	6.6
Conformance	All four samples satisfied the AGPT/T190 limits for tested properties.				

Note 1: The listed consistency 6% at 60 °C limit for S15E as agreed by the BSWG in May 2018.

Note 2: This property has been proposed by Austroads BSWG to be included in AGPT/T190 as a 'report' property when it is next revised.

Table 3.9 shows that the consistency at 60 °C and consistency 6% at 60 °C test results had noticeably high CoV values (i.e. 38.8% and 25.8%, respectively) indicating that the sample-to-sample variation of the S15E binder in terms of these test properties was quite large. The CoV values calculated for viscosity at 165 °C and torsional recovery at 25 °C test results (i.e. 12.4 and 15.8%, respectively) were slightly higher than 10%, but all individual results were well within their respective specification limits. The results obtained in segregation tests showed the highest CoV value (115.5%). As in the case of the results obtained for the A15E and S35E grade binder samples, this high CoV result is probably due to the low results obtained for each S15E grade binder sample in segregation tests (i.e. 0 to 2%).

An insight into the probability that S15E binders (which were sourced from a single supplier) would meet AGPT/T190 specification requirements is given in Table 3.10, where statistical analysis results and specification limits are presented the same way as Table 3.2. The results shown in Table 3.10 indicate that most 95% confidence limits were well within relevant AGPT/T190 specified limits. Although the lower 95% confidence limit obtained for the softening point property (54.6 °C) was slightly lower (0.4 °C) than the AGPT/T190 specified minimum limit of 55 °C, this degree of variation is less than the degree of rounding required by the softening point test method (0.5 °C). Due to this, S15E binders obtained from this supplier would be expected to meet AGPT/T190-specified requirements for softening point on most occasions.

Based on the confidence limit analysis described above, S15E binders (which were obtained from a single supplier in this study) would be expected to comply with AGPT/T190 requirements in terms of the tests performed (if batch results obtained from this supplier were normally distributed).

**Table 3.10: Conformance-probability of S15E binder samples**

Test property	AGPT/T190 requirement (min)	Lower 95% confidence	Mean (& range of test results)	Upper 95% confidence	AGPT/T190 requirement (max)
Viscosity at 165 °C (Pa.s)	–	–	0.446 (0.376–0.495)	0.54	0.55
Torsional recovery at 25 °C (%)	32	32.3	43.5 (38.0–53.0)	54.7	62
Softening point (°C)	55	54.6	64.0 (59.0–72.0)	73.4	75
Consistency 6% at 60 °C (Pa.s)	400 <sup>(1)</sup>	403.5	700.0 (560.0–947.0)	–	–
Segregation (%)	–	–	0.8 (0.0–2.0)	2.2	8.0
DSR stress ratio at 10 °C <sup>(2)</sup>	Report <sup>(2)</sup>	2.07	2.33 (2.11–2.48)	2.58	Report <sup>(2)</sup>

Note 1: The listed consistency 6% at 60 °C limit for S15E was agreed by BSWG in May 2018.

Note 2: This property has been proposed by Austroads BSWG to be included in AGPT/T190 as a 'report' property when it is next revised.

### 3.7 Test results obtained for a 10% crumb rubber binder

Table 3.11 shows the results of AGPT/T190-related tests which were obtained for a single 10% crumb rubber binder sample. This binder sample was subjected to the same tests as performed on other PMB grades except that segregation tests were not performed. Consistency-related tests were conducted on this sample using mould A test conditions as mould B test conditions are only required to be used for AGPT/T190 PMB grades which typically contain low levels of polymer additives (i.e. S10E, S35E and A25E PMB grades).



**Table 3.11: AGPT/T190-related test results obtained for the 10% crumb rubber binder**

Test property	Test method	Test result	AGPT/T190 requirement for an S15RF grade PMB
Viscosity at 165 °C (Pa.s)	AGPT/T111	0.912	–
Torsional recovery at 25 °C (%)	AGPT/T122	35	25 (min)
Softening point (°C)	AGPT/T131	55.5	55 (min)
Consistency at 60 °C (Pa.s)	AGPT/T121 (Mould A)	1008	Report
Consistency 6% at 60 °C (Pa.s)	AGPT/T121 (Mould A)	704	Report <sup>(1)</sup>
DSR stress ratio at 10 °C	AGPT/T125	1.29	–

Note 1: The listed consistency 6% at 60 °C 'report' requirement for S15RF was agreed by BSWG in May 2018.

As this binder type is not specified in AGPT/T190, the test results obtained were compared with the specified properties of an S15RF grade PMB (which nominally is a blend of 15% crumb rubber and C170 bitumen). AGPT/T190 does not require viscosity at 165 °C tests to be performed on S15RF grade binders and consistency 6% at 60 °C test results are proposed to be reported in the future for S15RF grade PMBs. DSR stress ratio at 10 °C tests have not been proposed to be performed on S15RF grade binders in a future version of AGPT/T190, as this binder grade is not currently subjected to low temperature stiffness tests.

The torsional recovery at 25 °C and softening point results obtained for the 10% crumb rubber binder met AGPT/T190 requirements for an S15RF grade PMB even though this binder contained less crumb rubber than the specified PMB grade. The consistency 6% at 60 °C test result obtained for the crumb rubber binder (1008 Pa.s) was somewhat lower than the average results obtained for the A15E, A35P and A10E samples tested (1302 to 1494 Pa.s), but was still higher than the minimum limits proposed for these grades (900–1000 Pa.s). This suggests that this crumb rubber binder would show slightly less to comparable resistance to rutting as A15E, A35P and A10E grade PMBs if it was used in asphalt applications.

The DSR stress ratio at 10 °C result obtained for the crumb rubber binder (1.29) was generally lower than the average results obtained for other elastomeric PMBs. This lower DSR stress ratio result suggests that the 10% crumb rubber binder would be generally less resistant to low temperature cracking in asphalt and sprayed seals than currently specified PMB grades which contain an elastomeric polymer. As the DSR stress ratio at 10 °C result obtained for the crumb rubber was similar to the average result obtained for the A35P grade binder (1.21; refer to Table 3.4), the cracking resistance of this binder if used in road construction would be expected to be similar to a currently specified PMB grade which typically contains a plastomeric polymer.

## 4 OBJECTIVE 2: COMPARISONS BETWEEN AGPT/T190 TEST PROPERTIES AND DSR RHEOLOGICAL PROPERTIES

During the second part of the study, the results obtained for PMB samples in consistency 6% at 60 °C, torsional recovery at 25 °C and softening point tests were compared with various DSR test results which were obtained for the same samples to determine whether there were correlations between Australian specified properties and DSR test properties. Although these specification tests are only required for PMBs in Australia, selected samples of bitumens that were used in the product-conformity work (Section 2) were also subjected to these PMB tests as well as DSR tests. Bitumen samples were included in the comparisons between conventional and DSR test results as this allowed binders with a variety of different visco-elastic properties to be included in the comparison work. Bitumen samples predominantly show viscous behaviour in rheological tests at intermediate to high road temperatures. PMBs can show various levels of viscous and elastic rheological behaviour in rheological tests over a wide range of road temperatures, which are dependent on the amount and type of polymer which is present in the PMB.

Consistency-related tests were conducted on the bitumen samples included in the test comparison work using mould A test conditions. Mould A test conditions were used as the majority of PMB samples in the study were tested using mould A test conditions.

### 4.1 Elastometer consistency 6% at 60 °C vs. DSR complex viscosity

Consistency 6% at 60 °C results were compared with DSR complex viscosity at 60 °C results recorded at 12% strain (DSR  $\eta^*$  12% at 60 °C) in this study to determine whether there was a correlation between AGPT/T190-specified test results and the results obtained for the same binders in a conventional DSR oscillation test. Consistency 6% at 60 °C tests are conducted by subjecting an annular ring of binder to a constant shear rate in one direction during a test. Consistency 6% at 60 °C results are determined at the point where all parts of a sample have been subjected to a strain of 0.06. During a consistency 6% at 60 °C test all parts of a binder sample are subjected to the same strain and strain rate conditions.

DSR complex viscosity results, by contrast, are obtained subjecting thin binder films to sinusoidal oscillation in a DSR where different parts of a binder sample are subjected to different shear rates and levels of strain. A peak strain level of 12% strain was chosen to be used in DSR tests so that binder samples were subjected to a maximum strain of 0.06 at all points that were half the distance between the edge and centre of the DSR spindle. It was initially thought that this level of maximum strain between the edge and centre of DSR samples would approximate the level of strain used in consistency 6% at 60 °C tests.

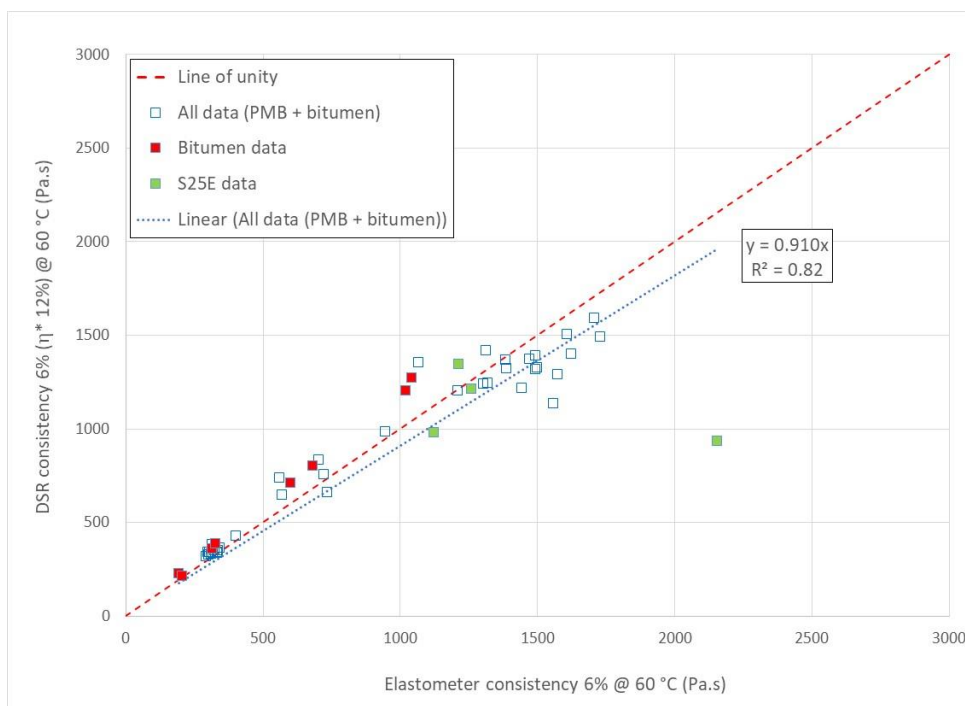
DSR complex viscosity results were obtained using an oscillation frequency 1.67 rad/s for all binder samples which had been subjected to consistency-related tests using mould A test conditions (i.e. all binder samples except samples of the S35E grade binder). This oscillation frequency was chosen to mimic as closely as possible the strain rate of 0.1 s<sup>-1</sup> used when mould A is used to conduct consistency 6% at 60 °C tests. DSR complex viscosity results were obtained for samples of S35E grade PMBs using an oscillation frequency of 5.00 rad/s. This oscillation frequency was chosen to mimic as closely as possible the strain rate of 0.3 s<sup>-1</sup> used when mould B is used to conduct consistency 6% at 60 °C tests. It was noted in Section 3.4 that S35E binder samples were tested using mould B test conditions to meet the requirements of AGPT/T190.

Figure 4.1 shows a plot of DSR  $\eta^*$  12% at 60 °C results versus consistency 6% at 60 °C results for the PMB and bitumen samples which were tested in the P74 project. The figure also includes a

linear fit to the experimental data obtained for these binders as well as a line of unity plot where the results of DSR  $\eta^*$  12% at 60 °C and consistency 6% at 60 °C tests are the same. The datapoints obtained for the bitumen samples subjected to both tests are shaded red in the figure. The datapoints associated with the S25E grade binder samples are shaded green in the figure.

A linear fit to the experimental data yielded an  $R^2$  value of 0.82 indicating that there was a very reasonable correlation between DSR  $\eta^*$  12% at 60 °C and consistency 6% at 60 °C results if all data was considered. The slope of the linear fit (0.91) was also similar to the line of unity (i.e. 1.00). Even though there appeared to be a good correlation between DSR  $\eta^*$  12% at 60 °C and consistency 6% at 60 °C results overall, one data point obtained for an S25E grade PMB (with a consistency 6% at 60 °C result of approximately 2100 Pa.s) appeared to deviate markedly from the overall trend.

Figure 4.1: DSR  $\eta^*$  12% at 60 °C versus Elastometer consistency 6% at 60 °C



In order to investigate whether the result obtained for one of the S25E binder samples was a spurious result, or that it reflected a lack of correlation between DSR  $\eta^*$  12% at 60 °C and consistency 6% at 60 °C results, the results obtained in the P74 project were combined with the results obtained by ARRB when three bitumen samples and five different PMB samples were subjected to the same two tests. A description of the binder samples used in ARRB studies is included in Table 4.1. These binder samples were extensively characterised by the ARRB laboratory during Austroads projects TT1823, TT2037 and APT2063. Each of the samples was found to meet the specified requirements of the listed bitumen or PMB grade when subjected to a wide range of specified tests.

Consistency-related tests on these binders were conducted at the ARRB laboratory using the same test method (i.e. AGPT/T124) as used by the TMR laboratory in the P74 project. All tests were conducted using mould A test conditions. DSR  $\eta^*$  12% at 60 °C results were obtained using ARRB’s DSR using the same test conditions as used by the TMR laboratory in the P74 project.

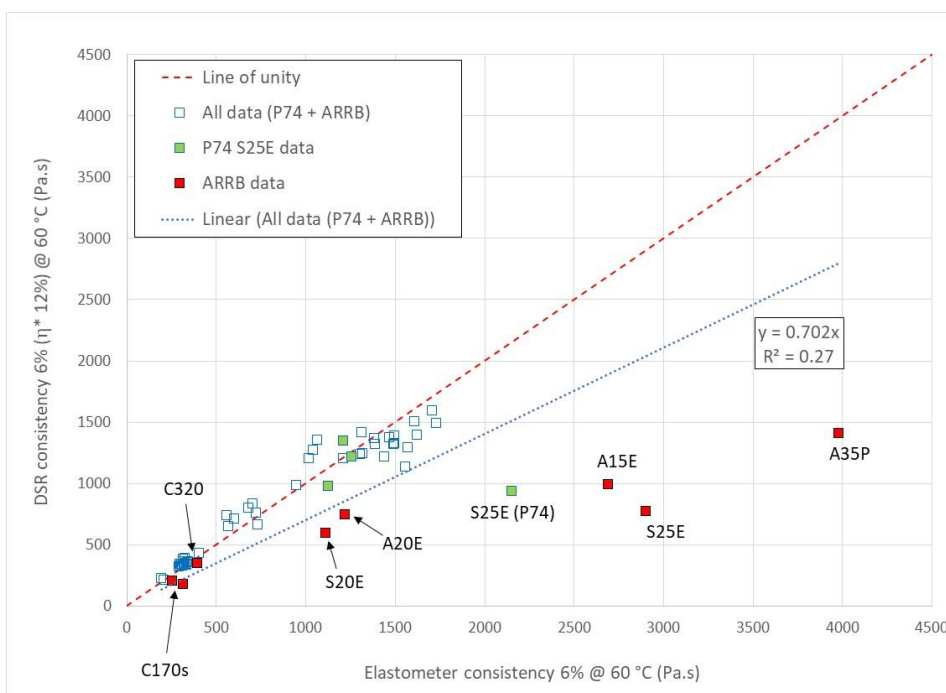
Table 4.1: Binder samples used in ARRB studies

Binder sample	Description
C170-A	C170 class bitumen used in Austroads APT2063: <i>Key properties of cutters for optimal sprayed seal performance</i>
C170-B	C170 class bitumen used in Austroads TT2037: <i>Development of a sprayed seal binder cracking test</i>
C320	C320 class bitumen used in Austroads TT2037: <i>Development of a sprayed seal binder cracking test</i>
A15E	A15E grade binder used in Austroads TT2037: <i>Development of a sprayed seal binder cracking test</i>
A20E	A20E grade binder used in Austroads TT2037: <i>Development of a sprayed seal binder cracking test</i>
S25E	S25E grade binder used in Austroads TT2037: <i>Development of a sprayed seal binder cracking test</i>
S20E	S20E grade binder used in Austroads TT2037: <i>Development of a sprayed seal binder cracking test</i>
A35P	A35P grade binder used in Austroads TT1823: <i>Optimising binder performance</i>

Figure 4.2 shows a plot of DSR  $\eta^*$  12% at 60 °C results versus consistency 6% at 60 °C results for all samples which were tested in the P74 project as well as by ARRB. The datapoints obtained by the ARRB laboratory are shaded red in the figure, while those obtained for S25E grade PMBs in the P74 project are shaded green in the figure. The datapoint associated with the S25E binder in the P74 project which did not follow the general trend has been labelled in the figure as 'S25E (P74)'. The binder grades associated with the bitumen and PMB samples which were tested by ARRB have also been labelled in the figure.

Figure 4.2 also includes a linear fit to the experimental data which was obtained for the combined P74/ARRB dataset as well as a line of unity plot where the results of DSR  $\eta^*$  12% at 60 °C and consistency 6% at 60 °C tests are the same. A linear fit to all experimental data yielded a very poor  $R^2$  value of 0.27. The slope of the linear fit (0.72) also deviated markedly from the line of unity (1.00). Based on the results shown in Figure 4.2 there no longer appears to be a good correlation between DSR  $\eta^*$  12% at 60 °C and consistency 6% at 60 °C results if binders tested outside of the P74 project are included in the dataset.

Figure 4.2: DSR  $\eta^*$  12% at 60 °C versus Elastometer consistency 6% at 60 °C for the combined P74/ARRB dataset



It is worthy of note that the datapoints associated with the C170 and C320 bitumen samples that were tested at ARRB appear to correlate well with the results obtained in the P74 project. Although the PMB samples tested at ARRB deviate from the general trend observed in the P74 project, the trend observed in these test results does not appear to be inconsistent with the result obtained for S25E binder which was tested in the P74 project which did not follow the general trend. Due to this, the datapoint labelled 'S25E (P74)' in Figure 4.2 does not appear to be a spurious result. The results obtained for this binder in the P74 project therefore appear to actually reflect the properties of this particular binder.

The most likely explanation for the deviation of some experimental results from the general trend is that the rheological properties of these binders are more sensitive to shear rate than the other binders studied. It was noted earlier that the shear rate and strain experienced by a binder sample during a consistency 6% at 60 °C test is constant throughout a sample. This, however, is not the case when conventional DSR oscillation tests are performed. The results included in Section 4.1 therefore appear to indicate that the DSR tests need to be performed in a way which better mimics the constant strain and shear rate conditions of a consistency 6% at 60 °C test for better correlations to be observed between DSR test results and consistency 6% at 60 °C results for all binder types.

As the current study indicated that conventional DSR oscillation tests are not able to sufficiently simulate the rheological conditions which occur during consistency 6% at 60 °C tests, two possible options for better matching the results of DSR tests with consistency 6% at 60 °C tests have been proposed below:

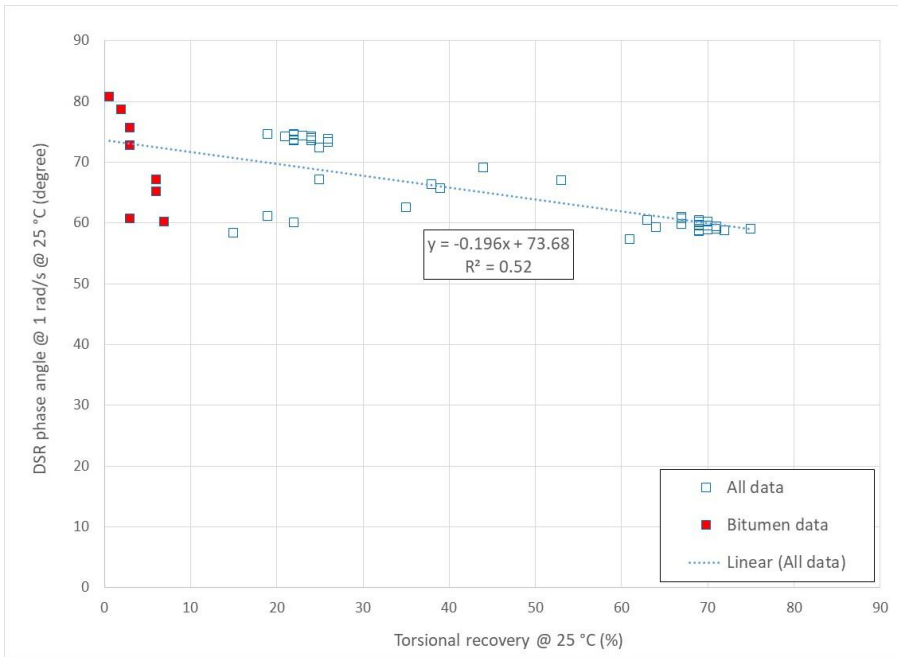
- The variation in shear rate that occurs during a conventional DSR oscillation could be reduced by conducting a test where a sample is sheared in one direction only (rather than oscillating in opposite directions). DSR stress ratio at 10 °C tests (AGPT/T125) are conducted by shearing samples in the DSR using this mode.
- During a conventional DSR test, the sections of sample which are at different radii from the centre of the DSR experience different shear rates during testing. This variation in shear rate can be reduced by using a cone-and-plate DSR spindle rather than a conventional parallel-plate DSR spindle.

## 4.2 Torsional recovery at 25 °C vs. DSR phase angle properties

Torsional recovery at 25 °C results (which give an indication of the elastic properties of a binder) were firstly compared with DSR phase angle ( $\delta$ ) results obtained at 25 °C in temperature-frequency sweep tests as the value of  $\delta$  obtained during these tests also gives an indication of the elastic properties of a binder. As in the case of the test comparison work described in Section 4.1, the results obtained in DSR tests at 25 °C were initially compared with torsional recovery at 25 °C results to ensure the temperatures of both tests were matched. It is well known that the rheological properties of binders change with test temperature.

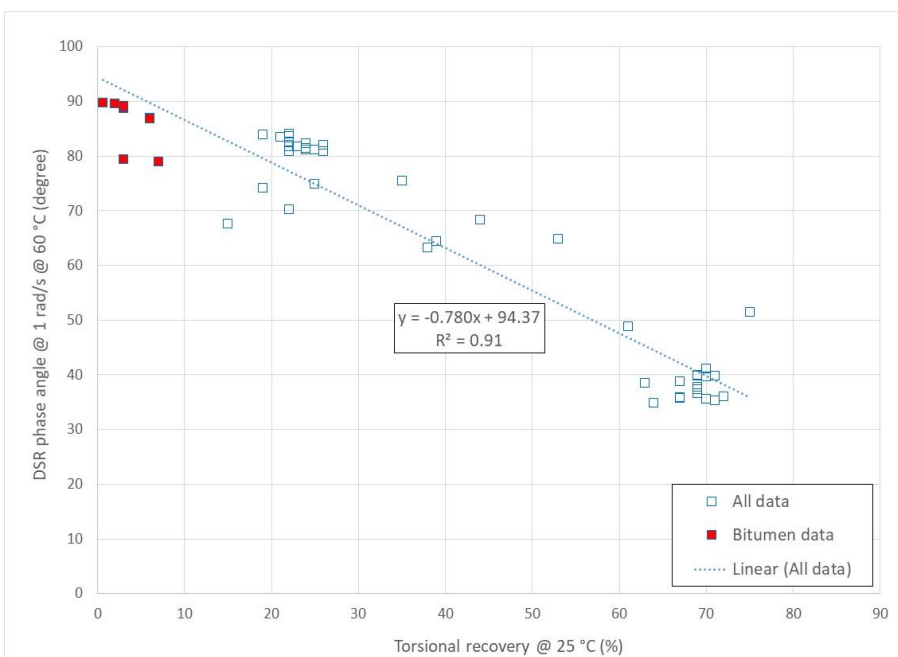
Figure 4.3 shows a plot of DSR  $\delta$  at 25 °C results versus torsional recovery at 25 °C results obtained for the bitumen and PMB samples tested in the P74 study. DSR  $\delta$  at 25 °C results were calculated by averaging the  $\delta$  results obtained at 20 °C and 30 °C when an oscillation frequency of 1 rad/s was used during temperature-frequency sweep tests. The datapoints associated with the bitumen samples tested are shaded red in the figure, while those associated with the PMB samples are unshaded. The linear fit shown in Figure 4.3 indicates that there was not a good correlation between DSR  $\delta$  results and torsional recovery results when results obtained in each of the two tests were compared at the same test temperature. The  $R^2$  value obtained from the fit was quite low (0.52). This lack of correlation is probably due to the samples being tested using different rheological conditions in the two tests.

Figure 4.3: DSR  $\delta$  at 25 °C versus torsional recovery at 25 °C



Torsional recovery at 25 °C results were then compared with DSR  $\delta$  at 60 °C results in order to determine if changing the temperature of the DSR test had any effect on the correlation between the two parameters. These  $\delta$  results were obtained at 60 °C when an oscillation frequency of 1 rad/s was used during temperature-frequency sweep tests. A plot comparing these two properties is presented in Figure 4.4. The datapoints associated with the bitumen samples tested are shaded red in this graph. A linear fit to the experimental results obtained for all binders yielded a very reasonable  $R^2$  value of 0.91 (Figure 4.4). This result was somewhat unexpected as it implied that there was a better correlation between DSR tests and torsional recovery tests if the experiments were performed at different temperatures.

Figure 4.4: DSR  $\delta$  at 60 °C versus torsional recovery at 25 °C



The observed correlation between DSR  $\delta$  at 60 °C results and torsional recovery at 25 °C results could be explained by varying contributions from the bitumen and polymer in the PMB samples when tests are conducted using different strain levels and different test temperatures. DSR temperature-frequency sweep tests are low strain tests whereas torsional recovery at 25 °C tests are relatively high strain tests. It is known that the  $\delta$  results obtained for elastomeric PMBs in DSR temperature-sweep tests are predominantly governed by the elastic properties of the polymer at high temperatures. This is because the bitumen in these types of PMBs behaves as a predominantly viscous material at high temperatures, whereas the polymer in the PMBs retains its elastic properties up to reasonably high temperatures (e.g. 90 °C). In low strain tests that are conducted at low temperatures (e.g. 25 °C), the bitumen in the PMB behaves as an elastic material and thus can mask the effect of polymer.

In high strain tests (such as torsional recovery at 25 °C tests) the bitumen component in a PMB does not contribute significantly to the overall elasticity of the binder. The results of torsional recovery at 25 °C tests therefore are most influenced by the elastic properties of the polymer in the PMB (in a similar way to  $\delta$  results obtained at 60 °C in low strain temperature-frequency sweep tests). Therefore, the observed correlation shown in Figure 4.4 is likely due to both tests predominantly measuring the elastic properties of the polymer in the PMBs.

Although a correlation was observed between DSR  $\delta$  at 60 °C results and torsional recovery at 25 °C for the P74 project binders, an ideal DSR test which could be used to replace the torsional recovery at 25 °C test would be conducted at the same test temperature. This test would preferably be performed by subjecting binder samples to a high strain in the DSR and then allowing the samples to recover to mimic the rheological conditions of the current specification test. Equivalent elastic recovery results could then be calculated in a similar way to that described in AGPT/T122. This type of test was not included in the study, as the project scope only included low strain DSR temperature-frequency sweep tests.

### 4.3 Softening point vs. DSR properties at 60 °C

Softening point results that were obtained in the P74 study were compared with both complex viscosity at 60 °C ( $\eta^*$ , Pa.s) and phase angle at 60 °C ( $\delta$ , degree) results which were obtained for the same samples in DSR temperature-frequency sweep tests. Softening point tests were compared with these two different DSR test parameters as they are high temperature tests which would be affected by both the viscous and elastic properties of a binder. Complex viscosity results are influenced by both the viscous and elastic properties of a binder, while phase angle results are good indicator of the elasticity of the binder at low strain. As softening point tests are not conducted at a single test temperature, it was not possible to select a specific temperature in DSR tests which would match the temperature used in softening point tests.

Complex viscosity results were calculated from the results obtained in temperature-sweep tests after fitting the data to a master curve function (Section 1). DSR  $\delta$  results were obtained at 60 °C when an oscillation frequency of 1 rad/s was used during temperature-frequency sweep tests.

Figure 4.5 shows a plot of DSR  $\eta^*$  at 60 °C results versus softening point test results obtained for the bitumen and PMB samples tested in this study. Figure 4.6 shows another plot of DSR  $\delta$  at 60 °C results versus softening point test results obtained for the same binders. The datapoints associated with the bitumen samples tested are shaded red in the figures, while those associated with the PMB samples are unshaded.



Figure 4.5: DSR  $\eta^*$  at 60 °C versus softening point

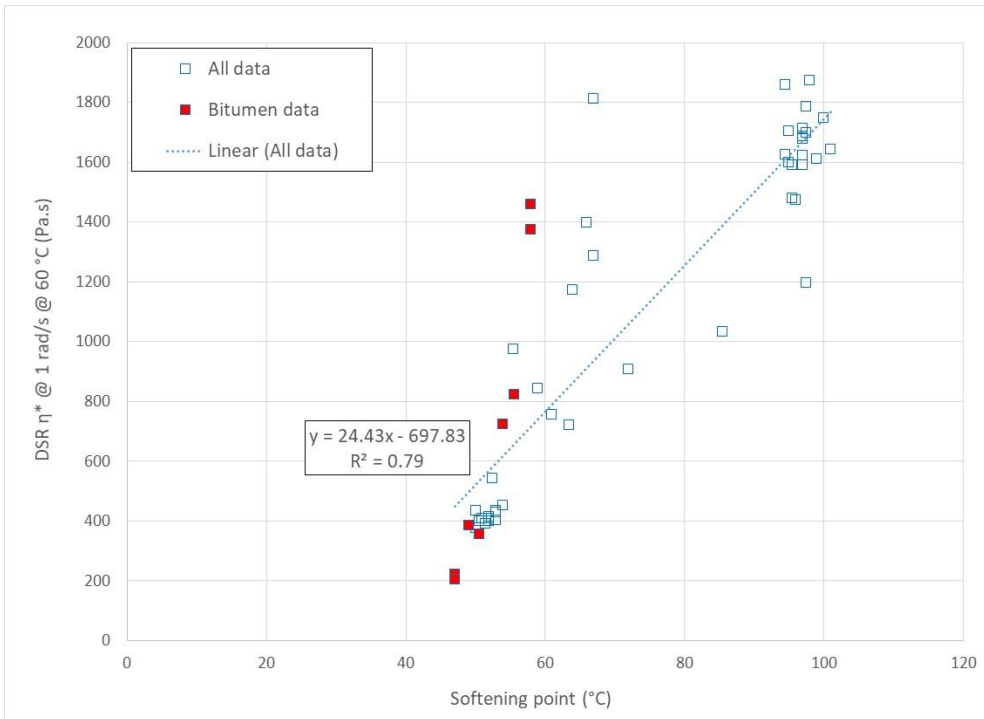


Figure 4.6: DSR  $\delta$  at 60 °C versus softening point

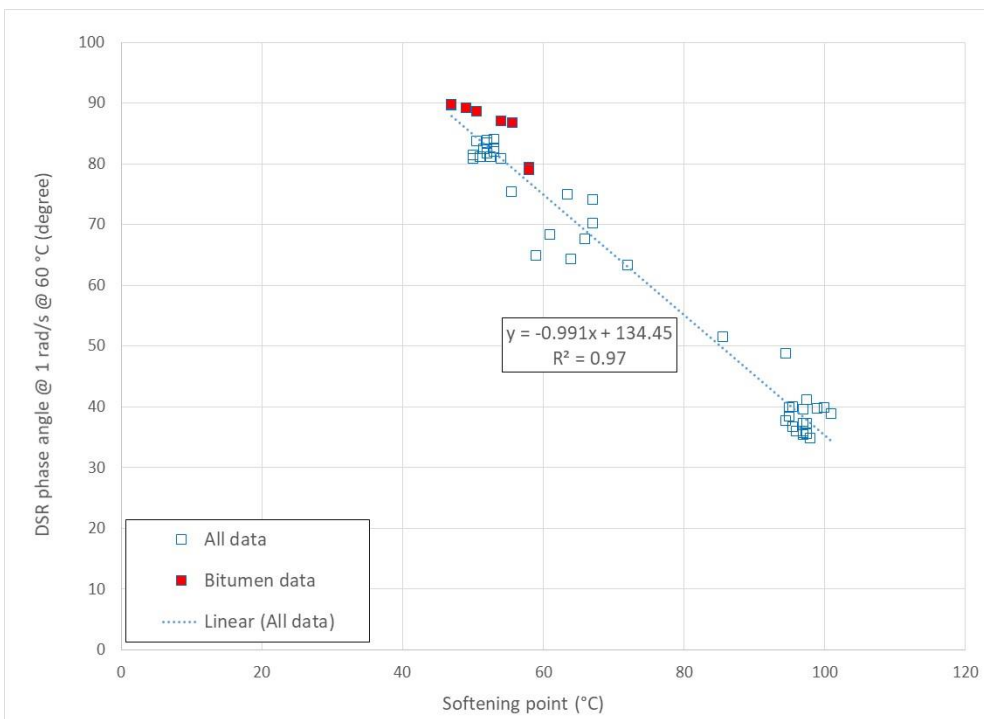


Figure 4.5 and Figure 4.6 also include linear fits to respective experimental data and the values of  $R^2$  obtained from the fits. The  $R^2$  values obtained for the two fits yielded reasonable (0.79) to very good (0.97) correlations indicating that the softening point results were affected by both the viscous and elastic properties of the binders. The higher value of  $R^2$  shown in Figure 4.6 suggests that softening point results are more sensitive to the elastic properties of the binders.



Although a very good correlation was observed between DSR  $\delta$  at 60 °C results and softening point results, these DSR tests do not exactly replicate the rheological or temperature conditions of a softening point test. As softening point values obtained for PMB samples used in this study ranged from about 50 to 100 °C, testing at higher temperatures (e.g. 75 °C ) may be able to mimic softening point testing better. It is also noted that other DSR parameters, namely the storage modulus ( $G'$ ) and the 'imaginary viscosity ( $\eta''$ )' which can also be obtained from conventional oscillation tests, may be better related to the softening point property (from the rheological perspective) than the  $\delta$  property trialled for this study.

Another factor that may be influencing the observed correlations between DSR  $\delta$  at 60 °C results, and both torsional recovery at 25 °C and softening point results, is the limited number of binder samples/grades which were included in the P74 study. It was noted in Section 4.1 that good correlations were observed between DSR  $\eta^*$  12% at 60 °C and consistency 6% at 60°C results for nearly all P74 binders tested, except for one binder sample that deviated significantly from the overall trend. This correlation, however, was found to no longer hold if additional PMB samples were included in the dataset.

Further work could involve testing other samples/grades of PMBs in the future to determine if the observed correlations still hold if additional binder samples are included. Other DSR tests, which are known to better simulate the rheological conditions of current AGPT/T190 tests as discussed in the relevant sections above, could also be considered as they would be expected to be more applicable to any type of PMB that was tested.

## 5 SUMMARY AND CONCLUSIONS

A comprehensive database of binder test properties was established during Years 1 and 2 of NACOE P74 project (2017-19). This database consisted of test results obtained for various grades of bitumens and PMBs which were sourced from a number of different suppliers in Queensland. The first part of investigation analysed the results obtained in AS 2008- and AGPT/T190-related tests in order to evaluate the variability and conformance in quality-control and performance-related properties of the supplied binders. The second part of investigation compared the results obtained for binders in three AGPT/T190 tests with the results obtained for the binders after they were tested in the DSR.

### 5.1 Variability/conformance in specification test properties

All C170, C320, C600 and M1000 bitumen samples which were tested in the study met the relevant specified requirements of AS 2008 in terms of the tests that were performed. The outcomes of a statistical analysis indicated that C170, C320 and C600 bitumens obtained from the Queensland suppliers used in this study would be expected to conform to AS 2008-specified requirements with a high level of probability. A statistical analysis of the results obtained for the M1000 samples indicated that M1000 binders, which were obtained from a single supplier in the study, may fail specified post-RTFO penetration at 25 °C test requirements on some occasions.

The A10E, S35E, S25E and S15E grade PMB samples that were tested in the study all met relevant AGPT/T190 specification requirements in terms of the tests that were performed. All 14 A15E binder samples that were tested met AGPT/T190 requirements except for one binder sample which failed the requirements of the consistency at 60 °C test. The four A35P binder samples tested in the study all failed segregation test requirements indicating that the polymer in these binders would be susceptible to polymer segregation during hot storage. The single sample of 10% crumb rubber obtained for this study conformed to the specified requirements for an S15RF grade PMB, even though its nominal rubber content was less than the typical rubber content of that grade.

The results obtained in the study indicated that A15E and S35E grade PMBs showed a relatively small variation in test results when several samples of these binder grades were tested. The test results obtained for the S25E and S15E binders showed a slightly greater amount of sample-to-sample variation. The samples of A35P binder that were tested showed the widest degree of sample-to-sample variation in terms of several different properties. This could reflect the instability of the polymer in these PMBs. As A35P grade PMBs are traditionally produced using an EVA polymer, which is known to be miscible with bitumen, the segregation results obtained for these binders suggest that another type of polymer, other than EVA, may be present in the PMBs.

A statistical analysis of the results obtained for the PMB samples indicated that A15E, A10E, S35E and S15E binders (which were obtained from the suppliers included in the study) would be expected to conform to AGPT/T190 requirements with a high level of probability. S25E binders which were obtained from the suppliers included in the study may fail softening point and proposed consistency 6% at 60 °C requirements on some occasions. All A35P samples tested failed segregation test requirements.

### 5.2 Specification test properties vs. DSR rheological properties

The results obtained for the bitumen and PMB samples in three tests included in AGPT/T190 (i.e. consistency 6% at 60 °C, torsional recovery at 25 °C and softening point tests) were compared to the test results which were obtained for the same binder samples using the DSR in order to determine if there were correlations between AGPT/T190-specified properties and DSR test results.

A good correlation was found between DSR  $\eta^*$  12% at 60 °C results and consistency 6% at 60 °C results for the binders included in the P74 study, except for one binder sample that deviated significantly from the overall trend. This correlation, however, was found to no longer hold if eight additional binders which were tested at ARRB laboratory were included in the dataset. This result appears to indicate that the test conditions in a DSR test need to better simulate the rheological conditions which occur during consistency 6% at 60 °C test, so that a relationship between DSR test results and consistency 6% at 60 °C results can be found for all types of binder samples that could be tested.

A good correlation was found between DSR phase angle ( $\delta$ ) at 60 °C results obtained in temperature-frequency sweep tests and torsional recovery at 25 °C test results, and also between DSR  $\delta$  at 60 °C results and softening point results. Correlations were found for these results for the P74 dataset of binders even though tests were performed using both different temperatures and different rheological conditions. Based on the results obtained during the consistency 6% at 60 °C correlation work however, it is not known if observed correlations between AGPT/T190 test properties and DSR  $\delta$  at 60 °C results are an artefact of the limited number of binder samples/grades tested in the P74 project. These correlations could be confirmed if more binder samples were tested. Other DSR tests, which are known to better simulate the rheological conditions of current AGPT/T190 tests as discussed in the relevant sections in this report, could also be considered as they would be expected to be more applicable to any type of PMB that was tested.