Louisiana Transportation Research Center

Final Report 594

Sustainable Materials for Pavement Infrastructure: Design and Performance of Asphalt Mixtures Containing Recycled Asphalt Shingles

by

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LIST OF TABLES

(Figure 19 continued)

Procedure C

- Determine specific gravities, gradations, RAS % asphalt content, consensus properties for virgin aggregate and RAS;
- Determine aggregate composite blend;
- Superheat virgin aggregate to 195°C (minimum) for 3 hours;
- Heat mixing bucket to 163°C;
- Place room temperature RAS on the bottom of the heated mixing bucket and the superheated virgin aggregate place on top of the RAS;
- Mix superheated virgin aggregate and RAS together;
- Place blended aggregate into 163°C oven till the blended aggregates reach suitable temperature for mixing with asphalt binder;
- Blend asphalt binder and blended aggregates together in heated mixing bucket;
- Short-term age the asphalt mixture; AASHTO R30
- Determine volumetrics and densification criteria, AASHTO M323 & R35 *[40]*, *[87]*

Procedure D

- Determine specific gravities, gradations, RAS % asphalt content, consensus properties for virgin aggregate and RAS;
- Determine aggregate composite blend;
- Superheat virgin aggregate to 195°C (minimum) for 3 hours;
- Heat mixing bucket to 163°C;
- Place room temperature RAS on the bottom of the heated mixing bucket and the superheated virgin aggregate place on top of the RAS;
- Mix superheated virgin aggregate and RAS together;
- Place blended aggregate into 163°C oven till the blended aggregates reach suitable temperature for mixing with asphalt binder;
- Blend soft asphalt binder (softening agent) and blended aggregates together in heated mixing bucket;
- Short-term age the asphalt mixture; AASHTO R30
- Determine volumetrics and densification criteria, AASHTO M323 & R35 *[40]*, *[87]*

(Figure 19 continued)

Procedure E

- Determine specific gravities, gradations, RAS % asphalt content, consensus properties for virgin aggregate and RAS;
- Determine aggregate composite blend;
- Heat RAS to 163°C, then mix with softening agent in heated mixing bowl. After mixing put RAS back in oven at 163°C for 30 minutes;
- Superheat virgin aggregate to 195°C (minimum) for 3 hours;
- Heat mixing bucket to 163°C;
- Place RAS on the bottom of the heated mixing bucket and the superheated virgin aggregate place on top of the RAS;
- Mix superheated virgin aggregate and RAS together;
- Place blended aggregate and RAS into 163°C oven till the blended aggregates reach suitable temperature for mixing with asphalt binder;
- Blend asphalt binder and blended aggregates together in heated mixing bucket;
- Short-term age the asphalt mixture; AASHTO R30
- Determine volumetrics and densification criteria, AASHTO M323 & R35 *[40], [87]*

Procedure F

- Determine specific gravities, gradations, RAP & RAS % asphalt content, consensus properties for virgin aggregate and RAP & RAS;
- Determine aggregate composite blend;
- Add 5% moisture content to RAP;
- Superheat virgin aggregate to 195°C (minimum) for 3 hours;
- Heat mixing bucket to 163°C;
- Place moisture laden RAP on the bottom of the heated mixing bucket, then the RAS (at room temperature) on top of the RAP and then superheated virgin aggregate place on top of the RAP and RAS;
- Mix superheated virgin aggregate and RAP & RAS together;
- Place blended aggregate into 163°C oven till the blended aggregates reach suitable temperature for mixing with asphalt binder;
- Blend asphalt binder and blended aggregates together in heated mixing bucket;
- Short-term age the asphalt mixture; AASHTO R30
- Determine volumetrics and densification criteria, AASHTO M323 & R35 *[40], [87]*

(Figure 19 continued)

Procedure G

- Determine specific gravities, gradations, RAP & RAS % asphalt content, consensus properties for virgin aggregate and RAP and RAS;
- Determine aggregate composite blend;
- Add 5% moisture content to RAP;
- Heat RAS to 163°C, then mix with RAs in heated mixing bowl. After mixing put RAS back in oven at 163°C for 30 minutes;
- Superheat virgin aggregate to 195°C (minimum) for 3 hours;
- Heat mixing bucket to 163°C;
- Place moisture laden RAP on the bottom of the heated mixing bucket, then the RAS on top of the RAP and then superheated virgin aggregate place on top of the RAP and RAS;
- Mix superheated virgin aggregate and RAP & RAS together;
- Place blended aggregate into 163°C oven till the blended aggregates reach suitable temperature for mixing with asphalt binder;
- Blend asphalt binder and blended aggregates together in heated mixing bucket;
- Short-term age the asphalt mixture; AASHTO R30
- Determine volumetrics and densification criteria, AASHTO M323 & R35 *[40]*, *[87]*

Procedure H

- Determine specific gravities, gradations, RAP & RAS % asphalt content, consensus properties for virgin aggregate and RAP & RAS;
- Determine aggregate composite blend;
- Add 5% moisture content to RAP;
- Superheat virgin aggregate to 195°C (minimum) for 3 hours;
- Heat mixing bucket to 163°C;
- Place moisture laden RAP on the bottom of the heated mixing bucket, then the RAS (at room temperature) on top of the RAP and then superheated virgin aggregate place on top of the RAP and RAS;
- Mix superheated virgin aggregate and RAP & RAS together;
- Place blended aggregate into 163°C oven till the blended aggregates reach suitable temperature for mixing with asphalt binder;
- Blend soft asphalt binder (softening agent) and blended aggregates together in heated mixing bucket;
- Short-term age the asphalt mixture; AASHTO R30
- Determine volumetrics and densification criteria, AASHTO M323 & R35 *[40]*, *[87]*

Asphalt Binder Test Results

The asphalt binder's rheological properties have an effect on the performance of the asphalt mixture pavement. Changes in the asphalt binders' rheological properties due to production and aging, which result from oxidation and environmental influences, must be addressed to reduce asphalt binder related pavement distresses such as raveling, cracking, stripping, and rutting. It is essential that the asphalt binders are tested to assure that the binder rheology meets specified criteria necessary to reduce pavement distresses due to changes of its rheological properties as a result of aging. Therefore, asphalt binders and extracted asphalt binder from mixtures were conducted to characterize an asphalt binder's rheology, which is necessary to minimize the ACs contribution to durability, high temperature (permanent deformation), intermediate temperature (fatigue) cracking, and low temperature (thermal) cracking performance.

Asphalt Binder Performance Grading

The binder from compacted mixtures was extracted and recovered according to AASHTO T319, "Standard Method of Test for Quantitative Extraction and Recovery of Asphalt Binder from Asphalt Mixtures" using trichloroethylene (TCE) as the solvent agent *[89]*. All of the binders presented in this study were graded according to AASHTO R29, "Grading or Verifying the Performance Grade of an Asphalt Binder" and AASHTO M320, "Standard Specification for Performance-Graded Asphalt Binder *[48]*, *[90]*." The actual PG grading is presented in Table 16. As shown in Table 16, the extracted binders were stiffer than their virgin counterparts. Comparison between the extracted control mixture (no RAS and/or RAP) (70CO) and the neat binder (PG70-22) show this trend. This can be explained by the short-term aging effects of mix/compaction heating and extraction heating. Binders extracted from the 70PG5PHG15RAP mixture showed the highest stiffness which corresponds with this mixture having the highest RBR, 41.5%. Comparing mixture 70PG5P with 70PG5P5HG, Table 16 indicates that the use of Hydrogreen adversely affected both the high temperature and low temperature PG properties. The low temperature PG performance was also adversely affected from the use of asphalt flux as shown in the comparison of extracted asphalt binder from mixture 70PG5P20FLUX. Whereas, binders from the 70PG5P and 70PG5P12CYCL had similar results. In comparison with the extracted asphalt cement binders from 52PG5P and 70PG5P, Table 16 indicated an improvement in low temperature properties from the use of soft asphalt binder. From Table 16, it is indicated that the use of 15% RAP with no RAs (70PG15RAP) as compared to the 70CO extracted asphalt binder showed no effect to the high temperature and low temperature grade. However, when 5% PCWS is included in the design (70PG5P15RAP), the extracted binder results as compared to the extracted 70PG15RAP asphalt binder showed a significant increase (4-PG grades) in the high temperature properties and decreases the low temperature property by 2-PG grades. It is shown that the use of Hydrogreen did not improve the high temperature and low temperature properties of the extracted asphalt binders utilizing PCWS and RAP. It is noted that the PCWS continuous PG Grading and designated PG grading is not shown in Table 16. An attempt was made to characterize the extracted binders from the PCWS however due to limitations of the DSR high temperature range (120°C) this was not possible. The extracted PCWS asphalt binder far exceeded the limitations of the DSR. Also the low-temperature capabilities of the bending beam rheometer (BBR) could not properly grade the post-consumer waste shingles.

Table 16
Asphalt binder PG characterization

	PG70-22	PG76-22	Asphalt Flux	RAP	70CO
Continuous PG Grading	73.5 - 22.9	77.7 - 22.7	32.3-46.6	99.3 - 16.2	80.7 - 22.0
PG Grading	70 -22	76 - 22	28 - 46	94 - 16	76 - 22
	70PG5P	70PG5M	70PG5P5HG	70PG5P12CYCL	70PG5P20FLUX
Continuous PG Grading	85.1 - 19.3	90.7 - 16.7	91.4 - 15.0	82.2 - 21.6	86.1 - 11.6
PG Grading	82 - 16	88 - 16	88 - 10	82 - 16	82 - 10
	70PG15RAP	70PG5P15RAP	70PG5PHG15RAP	52PG5P	52PG5P15RAP
Continuous PG Grading	80.7 - 22.0	104.1 -12.9	105.1 - 1.0	68.3 - 24.2	74.1 - 22.0
PG Grading	76 - 22	100 - 10	100 - 0	64 - 22	70 - 22

Rotational Viscosity

This test was conducted in accordance with AASHTO T 316-06, "Standard Method of Test for Viscosity Determination of Asphalt Binder Using Rotational Viscometer," for determining the viscosity of the asphalt binder at 135°C *[51]*. The test purpose of the Rotational Viscometer (RV) was to measure the binder properties at high construction temperatures to assure pumping and mixing during production. In addition, an asphalt binder's viscosity can also influence workability, which is the ability of the asphalt mixtures being placed and compacted with reasonable effort. Figure 20 indicates that the viscosity of the asphalt binders with the addition of RAP and/or RAS, with and without RAs generally had higher viscosities than the original virgin binder (PG70-22M). The asphalt mixture designated as 52PG5P had a lower viscosity than PG70-22M. When comparing the extracted asphalt binder viscosities of the mixtures evaluated to the conventional mixture, 70CO, extracted asphalt binder it is shown that all mixtures had higher viscosities with the exception of the two asphalt mixtures utilizing the soft asphalt binder, 52PG5P and 52PG5P15RAP respectively. Figure 20 illustrates that the 70PG5PHG15RAP asphalt mixture resulting rotational viscosity was unmeasurable (too stiff at 135°C) because of equipment limitations. A display of "EEEEE" on the digital viscometer appeared when testing this material. This display means the reading is over-range and the percent torque readings exceeded 100%. For the extracted asphalt binder for mixture 70PG5PHG15RAP, this error occurred at an approximate viscosity of 11 Pa·s. It is shown that the extracted binders from 70PG5M and 70PG5P5HG had the highest measured rotational viscosity, 4.04 Pa·s and 3.99 Pa·s, respectively.

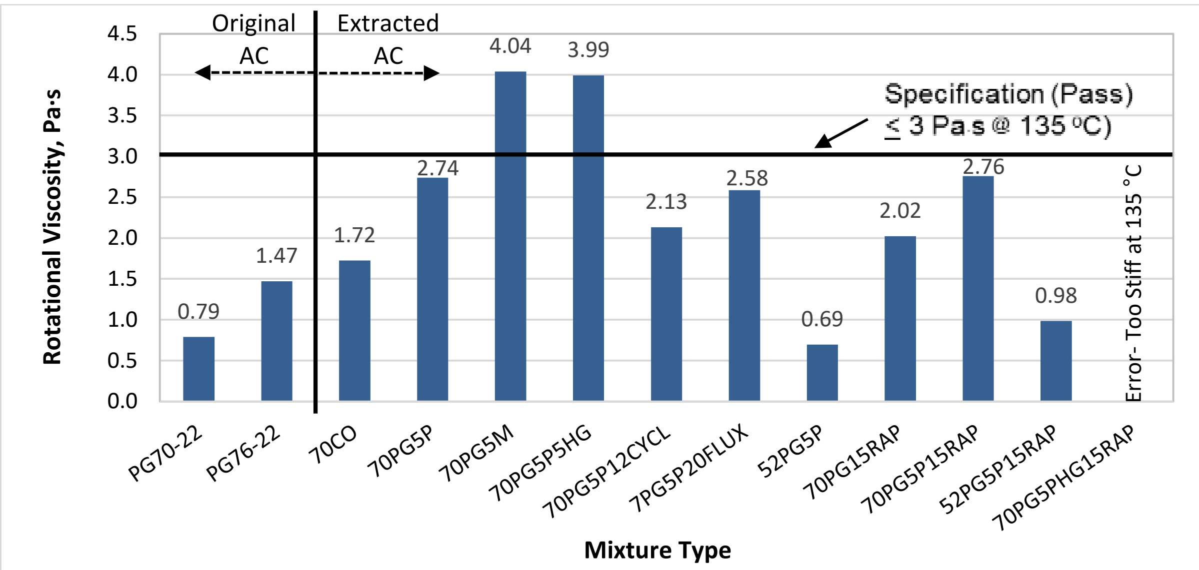


Figure 20
Viscosity of asphalt binders used in various types of mixtures

Linear Amplitude Sweep Test Results

This test was performed in accordance with AASHTO TP 101 at a testing temperature of 25°C *[57]*. The purpose of the LAS test is to evaluate an asphalt binder's ability to resist fatigue damage under cyclic loading by increasing the strain amplitudes to accelerate damage. The rate of damage accumulation is used to indicate fatigue performance. The extracted asphalt binders from the asphalt mixtures were considered as short-term aged since the asphalt mixtures were short-term aged during blending. The extracted binders were then long-term aged in accordance with AASHTO R 28 *[50]*. The extracted asphalt binders were then tested under PAV conditions. In this test, the greater the number of cycles to failure indicates an asphalt binder's resistance to fatigue damage.

Table 17 indicates N_f, the number of cycles to failure, from the LAS test. It is shown that there are two applied strains, 2.5% and 5.0%. These strain levels were chosen because the 2.5% applied strain is for strong pavements and 5.0% applied strain for weak pavements according to Hintz et al. *[63]*. It is shown that the extracted asphalt binder from mixture 70PG5PHG15RAP has the lowest N_f at both strain levels, which corresponds to an observation that the 70PG5PHG15RAP mixture has the highest RBR of 41.5%. Also, it is presented that both extracted asphalt binders from mixtures containing Hydrogreen (70PG5PHG15RAP and 70PG5P5HG) had the lowest N_f at both strain levels. In comparing the extracted asphalt binders from the 70CO mixture to the extracted binders from 70PG5P and 70PG5M, it is shown that both extracted asphalt binders utilizing MWS and PCWS had higher number of cycles to failure than the control at 2.5% strain level. It is indicated in Table 17 that the extracted asphalt binder from 70PG5M had a higher N_f than did the 70PG5P extracted binder. This would be expected since the PCWS are more aged due to oxidation than MWS. Table 17 presents that the use of recycling agents (Cyclogen-L, PG52-28 soft asphalt binder, and asphalt flux) generally increased the number of cycles to failure with the exception of Hydrogreen.

Table 17
LAS number of cycles to failure

	N_f, Number of Cycles to Failure	
Mixture Type	2.5% Applied Strain	5.0% Applied Strain
70PG5PHG15RAP	24733	223
70PG5P5HG	43936	1015
70CO	65481	3435
70PG5P15RAP	67121	1338
70PG15RAP	74975	3427
70PG5P	78762	2695
70PG5M	82790	2763
70PG5P12CYCL	106213	5016
52PG5P	106213	6934
52PG5P15RAP	112113	4924
70PG5P20FLUX	117114	3074

Christensen-Anderson Model, Glover-Rowe Parameter, and ΔTc Test Results

Table 18 presents the critical stiffness and m-value temperatures as measured by the bending beam rheometer, BBR. The critical temperature is the temperature at which the stiffness and the m-value meets specification of 300 MPa and 0.300 respectively. It is noted that these critical temperatures are independent of each other. As the aging takes place, in general, the m-value of asphalt binders changes at a much faster rate than the stiffness does. At a point during the aging, the material will change from S-controlled (stiffness controlled) to m-controlled. Also shown in Table 18 is the ΔTc, which indicates the difference between the critical stiffness temperature and the m-value critical temperature. Asphalt binders that are m-controlled have negative values, while S-controlled have positive values. All of the asphalt binders evaluated in this study clearly show that they are m-controlled, which would show the aging effects they were subjected to during the evaluations. The range of ΔTc was from -3.5 to -26.3°C. The extracted binder from the mixture 70PG5PHG15RAP had the largest difference as shown by ΔTc, followed by the 70PG5P20FLUX. The significance in this test that as the ΔTc increases oxidation increases which results in loss of mixture durability.

Table 18
ΔT_c from bending beam rheometer test results

	BBR				
	Stiffness, S		Relaxation Rate, m		
	Stiffness, MPa	$T_{c(S)}$, (°C)	m-value	$T_{c(m)}$, (°C)	ΔT_c (°C)
70CO	300	-25.5	0.300	-22.0	-3.5
70PG15RAP	300	-30.3	0.300	-22.0	-8.3
70PG5P12CYCL	300	-30.4	0.300	-21.6	-8.8
70PG5P	300	-29.7	0.300	-19.3	-10.4
70PG5P5HG	300	-25.7	0.300	-15.0	-10.7
70PG5M	300	-28.2	0.300	-16.7	-11.5
70PG5P15RAP	300	-28.2	0.300	-12.9	-15.3
52PG5P15RAP	300	-37.5	0.300	-22.0	-15.5
52PG5P	300	-44.0	0.300	-24.2	-19.8
70PG5P20FLUX	300	-35.6	0.300	-11.6	-24.0
70PG5PHG15RAP	300	-27.3	0.300	-1.0	-26.3

Figure 21 presents a comparison chart of the Glover-Rowe intermediate temperature cracking criteria and the conventional Superpave (i.e., G*sinδ) criterion at the intermediate temperature of 25°C. This figure is a damage curve in black space where the complex shear modulus, G*, at 25°C is plotted on the ordinate axis and the corresponding phase angle is plotted on the abscissa axis. Two Glover-Rowe parameter thresholds were proposed by Anderson et al.; one where non-load associated cracking begins and another when the cracking becomes significant *[65]*. These thresholds are respectively set at 180 kPa for the onset of cracking and 450 kPa for significant cracking, respectively. A total of 22 G* and phase angle (δ) data pairs of 11 asphalt binders measured by DSR tests at intermediate temperatures are plotted on this black space diagram. According to the data presented in Figure 21, 8 samples failed the G*sinδ criterion, while 12 samples failed the Glover-Rowe significant cracking criterion and 16 samples failed the cracking on-set criterion, respectively. In other words, 36.4, 54.5, and 72.7% of samples failed G* sinδ, Glover-Rowe significant cracking, and Glover-Rowe cracking on-set criteria, respectively. The observed disagreements between the conventional G*sinδ criterion and the two Glover-Rowe criteria may be in-line with a common belief that the G*sinδ parameter does not adequately capture the asphalt binder's susceptibility to the intermediate temperature cracking *[65]*.

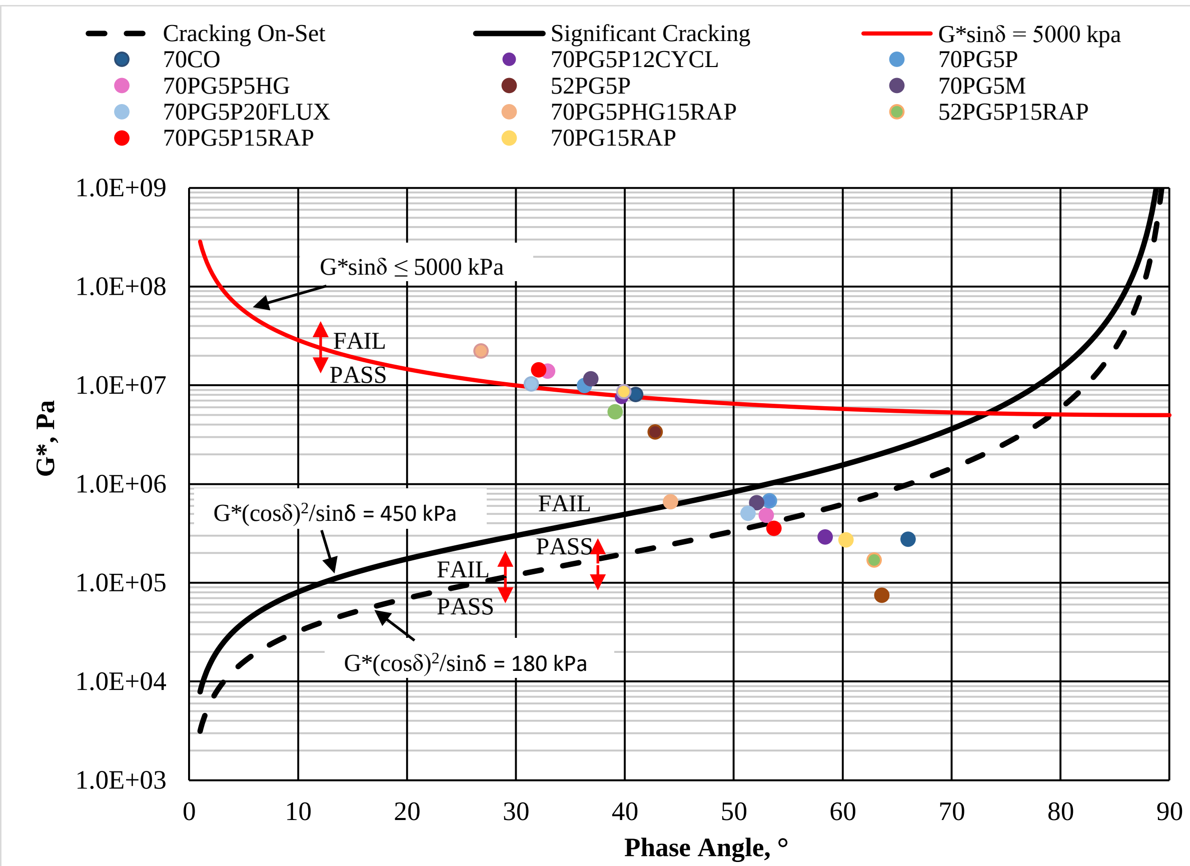


Figure 21
Comparison of conventional superpave and Glover-Rowe failure criteria

Figure 22 indicates the correlation between ΔTc and the rheological Index, R. Anderson et al. chose two parameters that relate ductility and the loss of flexibility with aging, ΔTc and R

[65]. As the asphalt binder becomes more m-controlled, the ductility of the asphalt binder decreases, which adversely affects an asphalt binder's capability to relax under loading and resist fracture. This is indicated by the ΔTc. Likewise, as the asphalt binder ages, there is an increase in the rheological index, R. It is shown in Figure 22 that there is a high correlation between ΔTc and R. As the rheological index increases, the ΔTc also increases.

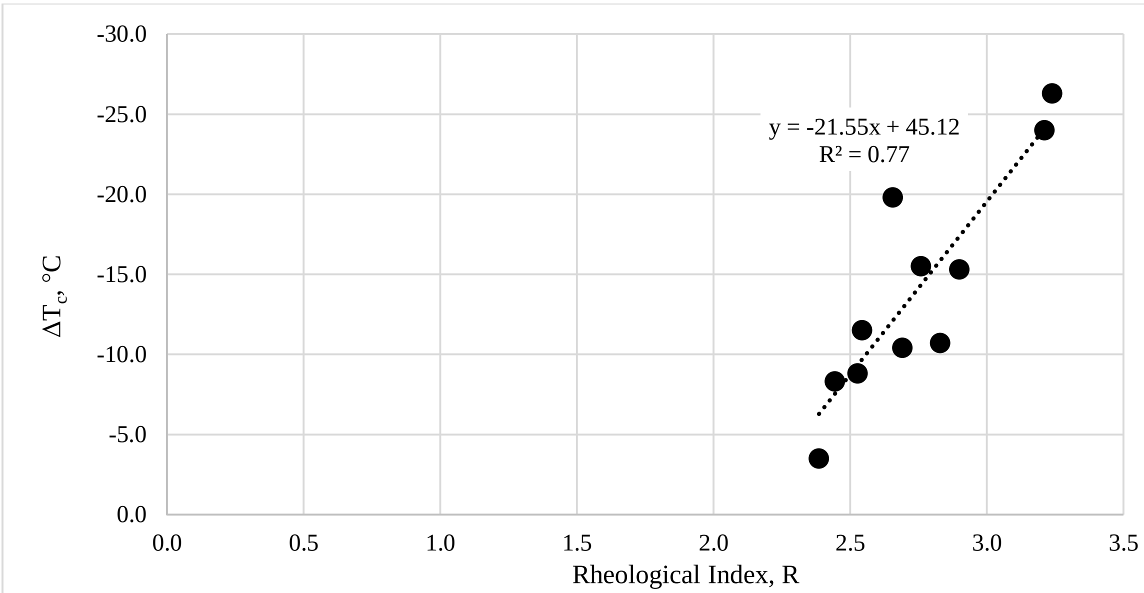


Figure 22
ΔT_c vs. R

Figure 23 illustrates the rheological index as it relates to mixture type. Also indicated is the recycle binder ratio, RBR, for each mixture. It is shown that asphalt mixture 70PG5PHG15RAP has the greatest R value of 3.24. It is also the asphalt mixture having the largest RBR (i.e., aged asphalt from recycled material within the asphalt mixture) of 41.5%. In addition, the conventional asphalt mixture, 70CO, containing no recycled binders had the lowest rheological index of 2.39, which indicates it is less aged than all mixtures evaluated in this study.

Figure 24 shows the correlation between ΔTc and RBR. This figure indicates that there is a trend between these parameters for the mixtures evaluated. It is indicated that as the RBR increases the ΔTc parameter increases. This would seem logical since the change in critical temperatures between stiffness and relaxation is representative of aging. Thus, the more aged binder one has in the asphalt mixture the greater the difference in ΔTc.

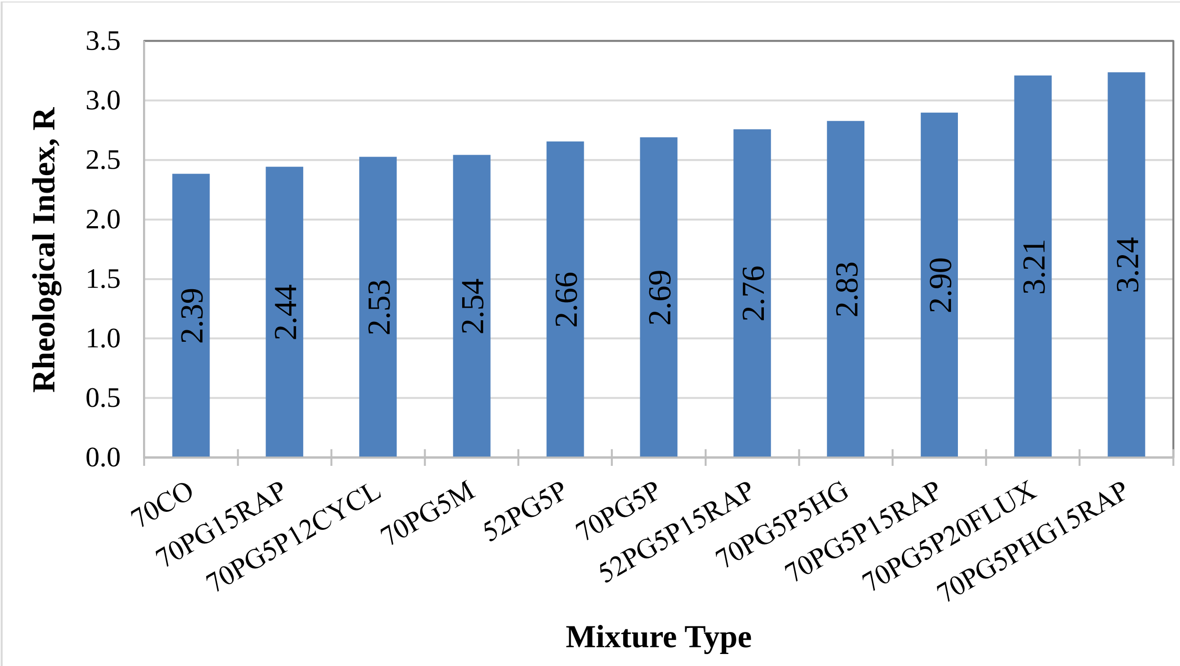


Figure 23
R vs. asphalt mixture type

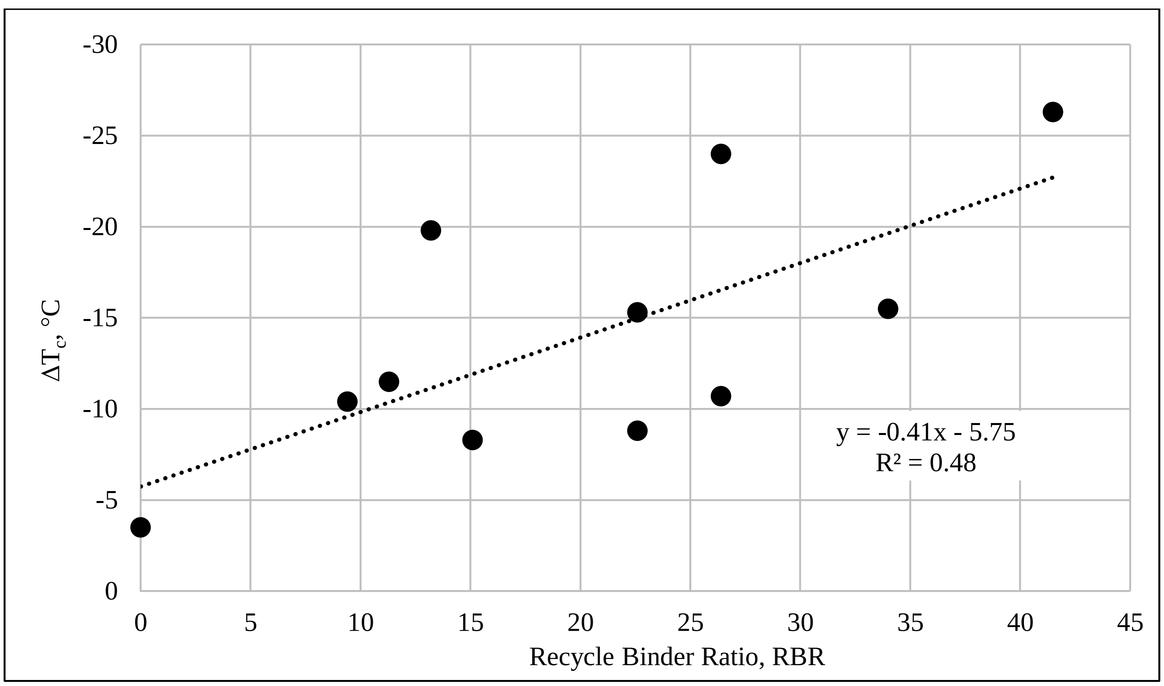


Figure 24
ΔT_c vs. RBR

Complex Shear Modulus, G*, Test Results

Figure 25 presents the complex shear modulus (G*) from the extracted binders at various test temperatures and frequencies for the 11 asphalt mixtures evaluated in this study. It is shown that the 70PG5PHG15RAP mixture had the highest G* values and the 52PG5P mixture had the lowest G* values at the low frequency range, which was expected because the 52PG5P mixture utilized the softest virgin binder. Whereas, the high stiffness results for the 70PG5PHG15RAP are due to 100% of the available aged RAS and RAP binder being utilized in this mixture. Also, there appears to be four groupings. The first grouping is mixture 70PG5PHG15RAP, which has the highest recycle binder ratio. The second grouping is comprised of mixtures 70PG5P5HG, 70PG5P, 70PG5M, 70PG5P20FLUX, and 70PG5P15RAP.

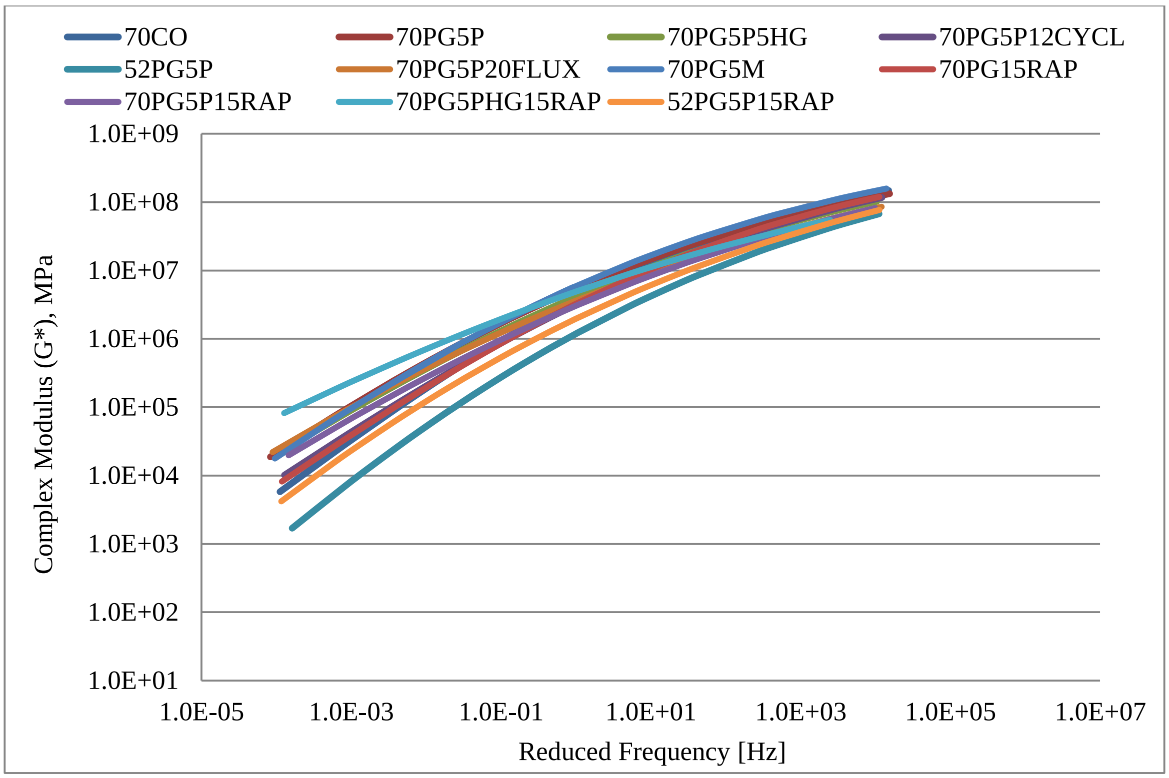


Figure 25
Dynamic shear rheometer test result

The remaining mixtures are grouped together with the exception of mixture 52PG5P. It can also be observed (Figure 26) that the low frequency part of the curves, which correspond to the higher testing temperatures, followed the same trend observed in the E* results obtained during the asphalt mixture testing. It is presented in Figure 25 that the 70PG5PHG15RAP asphalt mixture had the highest stiffness at the lower frequency. This would be indicative of a rut resistant mixture. Likewise, the 52PG5P mixture had the lowest stiffness at the lower frequency which could indicate a mixture’s propensity to rutting. It is shown in Figure 25 that all mixtures converge at the highest frequency which represents the low temperature response of the asphalt cement binders. It is indicated that the 52PG5P had the lowest stiffness at the highest frequency, which is indicative of a mixtures resistance to low temperature cracking.

Multiple Stress Creep Recovery Test Results

The multiple stress creep recovery (MSCR) test was conducted in accordance with AASHTO TP 70-13 to evaluate the effects of mixtures containing RAS and/or RAP, with and without recycling agents as compared to the control mixture containing no RAP, RAS, or recycling agents on rutting resistance *[56]*. This test was introduced to characterize the binder rutting resistance at high temperatures. D'Angelo et al. reported that the MSCR test parameters correlate well with mixture rutting performance as measured by accelerated pavement testing *[91]*.

As shown in Table 19, the increase in RAP and RAS content was associated with an increase in the percentage recovery and a decrease in the non-recoverable creep compliance. These are desirable characteristics as it would decrease the rutting susceptibility of the binders. Asphalt binders (virgin and extracted) were tested at 67°C, which is Louisiana's PG high temperature grade. Triplicate specimens were analyzed.

Table 19
MSCR test results

Extracted Binders from Mixtures	MSCR		
	$J_{nr3.2}$ @ 67°C, kPa^{-1}	%J_{nr} diff	% Recovery
70CO	0.51	16.6	35.07
70PG5P	0.23	13.1	43.25
70PG5M	0.10	7.1	53.85
70PG5P5HG	0.13	13.2	50.26
70PG5P12CYCL	0.33	17.2	39.39
52PG5P	3.37	22.1	21.33
70PG5P20FLUX	0.16	13.2	47.59
70PG15RAP	0.49	25.2	35.44
70PG5P15RAP	0.01	1.1	98.75
70PG5PHG15RAP	0.01	0.9	98.75
52PG5P15RAP	1.49	23.5	26.44

Asphalt Mixture Experiment Results

Several laboratory tests were conducted and evaluated to measure the performance characteristics of the asphalt mixtures considered in this study. The pavement performance characteristics were analyzed for the asphalt mixtures durability as measured by the Loaded Wheel Tracking Test (Hamburg Type) in terms of moisture sensitivity and permanent deformation, rutting. The asphalt mixtures performance in terms of resistance to fatigue cracking was evaluated from results obtained from the Semi-Circular Bend (SCB) and Dynamic Modulus (i.e., fatigue factor, $E^*\sin\delta$) tests. Furthermore, Dynamic Modulus (i.e., rutting factor, $E^*/\sin\delta$) and Loaded Wheel Tracking Test (Hamburg Type) was used to determine the mixtures' resistance to permanent deformation. Triplicate samples were prepared and tested for each laboratory test. To measure the low temperature performance characteristics (thermal cracking), the TSRST was conducted on the asphalt mixtures studied. The detailed analysis for these test results is included in the following sections of this chapter.

Laboratory Performance Tests

Dynamic Modulus (E*) Test Results. The purpose of the Dynamic Modulus test is to evaluate the viscoelastic response characteristics of asphalt mixtures over a given range of temperatures and frequencies. Figure 28 presents the dynamic modulus ($|E^*|$) at 5 test temperatures (-10, 4.4, 25, 37.8, and 54.4°C) and 6 frequencies (25, 10, 5, 1, 0.5, and 0.1 Hertz) for the 11 asphalt mixtures evaluated in this study. It is shown that the 70PG5PHG15RAP mixture had the highest $|E^*|$ values and the 52PG5P mixture had the lowest $|E^*|$ values. It was expected that the 52PG5P mixture would have the lowest stiffness as measured by $|E^*|$ because of the very soft virgin binder utilized in this mixture. Further, the high stiffness results for the mixture 70PG5PHG15RAP are attributable to the 100% of the available aged RAS and RAP binder being included in this mixture. Also there appears to be four mixture groupings. The first mixture grouping is mixture 70PG5PHG15RAP, which has the highest recycle binder ratio. This mixture also had the highest extracted binder stiffness as measured by the dynamic shear rheometer. The extracted asphalt binder from this mixture was approximately 270% higher than the others binders measured. The second mixture grouping is comprised of mixtures 70PG5P5HG and 70PG5P15RAP. The remaining mixtures are ranked together with the exception of mixture 52PG5P. It is worth noting that the low frequency part of the curves, which correspond to the higher testing temperatures, followed the same trend observed in the G* results from the PG grading of extracted binders; see Figure 25.

Figure 27 shows the mean phase angle results vs. the mean Dynamic Modulus values for all 11 asphalt mixtures considered in this study. This figure shows the phase angle for all materials increases with an increase in temperature and a decrease in frequency. Then the phase angle peaks, followed by a decline as the temperature increases further and the frequency continually decreases.

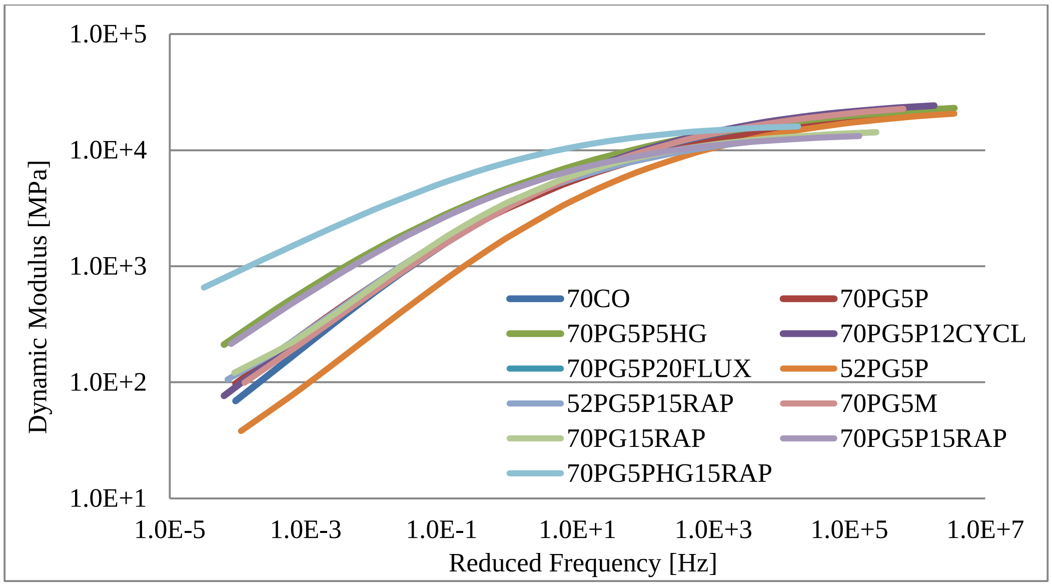


Figure 26
Dynamic modulus (|E*|) vs. reduced frequency

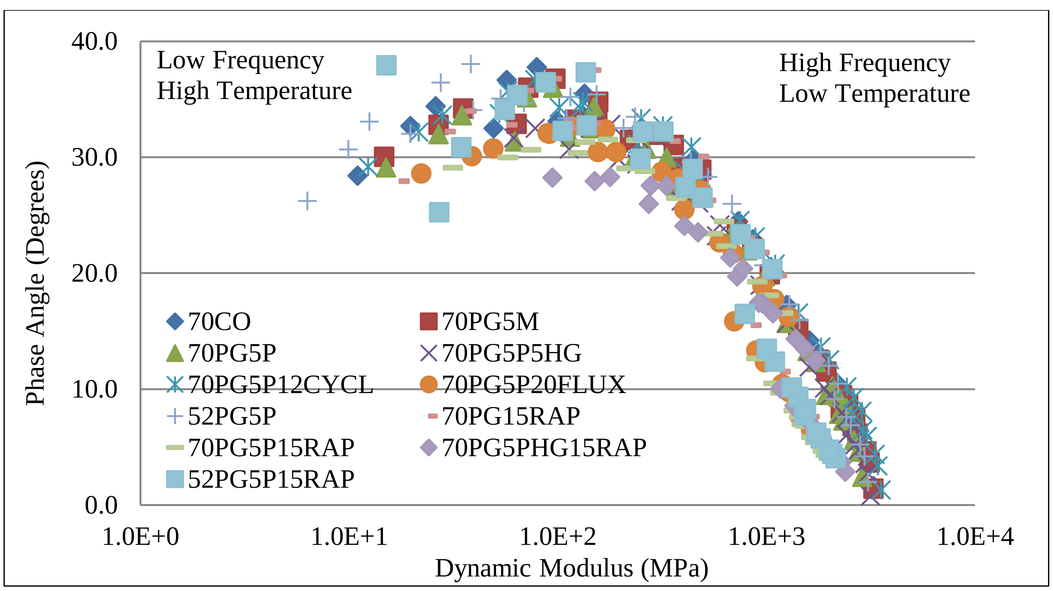


Figure 27
Phase angle vs. dynamic modulus (|E*|) relationship

Figure 28 indicates a normalized comparison to the control mixture (70CO) for all asphalt mixtures evaluated in this study based on the dynamic modulus |E*| at 5 test temperatures (-10, 4.4, 25, 37.8, and 54.4°C) and 6 frequencies (25, 10, 5, 1, 0.5, and 0.1 Hertz). For the purpose of comparison, the E* values calculated at various test temperatures and frequencies for the 70CO asphalt mixture was considered as the unit value (i.e., E* = 1.0).

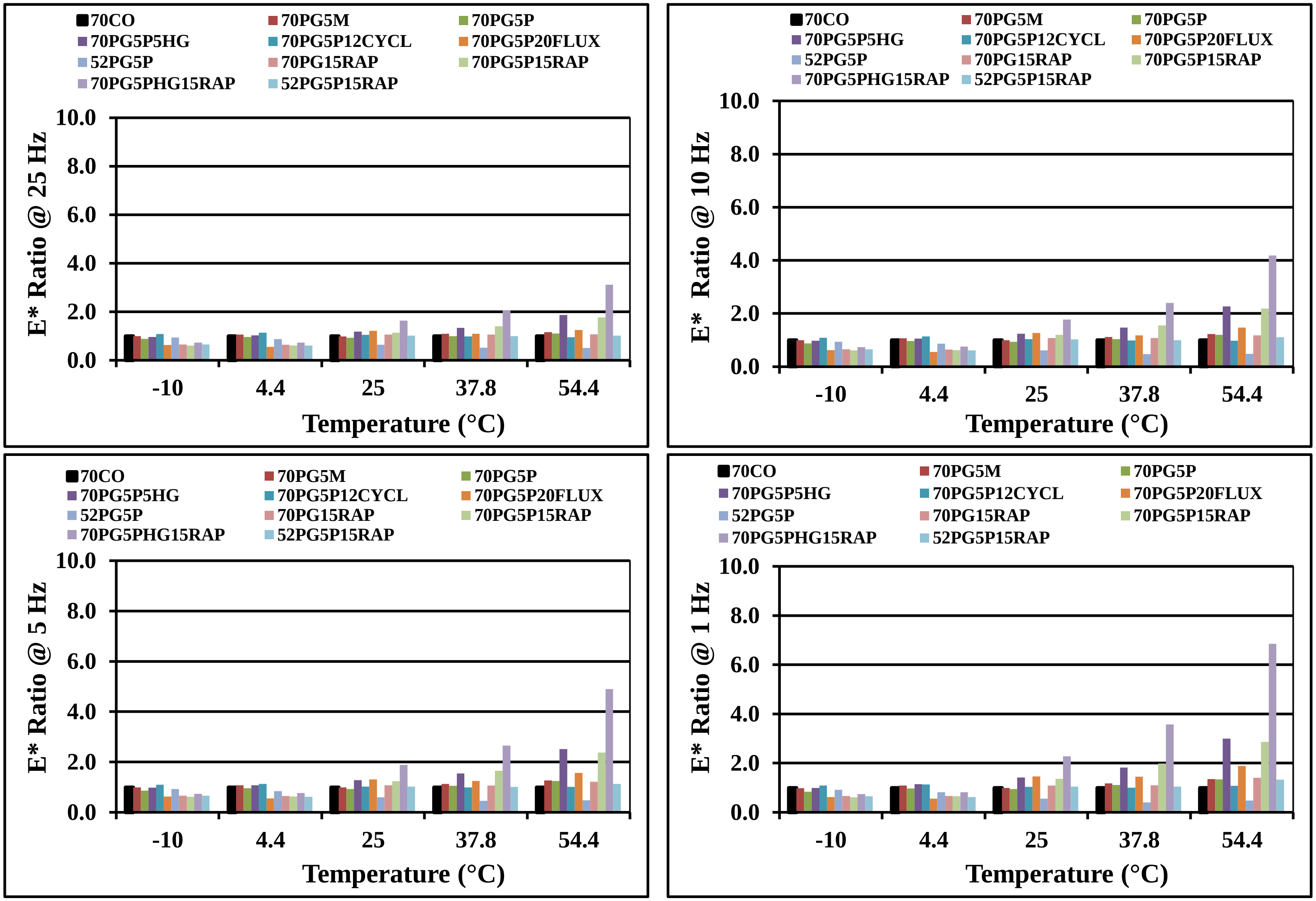
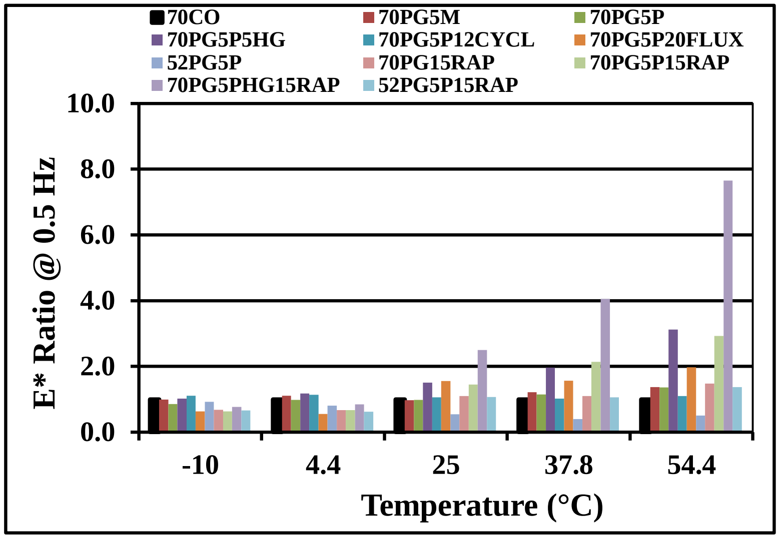


Figure 28
Dynamic modulus ratio comparison (E* Ratio)

(Figure 28 continued)



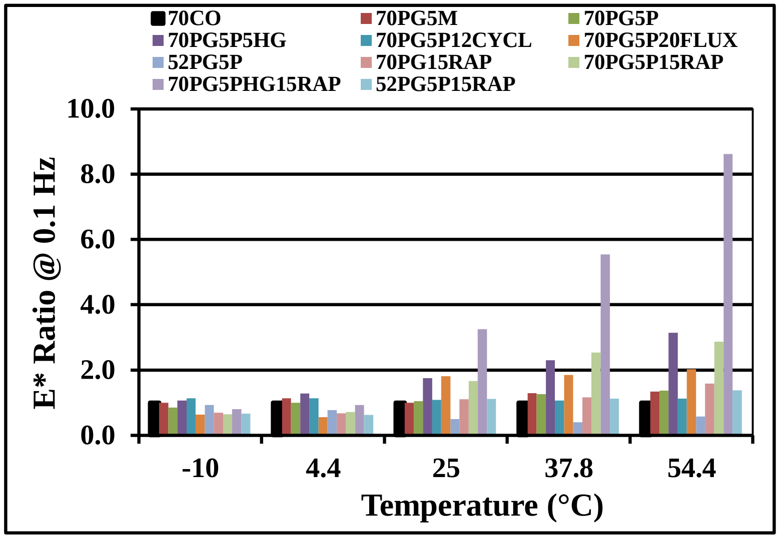


Figure 28
Dynamic modulus ratio comparison (E* Ratio)

To illustrate this concept, the E* values for the 70PG5P and 70CO mixtures at 54.4°C and 5 Hz were 71.2 ksi and 57.4 ksi, respectively. The comparative E* ratio is 71.2/57.4 = 1.2. Any mixtures exhibiting an E* ratio greater than 1.0 has greater stiffness than the 70CO mixture. For high temperature and low frequency, it is important to have an E* ratio greater than 1.0 because this is indicative of a mixtures propensity to resist rutting. Likewise, it is advantageous to have an E* ratio less than 1.0 at the other extreme (low temperature and high frequency). These mixtures have the potential to resist low temperature cracking. Mixture 70PG5PHG15RAP exhibited the highest stiffness at the temperatures of 25°C, 37.8°C, and 54.4°C for all frequencies. Figure 28 illustrates that the mixtures containing RAS and/or RAP, and PG 52-28 asphalt binder generally had lower stiffness than 70CO for temperatures at or below 25°C for all frequencies. In addition, generally the mixtures containing MWS (70PG5M) and PCWS (70PG5P) without recycling agents had comparable stiffness with 70CO for all temperatures and frequencies.

To determine a mixtures resistance to fatigue cracking, a parameter termed fatigue factor is calculated from dynamic modulus test results at a test temperature of 25ºC and a loading frequency of 5 Hz for this study *[92]*. The fatigue factor is computed as E*sinδ, where δ is the phase angle at the selected temperature and frequency *[92]*. For a mixture to resist fatigue cracking, its corresponding E* value should be lower as well as the phase angle at the in-service temperature of 25 ºC. A lower fatigue factor value indicates a better resistance to fatigue cracking.

Figure 29 shows the fatigue factor values for all mixture types and statistical analysis evaluated in this study. There are three distinct groups as shown in Figure 29. Figure 29 indicates that there is a statistical difference between mixtures 52PG5P, 70PG5M, and 70PG5P (Group A) and mixture 70PG5PHG15RAP (Group B). The remaining mixtures (Group A/B) indicate that there is no clear-cut statistical difference between Groups A and B since their mean values are close to both groupings. Statistically, the grouping of mixtures 52PG5P, 70PG5M, and 70PG5P (as shown in Figure 29) are best in fatigue cracking resistance of the 11 mixtures evaluated in this study. Mixture 70PG5PHG15RAP exhibited the highest fatigue factor values; therefore it is the least resistant to fatigue cracking. This can be contributed to this mixture having the highest RBR, the stiffest complex shear modulus (G*), and the highest dynamic modulus (E*). The remaining mixtures (Group A/B) indicate that there is no clear-cut statistical difference between Groups A and B since their mean values is close to both groupings.

An asphalt mixture’s propensity to resist permanent deformation (rutting) can be characterized by using the dynamic modulus test results from various temperatures and frequency. The rutting factor is defined as E*/sinδ, where δ is the phase angle, at a particular temperature and frequency. A loading frequency of 5Hz and test temperature of 54.4ºC was used for computation of the rutting factor, E*/sinδ in this study *[92]*. For mixtures to be rut resistant and exhibit higher stiffness necessitates a higher E* value and a lower phase angle. The higher the rut factor value indicates a mixture greater resistance to permanent deformation.

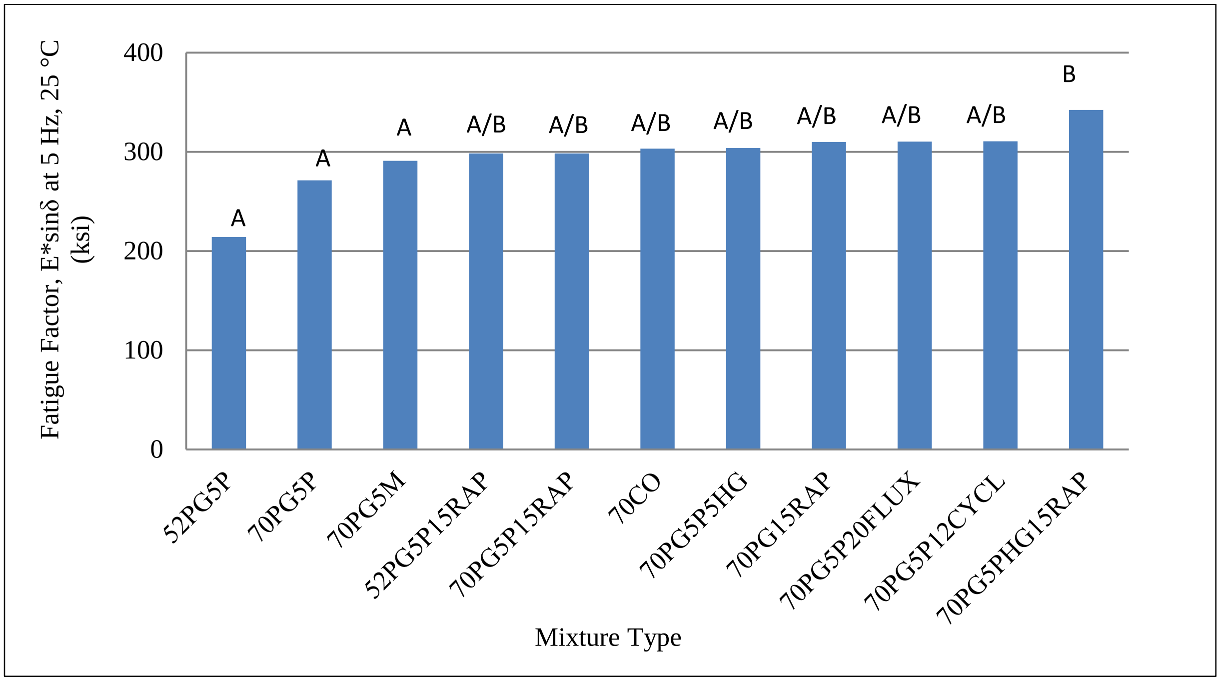


Figure 29
Dynamic modulus test result – fatigue factor

Figure 30 shows the rutting factor values and statistical analysis for all mix types evaluated in this study. Figure 30 indicates that there are five statistical groupings. It clearly shows that the 70PG5PHG15RAP mixture has the greatest resistance to rutting followed by 70PG5P5HG and 70PG5P15RAP. Mixture 70PG5PHG15RAP's resistance to rutting can be contributed to this mixture having the highest RBR, the stiffest binder complex shear modulus (G*), and the stiffest dynamic modulus (E*). Mixtures having the least resistance to permanent deformation are 52PG5P, 70CO, 70PG5P12CYCL, and 52PG5P15RAP. It is noted that there is a grouping of similar results for the 70PG15RAP, 70PG5M, 70PG5P, and 70PG5P20FLUX asphalt mixture types.

Figure 30 indicates that there is a statistical difference between mixture 70PG5PHG15RAP (Group A) and all other mixtures studied. Group A has the highest rut factor and is the most resistant to rutting. Five mixtures are grouped in the "B" and "B/C/D," 70PG5P5HG, 70PG5P15RAP, 70PG5P20FLUX, 70PG5P, and 70PG5M, respectively. There are no discernable statistical difference in this grouping. The remaining mixtures (C/D and D) are the least resistant to rutting because they have the lowest rut factor value. Statistically, the grouping of mixtures 52PG5P, 70PG5M, and 70PG5P (as shown in Figure 29) are best in fatigue cracking resistance of the 11 mixtures evaluated in this study.

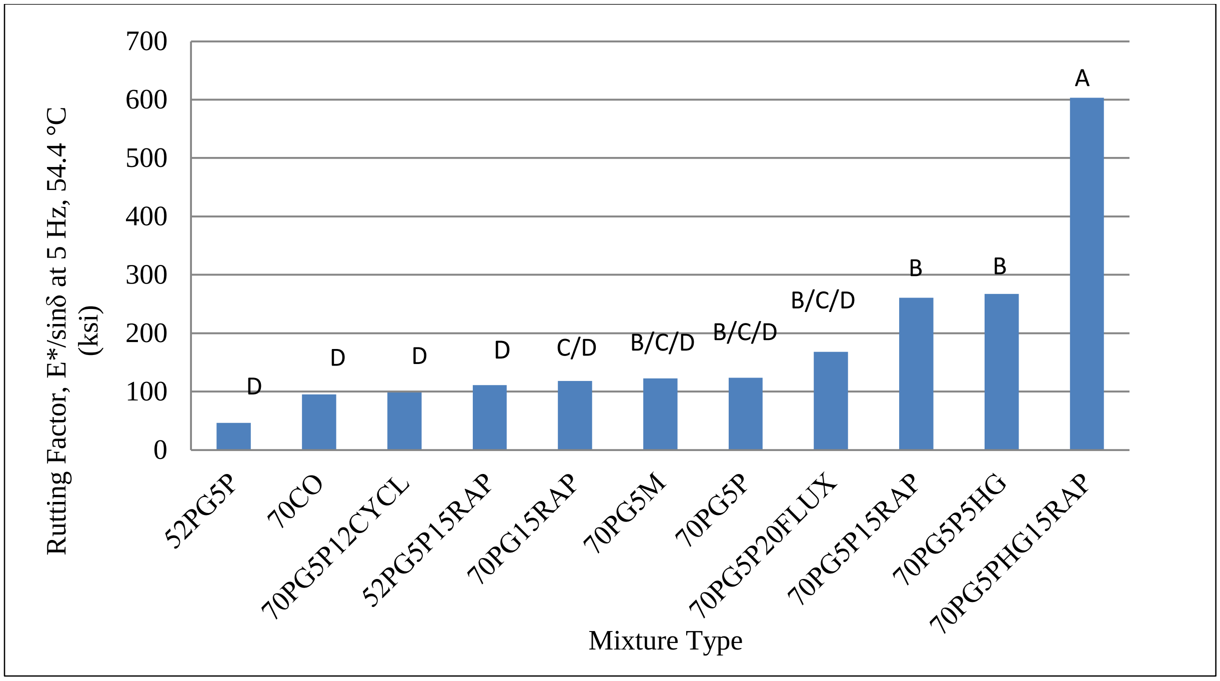


Figure 30
Dynamic modulus test result – rutting factor

High Temperature Mixture Performance

LWT (Hamburg Type) Test Results. Figure 31 illustrates the average permanent deformation depth for the 11 asphalt mixtures evaluated in this study. It is shown that the mixture 70PG5PHG15RAP was the most resistant to permanent deformation, whereas, the mixture 52PG5P containing the PG 52-28 soft asphalt binder and no recycling agent was the least resistant to rutting. However, it is noted that all mixture evaluated performed less than many state specifications for a 12.5-mm NMAS mixture. It is observed that the addition of RAP and RAS reduced the terminal rut depth as compared to the asphalt mixture with no RAS, 70CO. It is also noted that the mixture containing the Cyclogen-L ranked second in least resistance to rutting. These findings are in agreement with the rutting factor results shown in Figure 30. The mixture containing Hydrogreen, RAP and RAS (70PG5PHG15RAP) had the highest stiffness, highest RBR (Table 15), and, therefore, one would expect that it would be the most resistant to permanent deformation. Likewise, it is seen that the mix containing the soft binder (52PG5P) had the lowest dynamic modulus values, and, therefore, should be the most susceptible to rutting. As shown in Figure 32, generally the remaining mixtures were clustered together and are expected to perform similarly against rutting. No tertiary regions were seen in the asphalt mixtures studied (no stripping inflection points); therefore, no susceptibility to moisture damage as measured by the LWT could be observed.

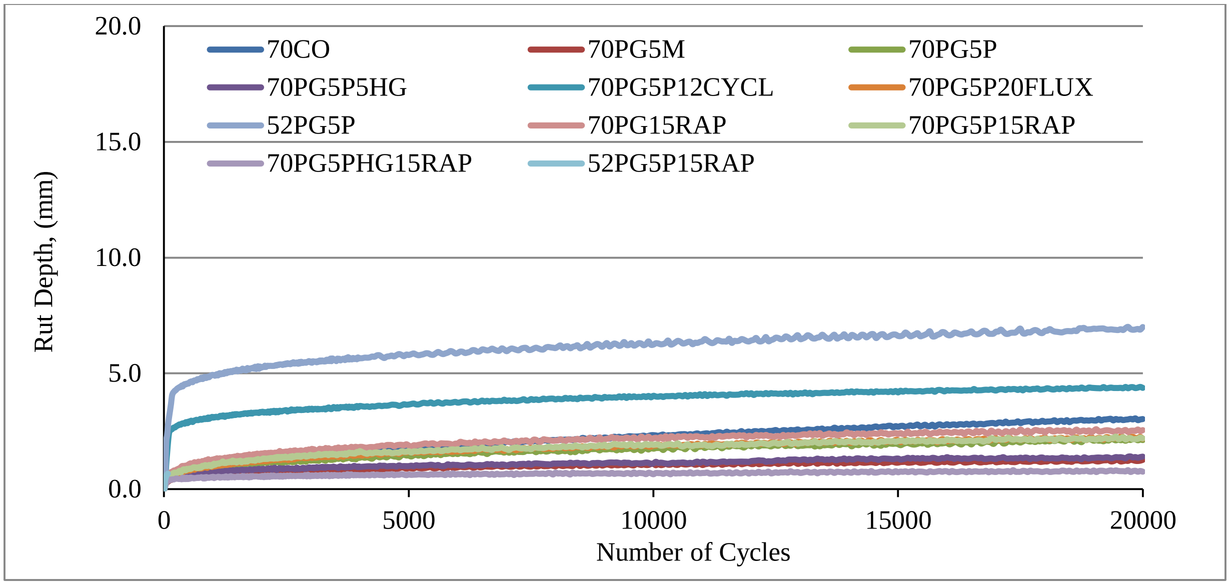


Figure 31
LWT test result, 50°C, wet

Figure 32 indicates the statistical differences between permanent deformation and mixture type. Laboratory test data were statistically analyzed using the analysis of variance (ANOVA) procedure (Tukey's studentized range HSD (honestly significant difference)) provided in the Statistical Analysis System (SAS) program. A multiple comparison procedure with a confidence level of 0.05 was performed on the means. The groupings represent the mean for the test results reported by mixture type. The results of the statistical grouping are reported with letters A, B, C, D, and so forth. Letter A was assigned to the highest mean followed by the other letters in appropriate order. A double (or more) letter designation, such as A/B (or A/B/C) indicates that the difference in the means is not clear-cut, and that the mean is close to either group in the analysis. Figure 32 indicates that there are only two statistical groupings. Statistically there is not much difference in these groupings and the difference is not significant.

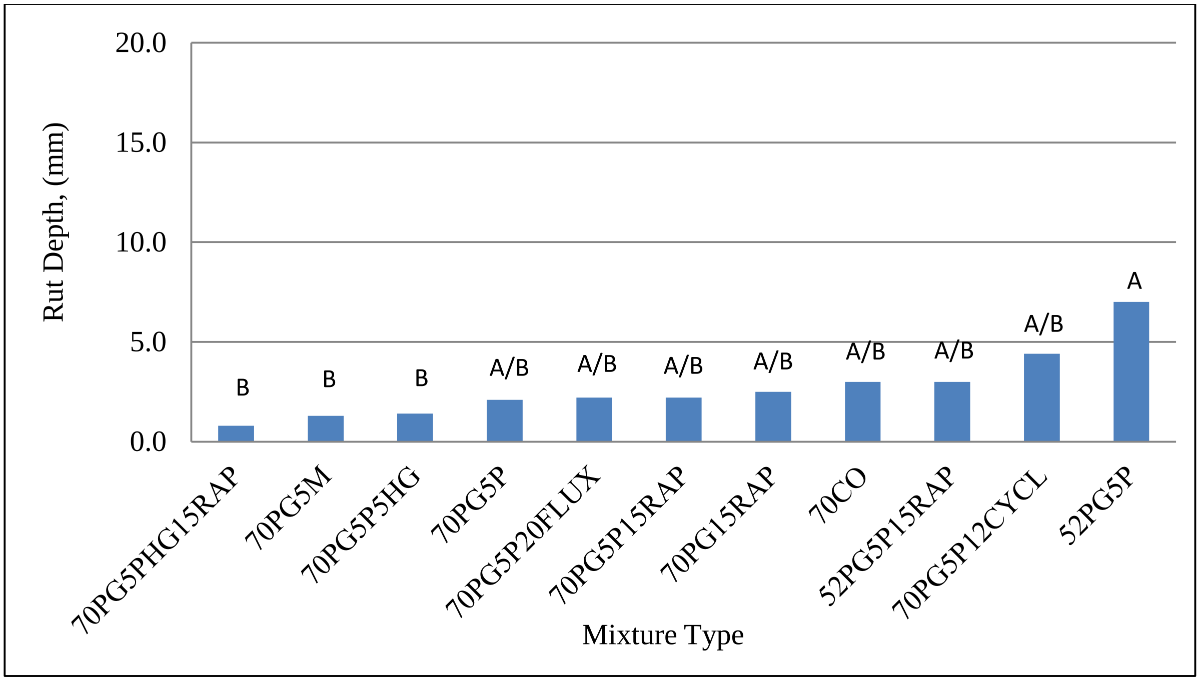


Figure 32
Statistical comparison: LWT test result, 50°C, wet

Figure 33 presents the characterization laboratory test correlation between the binder non-recoverable creep compliance, J_{nr}, (measured at an applied constant stress of 3.2 kPa and at a testing temperature of 67°C), and the LWT rut depth (permanent deformation) measured at 20,000 passes at a testing temperature of 50°C submerged in water for the asphalt mixtures evaluated in this study. A decrease in the non-recoverable creep compliance indicates an improved resistance to rutting damage. This figure shows that as the J_{nr} decreases the rut depth also decreases. It is indicated in Figure 33 that there is a good correlation between the non-recoverable creep compliance, J_{nr}, and LWT test results.

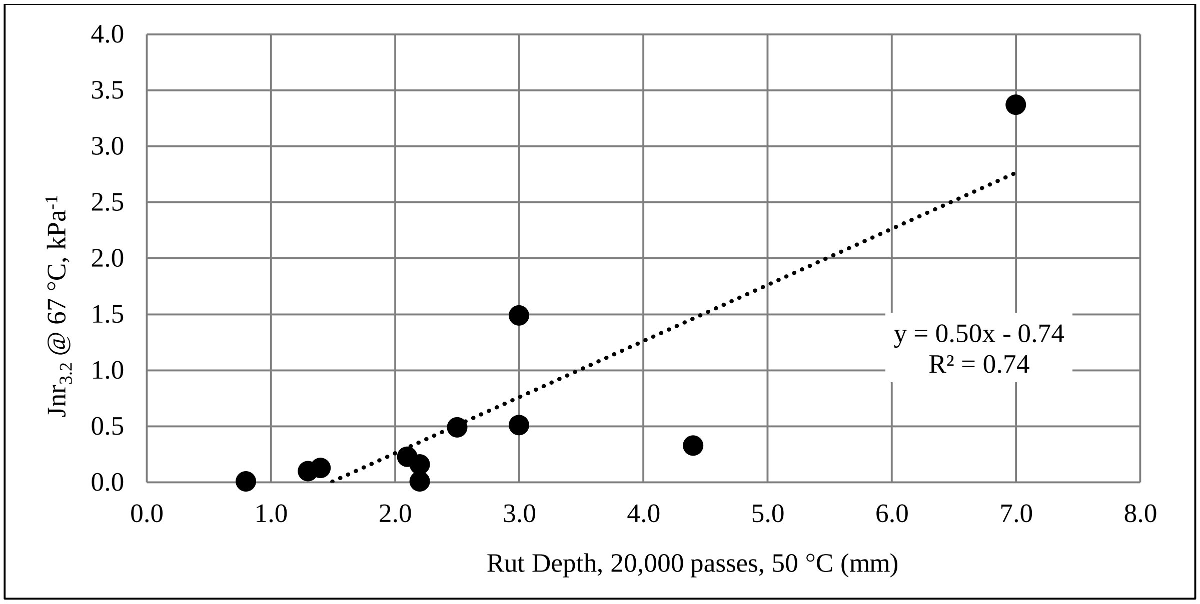


Figure 33
$J_{nr3.2}$ @ 67°C vs LWT rut depth

Figure 34 indicates the characterization laboratory test correlation between the Rutting Factor, E*/sinδ at 5 Hz, 54.4°C, and the LWT rut depth (permanent deformation) measured at 20,000 passes at a testing temperature of 50°C for the asphalt mixtures evaluated in this study. This figure shows that there is a trend between the Rutting Factor and rut depth test results. For mixtures to be rut resistant and exhibit higher stiffness, this necessitates a higher E* value and a lower phase angle. The higher the rutting factor value indicates a mixture greater resistance to permanent deformation. It is illustrated in Figure 34 that as the Rutting Factor increases the rut depth decreases. This is desirable trend since higher rutting factor values indicate an asphalt mixtures stronger propensity for rut resistance.

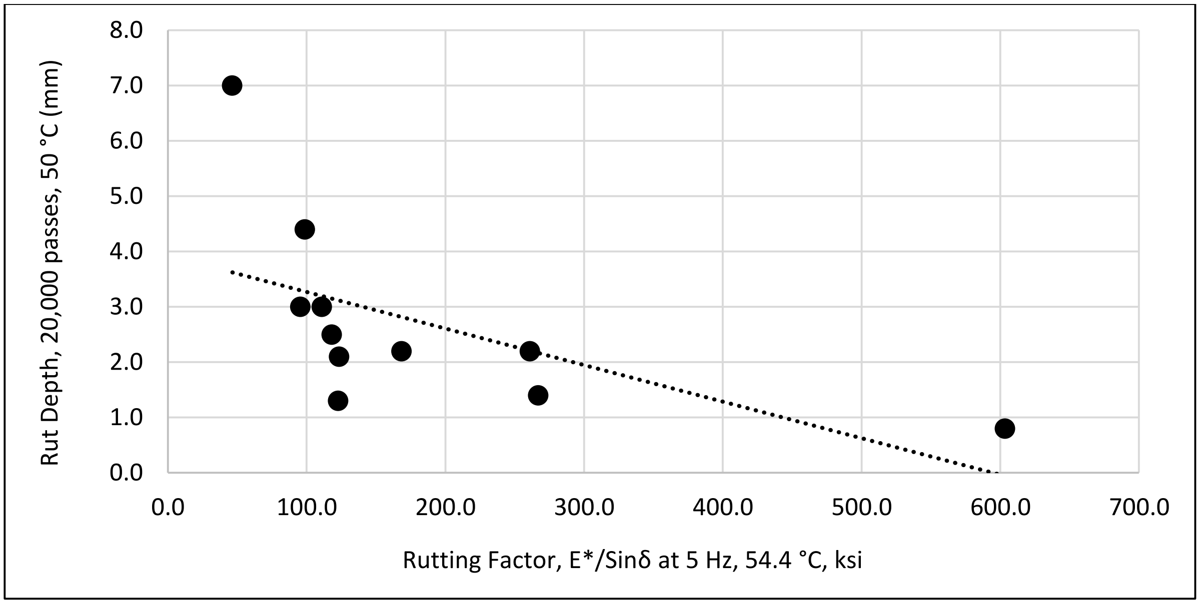


Figure 34
LWT rut depth vs rutting factor, E*/sinδ at 5 Hz, 54.4°C

Intermediate Temperature Mixture Performance

Semi-circular Bend Test Results. Figure 35 presents the calculated critical fracture resistance (J_c) values for the 11 asphalt mixture types evaluated. Mixture aging was performed according to AASHTO R30 by placing compacted specimens in a forced draft oven for five days at 85°C *[93]*. After aging, the specimens were loaded at a monotonic rate of 0.5mm/minute until failure. The higher the J_c value, the greater the fracture resistance the asphalt mixtures possess. It is shown that the 70PG15RAP asphalt mixture had the highest J_c value, and, therefore, has the greatest fracture resistance of all mixtures evaluated in this study. It is suspected that the 70PG15RAP had a the highest J_c value as compared to the control mixture (70CO) because the RAP utilized in this study comprised of a high percentage of polymer modified asphalt as measured by the molecular weight species by gel permeation chromatography.

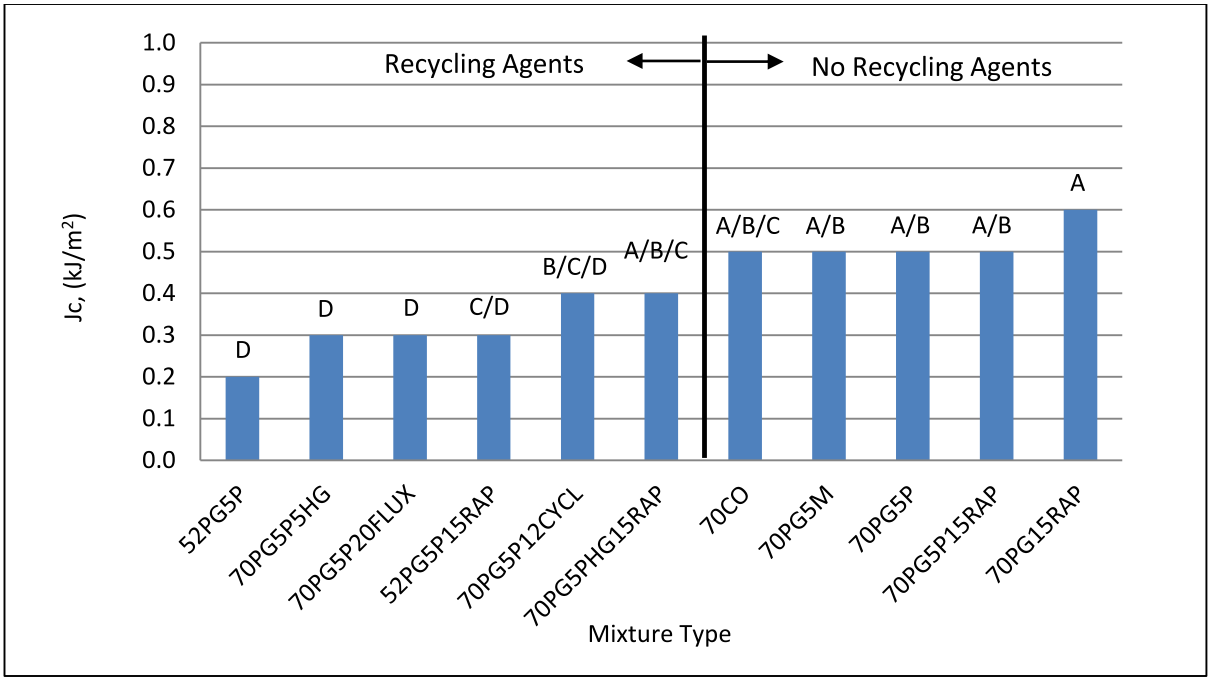


Figure 35
Semi-circular bend test results, 25°C

A minimum threshold J_c value of 0.50 kJ/m^2 is typically used as a failure criterion and is currently being implemented in Louisiana as an acceptance criterion for mixtures containing a PG 70-22 asphalt binder. A J_c of 0.5 kJ/m^2 or above is considered resistant to intermediate temperature fracture *[83]*. It is shown in Figure 35 that asphalt mixtures containing no recycling agents had higher J_c values than the mixtures containing recycling agents, rejuvenators and softening agents. In addition, those mixtures that do not have recycling agents also passed Louisiana's proposed J_c threshold specification of 0.5 kJ/m^2. This can be contributed to these mixtures having more virgin asphalt binder and lower recycle binder ratios than those mixtures containing recycling agents. Although some activation of the RAS binder was achieved without the use of recycling agents, some of the RAS acted as a black rock. This necessitated the use of more virgin asphalt binder to meet volumetric and densification criterion. When the RBR ratio for mixtures increased due to the addition of recycling agents, the resistance to fracture was adversely affected even though the recycling agents are classified as rejuvenators and softening agents. In addition, the use of the soft asphalt binder (PG 52-28) showed very little to no improvement in its resistance to fracture, mixtures 52PG5P and 52PG5P15RAP respectively. In fact, the asphalt mixture designated as 52PG5P is the least resistant to fracture as evaluated in this study.

Figure 35 indicates the statistical differences between J_c and mixture type. It is shown in Figure 35 that there are three statistical groupings. The first statistical grouping is represented by letter designations of A, A/B, A/B/C (mixtures 70PG15RAP, 70PG5P15RAP, 70PG5P, 70PG5M, 70CO and 70PG5pHG15RAP). The second grouping is B/C/D and C/D comprised of mixtures 70PG5P12CYCL and 52PG5P15RAP. The last grouping is "D" which is comprised of mixtures 52PG5P, 70PG5P5HG, and 70PG5P20FLUX and had the lowest SCB test results as measured by J_c. It is indicated in this figure that there is a statistical difference between mixtures containing recycling agents and those mixtures not containing recycling agents. Generally, there is no statistical difference between mixtures that did not utilize recycling agents. It is noted that some of these mixtures have statistical designations of A, A/B, and A/B/C, but this indicates that there is no clear-cut statistical difference between Groups A, B, and C since their mean values are close to both groupings.

Low Temperature Mixture Performance

Thermal Stress Restrained Specimen Tensile Strength Test (TSRST) Results. Figure 36 shows the low temperature fracture for the mixtures studied as measured by TSRST. Mixture aging was performed according to AASHTO R30 by placing compacted specimens in a forced draft oven for five days at 85°C *[93]*. After aging, the specimens were loaded at an applied rate of -10°C/hour.

The test was stopped either at -50°C (coolant limitation) or at fracture, whichever occurred first. The asphalt binder utilized in this study was modified with SBS with a low temperature grade of -22°C with the exception of the PG 52-28 asphalt binder. It is shown that the results indicate seven statistical groupings. However, several of the groupings have double (or more) letter designation, such as A/B (or A/B/C), which indicates there are no clear-cut statistical difference between Groups A, B, and C since their mean values are close to both groupings. Thus there are essentially three groupings. It is shown that mixture 70PG5P had the lowest fracture temperature and that this mixture is statistically different from mixture 70PG5PHG15RAP. It is noted that mixture 70PG5PHG15RAP had the highest fracture temperature (more susceptible to low temperature fracture) of the groupings, and this mixture had the highest recycle binder ratio and utilized a rejuvenating type recycling agent. Figure 36 also shows that generally the mixtures that contained no recycling agents were less susceptible to low temperature fracture, with the exception of mixture 52PG5P which utilized a soft asphalt binder (PG 52-28). Fracture occurs at the low temperature PG grade due to thermal contraction as the specimens are cooled at a rate of 10°C per hour. This may be contributed to only a portion of the recycled binder being activated within the mixture and the remaining recycled materials acting as a black rock.

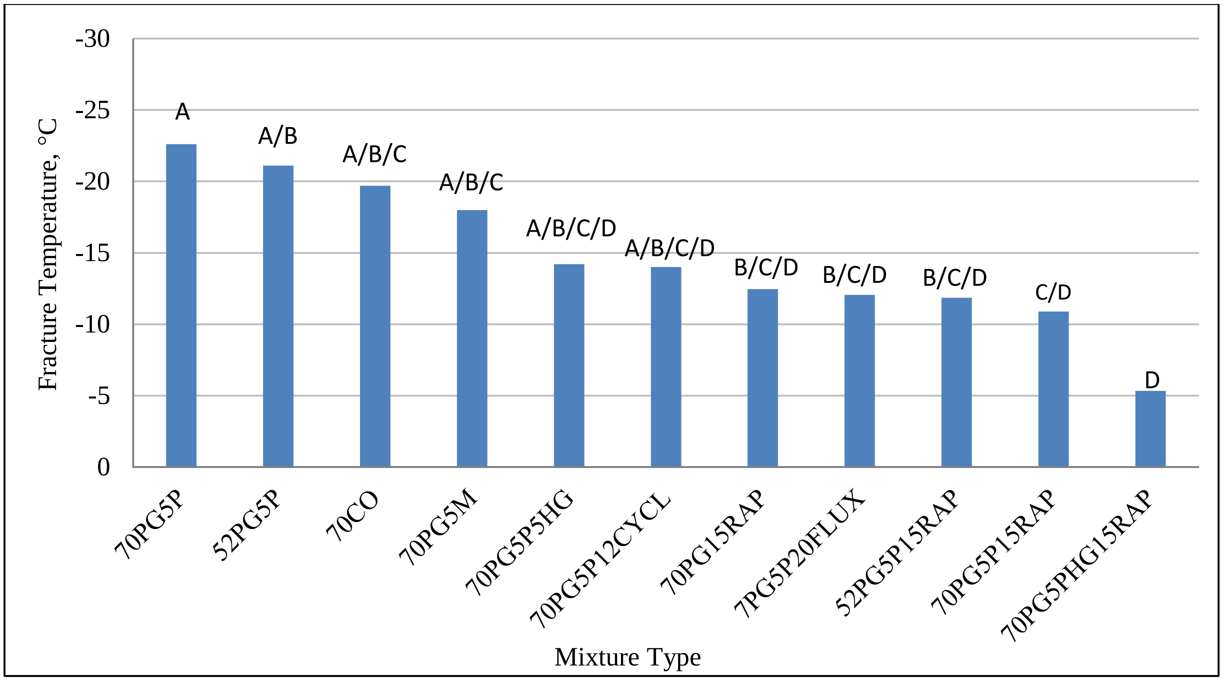


Figure 36
TSRST temperature vs. mixture type

Figure 37 compares the critical low temperatures of asphalt binders determined by the TSRST and by the BBR. It is generally expected that the fracture temperature measured by the TSRST corresponds to the PG low temperature of the asphalt binder utilized in the mixture. However, this assumption would only be valid for mixtures containing virgin asphalt binders. In fact, it has been observed that the correspondence, at some degree, depends on the contents of recycled materials utilized in the asphalt mixtures. As shown in Figure 37, the TSRST fracture temperatures were generally in good agreement with the extracted asphalt binders' PG low temperature values, but for the mixtures 70PG5P12CYCL, 70PG15RAP, and 52PG5P15RAP, the extracted asphalt binders' PG low-temperature were significantly lower than the TSRST fracture temperatures.

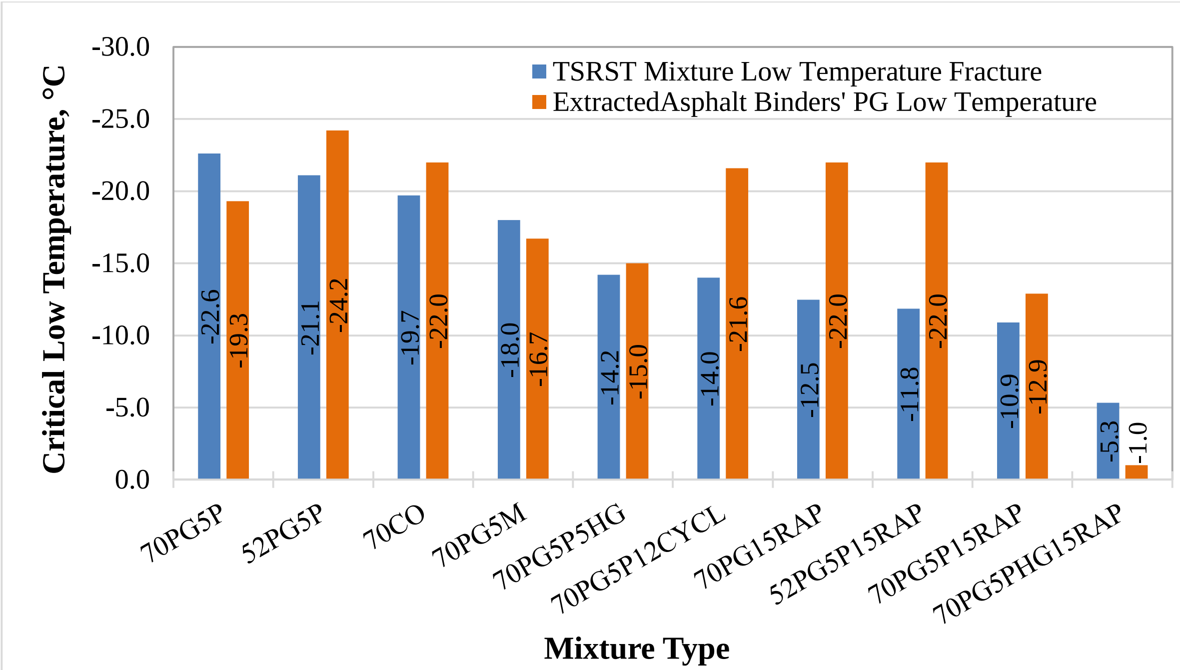


Figure 37
Critical low temperatures: TSRST vs. BBR

Figure 38 shows the correlation between the rheological index, R, and the PG low-temperature of the extracted asphalt binder as determined by BBR. This figure shows that there is a high correlation between these parameters. It is indicated in Figure 38 that as the rheological index becomes greater there is a decrease in the low temperature grade of the asphalt binder. It is expected that since an increase in R represents the increase in aging, which is known to adversely affect the low-temperature performance of the asphalt binder. As the binder ages, the asphalt binder loses the ability to relax under loading, and this is represented in the determination of the low-temperature PG property of the material, m-value. The 70PG5PHG15RAP as shown in Figure 37 has the highest fracture temperature (-5.3°C) and also has the highest rheological index of 3.24 as previously shown in Figure 23.

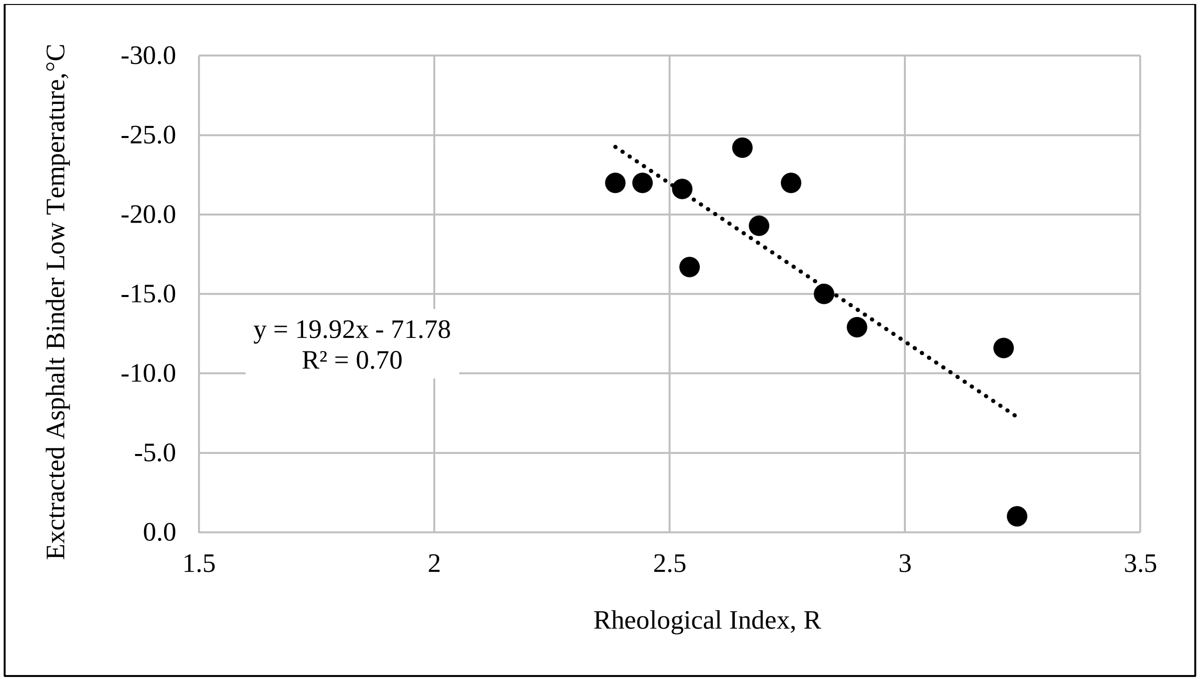


Figure 38
Extracted binder low temperature vs. R

Binder Experiment Results

Performance of RAS in Asphalt Pavements

The industry has been addressing the stiffness and blending concerns by using softer binders when using higher RAP/RAS contents. Since fatigue cracking is influenced more by the intermediate temperature binder properties, using soft (modified) binders is an effective method to improve cracking resistance of RAS mixes. Zhou et al. conducted a comprehensive investigation of asphalt mixtures containing RAS *[94]*. This study characterized the RAS asphalt binder including the evaluation of blending charts for virgin binder blended with RAS binders. In addition, the impact of RAS content on the optimum asphalt binder content and respective engineering properties on mixtures containing RAS was evaluated. It was concluded that the use of RAS had no significant influence on dynamic modulus, though it improved mixture resistance to rutting and moisture damage. However, mixtures containing RAS had very poor cracking resistance as compared to mixtures containing no RAS. Zhou et al. explored two approaches to improve cracking resistance of mixtures containing RAS *[94]*. It was stated that the use of soft binder and increasing the design density can improve cracking resistance. In terms of rutting and moisture damage, the use of soft binders was superior to increasing the design density. When using the softer binder and low air void approaches, one should be aware that, if the RAS is not well blended into the mixture or, if segregation occurs during mixing and/or placement, spots appear on the pavement of "softer" mix, which may fail due to rutting *[94]*.

Asphalt rejuvenating and softening agents are manufactured to restore the rheological properties of the reclaimed asphalt binder by diffusing into it and restoring its colloidal structure and reconstituting its chemical components. Therefore, rejuvenators have been extensively used in pavement preservation to revive the hard and oxidized top layer by penetrating into the pavement and fluxing with the aged binder to balance the maltenes to asphaltenes ratio *[95]*.

Diffusion is the key factor in blending rejuvenators with asphalts. Diffusion of rejuvenators in RAP binders has been of interest since several decades ago. Although there is experimental evidence that the rejuvenators penetrated the RAP binders, corresponding evidence on the impact of these agents on RAS is limited *[96], [97]*. Im et al. studied the impacts of three different rejuvenators on mixtures containing various contents of RAS and RAP, using LWT, the Overlay Test Repeated load test, and the dynamic modulus to characterize the mixtures *[98]*. It was concluded that the rejuvenators improved the cracking resistance, moisture susceptibility, and rutting resistance comparatively to the control mixture. However, the ranking of the three rejuvenators used in the study depended on mixture types and properties evaluated.

With the increased interest in using RAS, the use of recycling agents is considered essential in order to soften and/or to rejuvenate the aged and stiff binders in RAS. Recycling agents are classified as two types: rejuvenating agents and softening agents. Softening agents lower the viscosity of the aged binder while rejuvenating agents are intended to restore the rheological and chemical properties of the aged binder *[98]*. Examples of softening agents include asphalt flux oil, lube stock, and slurry oil. Examples of rejuvenating agents include lubricating and extender oils, which contain a high proportion of maltenes constituents and low saturate contents that do not react with asphaltenes *[95]*. The design and production of asphalt mixtures containing RAS requires provisions to assure that the final product will meet and/or exceed the expected pavement life as required by construction and performance specifications.

Extraction of Binder from RAS Samples

The RAS samples and mixtures containing RAS were extracted with refluxing toluene under nitrogen using a Soxhlet extractor. The solution of asphalt binder in toluene was cooled to room temperature and then filtered to remove most of the fine particles of sand present. The filtered solution was allowed to stand overnight, decanted, and concentrated under vacuum using a rotary evaporator. The concentrated asphalt binder solution in toluene was then dried for 36 to 48 hours in a vacuum oven first at room temperature (ca. 24 hrs), then at 50°C for 12 hrs.

Saturate, Aromatic, Resin, and Asphaltenes (SARA) Analysis Results

The SARA analysis of the binders performed in this study is compiled in Table 20. The asphaltenes are reported as n-heptane insolubles and the maltenes containing resins, aromatics, and saturates were determined by TLC/FID with an Iatroscan instrument. It is noteworthy that the asphaltenes component as determined by precipitation is considerably smaller than the sum of all the components with molecular weights greater than 3,000 Daltons estimated from deconvoluted GPC chromatograms, designated as DEC ASPH in Table 20. The higher percentage of DEC ASPH is composed of associated asphaltenes species with molecular weights as high as 100,000 *[99]*. The high molecular weight component (HMW) of the DEC ASPH (i.e., a sum of the species with molecular weights >19,000 Daltons), is also reported in Table 20. It is noted that the SARA asphaltenes analysis by precipitation does not capture the total amount of associated asphaltenes in the binder as some fractions of associated asphaltenes remain in the resin fraction.

Table 20
Chemical composition of extracted binders

Mix Designation	SARA Analysis, %					DEC ASPH, %	HMW, %	DEC MALT, %	Jc, kJ/m^2
	Asphaltenes	Resins	Aromatics	Saturates	Sum resins, aromatics, saturates				
70CO	23.2	32.7	42.4	1.7	76.8	30.0	1.0	70.0	0.5
70PG5M	23.3	36.0	42.4	1.7	80.1	34.0	3.0	66.0	0.5
70PG5P	23.6	26.7	46.0	3.7	76.4	39.0	5.7	61.0	0.5
70PG5P5HG	27.6	30.0	39.3	3.0	72.3	41.0	8.5	59.0	0.3
70PG5P12CYCL	24.8	26.7	42.1	6.4	75.2	38.6	6.0	61.4	0.4
52PG5P	20.2	29.2	45.6	5.0	79.8	30.0	4.7	70.0	0.2
70PG5P20FLUX	25.6	22.8	44.2	7.5	74.5	35.3	6.0	64.8	0.3
70PG15RAP	22.1	30.7	45.3	1.9	77.9	41.8	8.3	58.2	0.6
70PG5P15RAP	23.5	30.3	43.5	2.7	76.5	46.4	5.5	53.7	0.5
70PG5PHG15RAP	29.8	28.4	36.9	5.0	70.3	44.7	5.1	55.4	0.4
52PG5P15RAP	22.9	29.8	44.1	3.2	77.1	33.2	4.0	66.8	0.3

DEC ASPH = deconvoluted asphaltenes;
HMW = high molecular weight;
DEC MALT = deconvoluted maltenes; and

J_c = critical strain energy release rate.

Figure 39 presents a correlation between the cracking resistance of asphalt mixtures expressed by critical strain energy release rate, J_c, and the SARA analysis derived asphaltenes contents of extracted binders from corresponding mixtures. For the mixtures with PG70-22m binder, a decent linear relationship ($R^2 = 0.6$) is observed: it is noteworthy that the mixture's cracking resistance is inversely proportional to the asphaltenes contents. On the other hand, mixtures prepared using PG52-28 exhibited no appreciable relationships between the J_c and asphaltenes contents. It can be also noted that the asphaltenes content was increased in 70PG5PHG15RAP compared to 70PG5P5HG, but the J_c was also increased, which is opposite from the general trend. This could be attributed to the fact that the RAP binder used in this mixtures contains approximately 2% polymer, which was beneficial in increasing the intermediate temperature cracking performance.

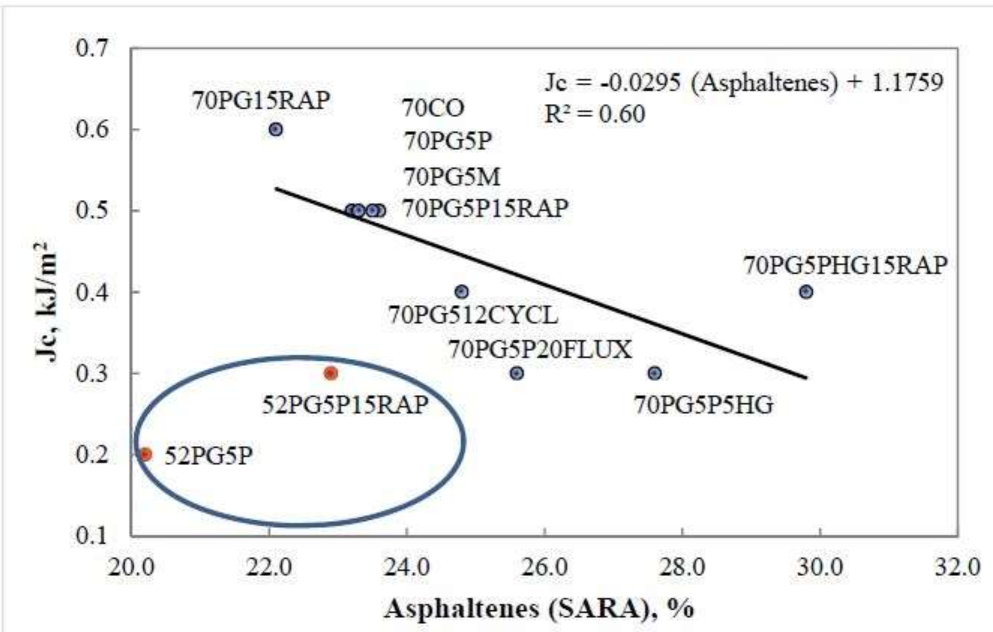


Figure 39
Mixture critical strain energy release rate (J_c) vs. asphaltenes content obtained from SARA fractionation

GPC Analysis Results

As presented in Figure 40 for a PG 64-22 binder, quantification of asphalt components is readily made by determination of area of the respective eluted fraction calculated based on the fact that the area under the curve represents 100% of the sample molecules injected into the GPC system. The total GPC curve can be deconvoluted to show the contributions of the asphalt components using commercially available software Origin 7.

Earlier determinations by osmometry indicated that the average MW of maltenes (as heptane soluble binder fraction) is 700-900 Daltons and that of asphaltenes (as heptane insoluble binder fraction) ranges between 2,000 and 10,000 Daltons *[100]*. These MW data have been confirmed by GPC method, which became a routine technique in Louisiana for analysis of asphalt binders *[101]*.

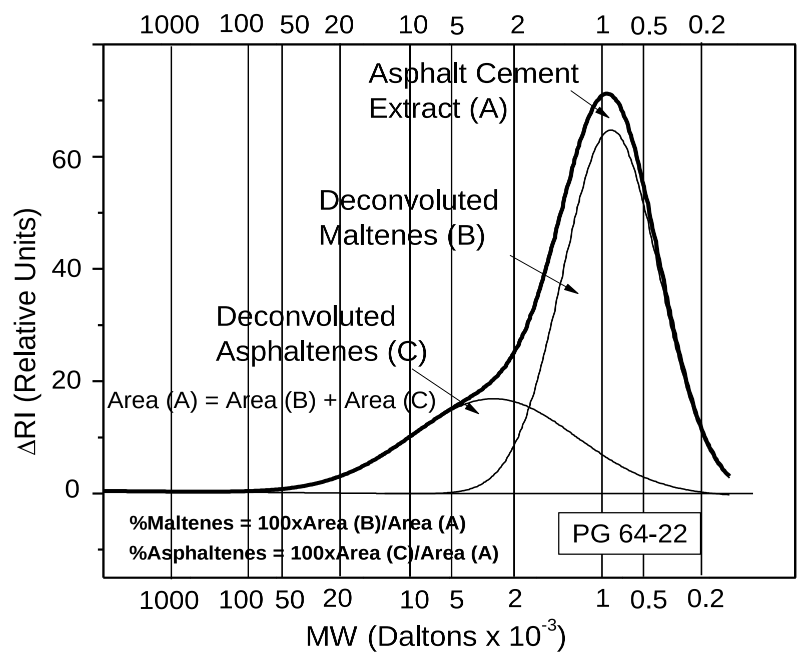


Figure 40
Maltenes and asphaltenes content of PG 64-22 binder by deconvolution of the GPC curve

Since the MW of polymers used in asphalt industry is higher than 10,000 Daltons, the polymer and asphalt components of polymer modified asphalt binders could be separated completely with accurate determination of molecular weight of species achieved by calibration with standard polystyrenes of narrow MW (Figure 41). It is noted that the GPC elution curve was from a mixture that was aged for 5 days at 85°C.

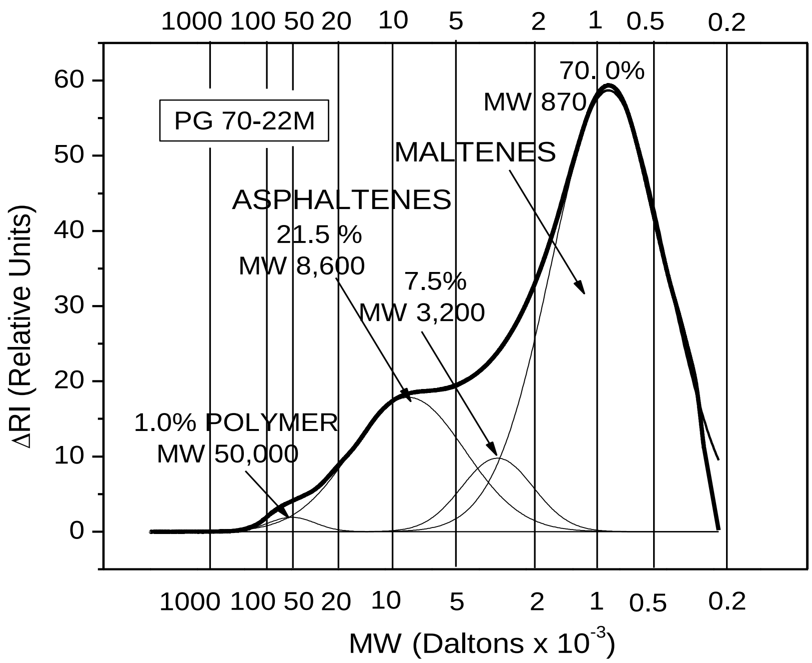


Figure 41
GPC elution curve of PG 70-22M containing 1% polymer extracted from an aged mixture

Asphaltenes, by virtue of their molecular size as the higher MW component, are the bodying agent for the maltenes, having a significant influence on asphalt performance *[101]*. The largest "molecules" are assemblies of smaller molecules held together by one or more intermolecular forces. Through changes in the polarity of the solvent used in the analysis, the ability of the samples to undergo self-assembly by different interactive mechanisms has been probed *[102]*. Therefore, by analyzing the asphaltenes in various asphalt mixture combinations, such as asphalt binders and polymer-modified binders, with and without reclaimed asphalt materials (RAP and RAS), one can correlate the performance of various asphalt mixtures to the content and MW magnitude of asphaltenes species.

The material used to manufacture roofing shingles is a highly oxidized blown asphalt as confirmed by the GPC chromatograms; the assemblies of asphaltenes species from blown asphalt and MWS are practically identical (Figure 42). In addition to oxidation, the blowing process increases the asphalt aromaticity (conjugation) and average molecular size, which improves opportunities for self-assembly. A bi- or tri-modal peak shape showing the presence of two or three distinct populations of molecular sizes is regarded as evidence of intermolecular association in the large molecular size (LMS) region on the left of the chromatogram *[103]*. Over 25% of associated asphaltenes in blown asphalt and MWS have an apparent average MW’s of 10K-50K Daltons. Asphalt binders extracted from MWS and post-consumer waste shingles (PCWS) typically have different properties because of the aging of the later that occurs on roofs. A further major concern with using recycled asphalt shingles relates to the variability in the properties of the RAS materials originating from different sources *[76]*, *[77]*.

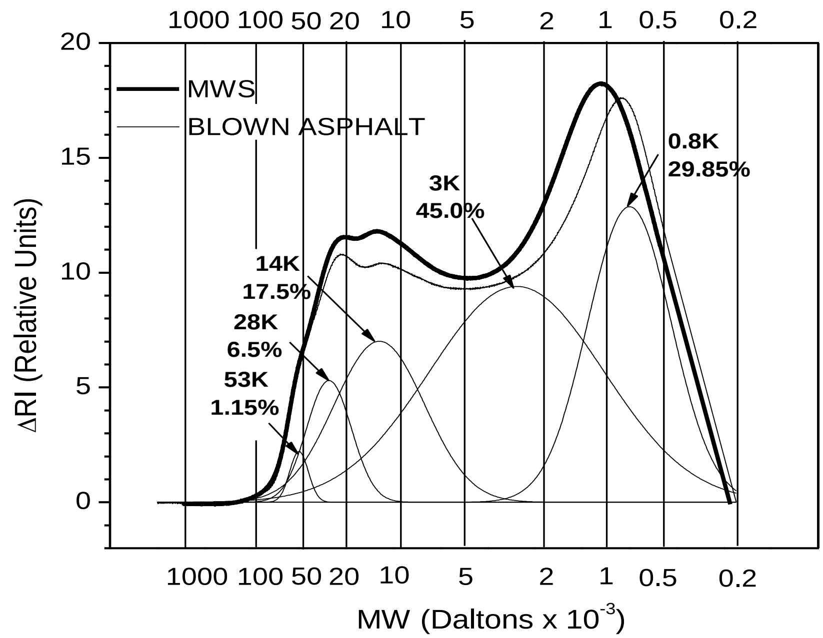


Figure 42
GPC data of blown asphalt and MWS extracted binders

The GPC traces of extracted binders from the Texas RAS used in this study is shown in Figure 43. Discernible differences between RAS sample are evident when one compares the maltenes component to the high end asphaltenes (MW>10K Daltons) component of the extracts. The ratio of the areas shown in the de-convoluted chromatograms identified possible problems with the compatibility of component species when blended with virgin asphalts. For example, the ratio for the binder extracted from RAP originating from the Texan source is ~55/33. In contrast, the corresponding ratio for a Minnesota PCWS extract is quite different, viz., ~80/20. The high molecular weight asphaltene content in the Texas PCWS suggests that this material was less compatible with virgin asphalt binders.

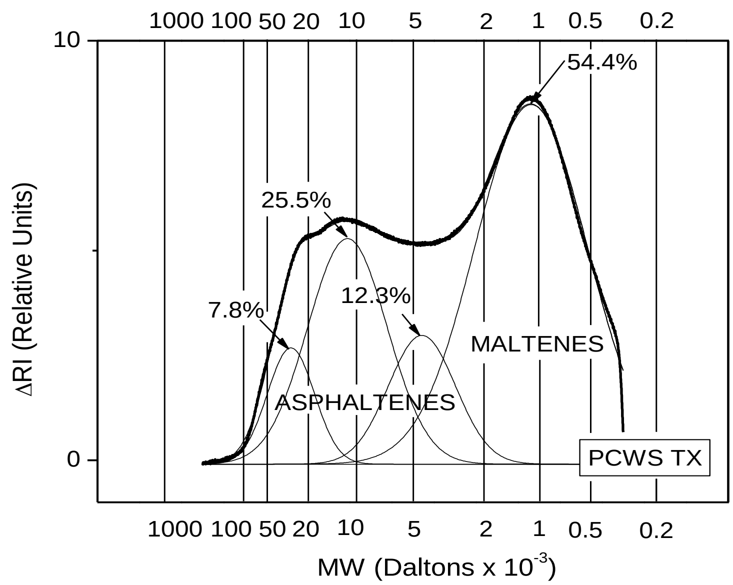


Figure 43
GPC traces of RAS binders extracted from PCWS of Texas origin

In Figure 44, the asphaltene fraction isolated by heptane precipitation of an asphalt binder extracted from Texas MWS was further examined. The MW of the asphaltenes components of MWS greatly surpasses that of a similar precipitation of a PG 64-22 binder in Figure 45. Asphaltenes from PG 64-22 can be separated into two fractions (i.e., molecules with an average MW of 2,000 Daltons (60%) and that with a peak average MW of 6,700 Daltons (22%)). The asphaltenes from MWS could be separated into three fractions with an average MW of 3,000 Daltons (52%), 12,000 Daltons (32%), and 24,500 Daltons (15%). Thus, the asphaltenes content of the MWS sample with molecular weights higher than that in PG 64-22 is approximately 47%.

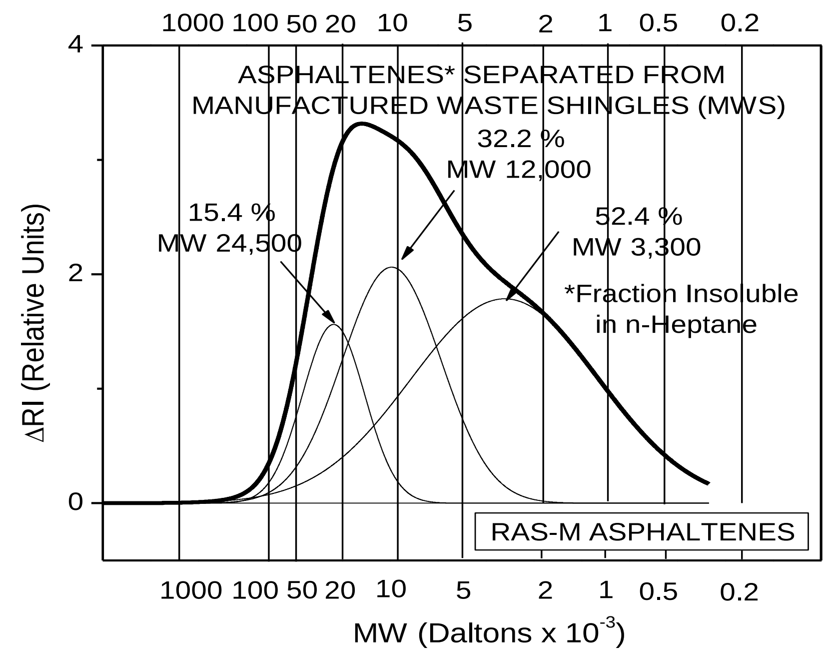


Figure 44
Average MW distributions of n-heptane insoluble asphaltenes species isolated from MWS

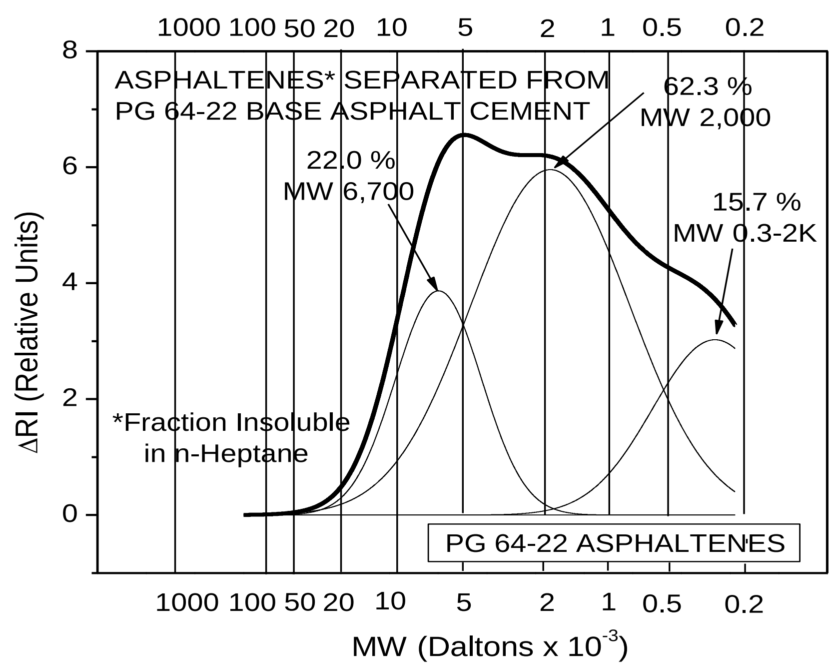


Figure 45
Average MW distributions of n-heptane insoluble asphaltenes species isolated from PG64-22 binder

The GPC chromatograms from an asphalt binder extracted from mixtures 70PG5P and 70PG5M, containing 5% PCWS and 5% MWS, are presented in Figures 46 and 47, respectively. The contribution of extremely oxidized components in PCWS is apparent in Figure 46. The 70PG5P mixture contained over 18% species with average MW>10K Daltons, out of which approximately 6% are of MW averaging 33,000. In contrast, a mixture prepared with 5% MWS, 70PG5M, contained only 3% of species with an average MW of 24,000 Daltons as shown in Figure 47. The maltenes to high end asphaltenes (MW>10K Daltons) ratio of 70PG5M (66/3) is significantly different from that of mixture 70PG5P (61/18), suggesting a higher potential compatibility of mixtures containing MWS.

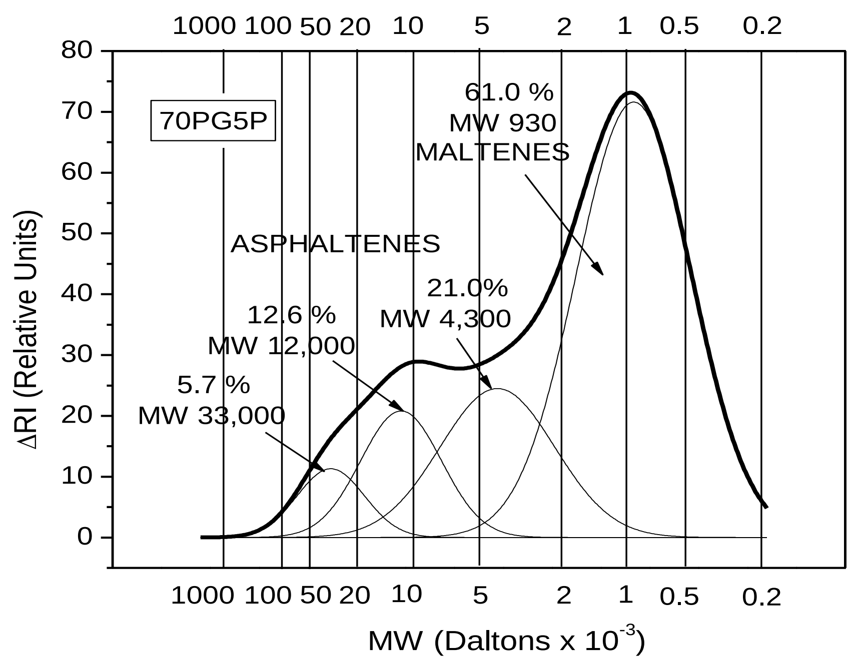


Figure 46
MW distribution of molecular species of extracted 70PG5P binder

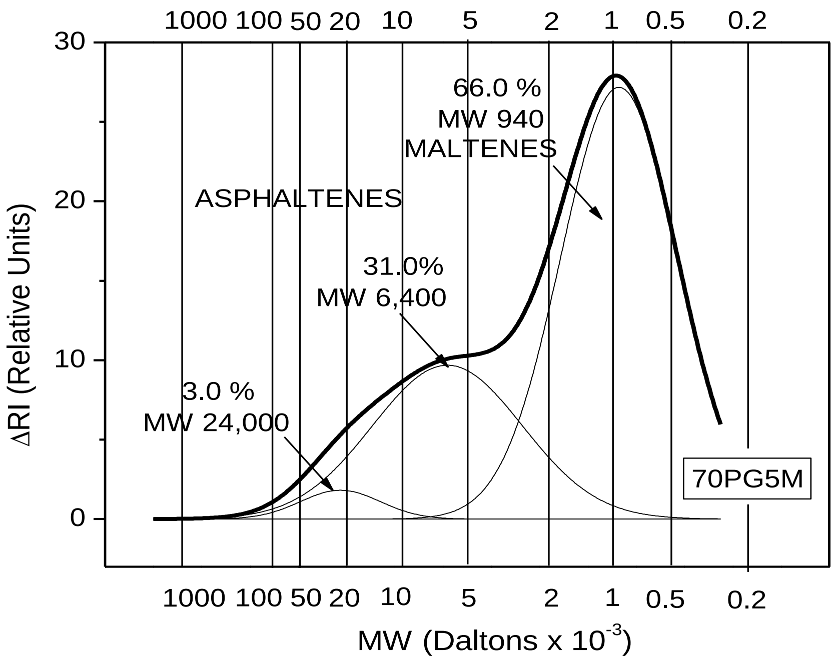


Figure 47
MW distribution of molecular species of extracted 70PG5M binder

According to Cooper, Jr., the high end MW of RAS asphaltenes can exceed even 100,000 Daltons *[87]*. The extremely large difference between MW's of associated asphaltenes from RAS and those from base binders when shingles are incorporated in paving asphalt materials impacts the compatibility of the mixes. It has been reported that some blending occurred between virgin PG 64-22 binder and RAS binder during the asphalt mixture mixing and curing (or short-term aging) processes *[100]*. The blending was not 100% since the high-temperature grades of the MWS and PCWS extracted binders, were 122°C and 166°C. The mixing temperature for a PG 64-22 binder is approximately 143°C (290°F). Zhou et al. stated that "extremely, impractically high temperature is required in order to make the RAS binder flow and comingle with virgin binder" whether using tear-off asphalt shingles or manufactured waste asphalt shingles *[94]*. Therefore, much higher blending temperatures are required in order to make the RAS binder flow and comingle with virgin binder. Moreover, earlier investigations on cross-blending of asphaltenes and maltenes fractions among several asphalts indicated that the asphaltene fractions are not as equally interchangeable as the maltenes components, and the effect of both molecular weight and the chemical nature of the asphaltenes must be taken into account to predict properties of asphalts from chemical composition *[103]*. Two types of segregation causing phase separations can occur when blending of asphalts containing dissimilar asphaltenes and maltenes fractions: one is simply that separation occurs in supersaturated solutions when components are ejected because of insufficient solvent (e.g., flocculation in blends of high asphaltenes content); the other is the rejection of a component when its amount exceeds the mutual compatibility limit, in the form of physical ejection of a liquid from a gel *[67]*. In view of these observations, the large difference between the MW of asphaltenes fractions of the base PG64-22 (ca. 20% of maximum MW ≈ 7-8,000) and the asphaltenes present in Waste Shingles (MWS and PCWS), ~ 40% of MW >12,000, with 15% MW ≈ 25-30,000 Daltons) makes difficult the dispersion of large RAS asphaltenes associations by the maltenes of the base asphalt with which the shingles was blended. To this aim, one must consider also the maltenes/high end asphaltenes (MW>10K Daltons) ratio mentioned above: the higher the ratio, the better. It has been shown earlier that an increase in the binder content of LMW (i.e., MW<3K), or in other words of the content ratio of Maltenes/Asphaltenes, resulted in an increase in its elongation properties at intermediate and low temperatures *[103]*.

Intermediate Temperature Cracking Performance of Asphalt Mixtures Containing RAS

Since the asphaltenes content in asphalts is related to the stiffness, the question arises: When blending virgin asphalt binder with RAS, will the asphaltenes content of the resulting binder follow the additive rule and will the J_c change correspondingly? The answer is no in most cases because the non-polar maltenes of the virgin asphalt are not compatible with the very highly oxidized RAS asphaltenes species. It has been shown that the virgin/RAS binder blending was nonlinear, unlike the well-known virgin–RAP binder linear blending *[94]*. PCWS binders were much stiffer than MWS binders *[100], [98]*. Compared with PCWS, MWS binders had much less impact on properties of blended virgin/RAS binders *[9], [1]*. Cooper et al. reported that the addition of 5% PCWS decreased the intermediate temperature cracking resistance (expressed by critical strain energy J_c) of a PG 70-22M binder when compared to that of a similar mixture in which PCWS has been substituted with MWS *[1]*.

Analyzing the GPC data obtained for the same materials (i.e., 70PG5P and 70PG5M) presented in Figures 46 and 47, respectively, it is considered that the main reason for higher stiffness of the 70PG5P PCWS binder reported by the authors is the degree of association of its large MW end asphaltenes (~ 6% of MW 33,000), which is higher than that found in the 70PG5M binder containing 5% MWS (3% MW 24,000 Daltons) *[1]*. It has been pointed out before that the ability of asphalts to form an intermolecular network by associations could lead to cracking with time and under cold conditions *[84]*.

Table 21 summarizes the data for a series of asphalt binders extracted from RAS containing mixtures. The asphaltene and maltenes fractions of 11 asphalt are the values of carbonyl index and of J_c integral in order to find a correlation between these data to predict, to a limited extent, the field performance of considered mixtures. Data listed in Table 21 show that rather large carbonyl indices and lower than 0.5 kJ/m^2 of J_c values were registered for mixtures containing PG 52-28 and PG 70-22M binders in which 3-6% asphalt species had MW > 19K (i.e., asphaltenes).

Table 21
MW distribution, C=O, and J_c values of materials utilized

Mixture Designation	VHMW (%) (Polymer & highly associated Asphaltene) 1000K - 19K	Asphaltene (%) 19K - 3K	Maltene (%) <3K	Carbonyl Index (C=O)	J_c (kJ/m^2)
Hydrogreen	0.00	0.88	99.12	4.0000	----
Cyclogen-L	0.00	0.00	100.00	0.0606	----
Asphalt Flux	0.00	0.00	100.00	0.0348	----
PG52-28	0.36	17.65	81.99	-0.0058	----
PG70-22M	3.15	22.85	74.00	0.0000	----
PCWS	15.56	32.04	52.40	0.2200	----
MWS	13.18	32.85	53.97	0.2500	----
RAP	2.06	27.83	70.11	0.1691	----
70PG5M	5.29	26.51	68.20	0.1202	0.5
70CO	3.57	25.00	71.43	0.0681	0.5
70PG5P	7.15	28.22	64.63	0.2124	0.5
70PG5P5HG	10.88	29.05	60.07	0.1713	0.3
70PG5PHG15RAP	13.07	29.84	57.09	0.2120	0.4
70PG5P12CYCL	9.08	27.35	63.57	0.1305	0.4
70PG5P20FLUX	8.56	29.23	62.21	0.1667	0.3
70PG15RAP	8.38	28.26	63.36	0.0950	0.6
70PG5P15RAP	10.50	30.23	59.27	0.1406	0.5
52PG5P	4.34	25.10	70.56	0.1174	0.2
52PG5P15RAP	5.81	28.56	65.63	0.1256	0.3

Figure 48 presents a comparison plot of the content of asphalt species with MW larger than 19K Daltons vs. J_c values of the 11 asphalt mixtures. Note that 19K Daltons of MW is the MW threshold of asphaltenes related to the stiffness of asphalt binders *[101]*. Higher J_c values are desirable for fracture-resistant mixtures. A minimum J_c value ranging from 0.50 to

0.65 kJ/m^2 is typically used as a failure criterion *[83]*. In Louisiana, a minimum J_c value of 0.5 kJ/m^2 is being considered as an acceptance criterion for mixture design.

Apparently, more than half of the 11 asphalt mixtures failed to meet the minimum design J_c requirement for Louisiana asphalt pavements. Among the five mixtures that showed greater than or equal to 0.5 kJ/m^2 of J_c value, four were on the borderline and only one asphalt mixture, i.e., 70PG15RAP, passed the requirement. In addition, it can be observed that all mixtures meeting or exceeding the minimum J_c criteria of 0.5 kJ/m^2 were mixtures that were produced with PG70-22M SBS modified asphalt binders and did not contain recycling agents.

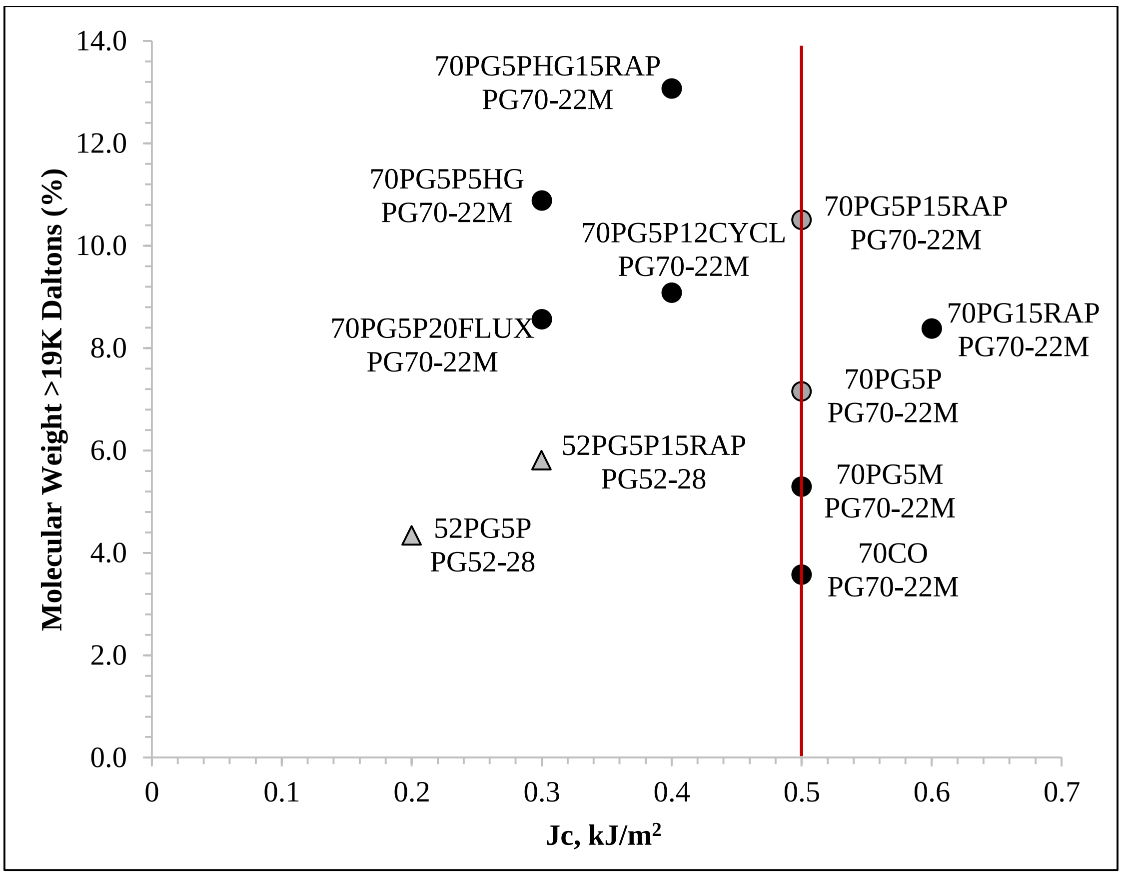


Figure 48
High MW fractions of 11 asphalt binders vs. corresponding J_c values

When comparing 52PG5P and 70PG5P, it looks clear that the high PG binder (i.e., PG70-22M) contains considerably more percentage of high MW fractions (7.15%) than the PG52-28 binder does (4.34%), which may have contributed to the higher fracture resistance of the 70PG5P mixture (0.5 kJ/m^2) than that of the 52PG5P mixture (0.2 kJ/m^2). This observation holds true when comparing 52PG5P15RAP and 70PG5P15RAP mixtures, i.e., 5.81% vs.

10.5% high MW fractions and 0.3 kJ/m^2 vs. 0.5 kJ/m^2 of J_c values for 52PG5P15RAP and 70PG5P15RAP, respectively. Generally speaking, polymers have very high MW and the SBS used in the PG70-22M binder is believed to be responsible for the observed increase in the high MW fractions of 70PG5P and 70PG5P15RAP compared to 52PG5P and 52PG5P15RAP, respectively. Thus, the increase in J_c of the two-70PG mixtures can be attributed to the SBS polymer modification, which is a direct evidence of the improved fracture resistance.

In a similar way, correlations can be found between various binders' high MW fractions and the intermediate temperature cracking resistance.

Intermediate Temperature Cracking Performance of Asphalt Mixtures Containing RAS and RAs

Figure 49 separates four asphalt mixtures (i.e., mixtures containing PG70-22M binder and 5% PCWS) from Figure 48 to see the effects of three RAs (i.e., Cyclogen, Hydrogreen-L, and Asphalt Flux) on the high MW fractions and J_c values clearer.

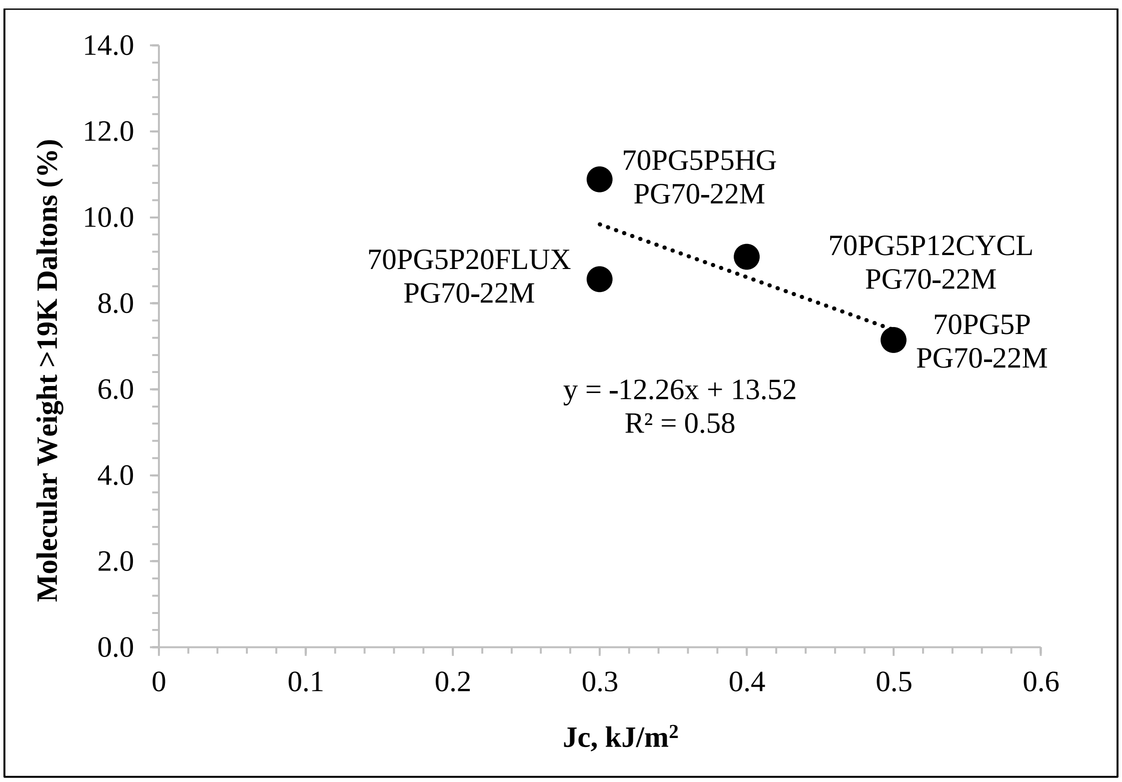


Figure 49
High MW contents for mixtures containing PG70 and 5P vs. J_c

A decent linear correlation can be observed between the high MW fractions and J_c values of these four asphalt mixtures. Interestingly, addition of the three RAs to the 70PG5P mixture increased the high MW fractions and decreased J_c values. Considering the anticipated benefits of these recycling agents, such as rejuvenating or softening of hardened asphalt binders, the trend is in the opposite direction.

Figure 50 illustrates the %MW asphalt fraction species >19K Daltons as it relates to the critical strain energy release rate, J_c, from mixtures containing PG70-22M asphalt binder, 5% PCWS, and 15% RAP. It is shown that there is a good correlation between the MW >19K Daltons and J_c. Figure 50 illustrates that as the MW >19K Daltons decrease the J_c increases.

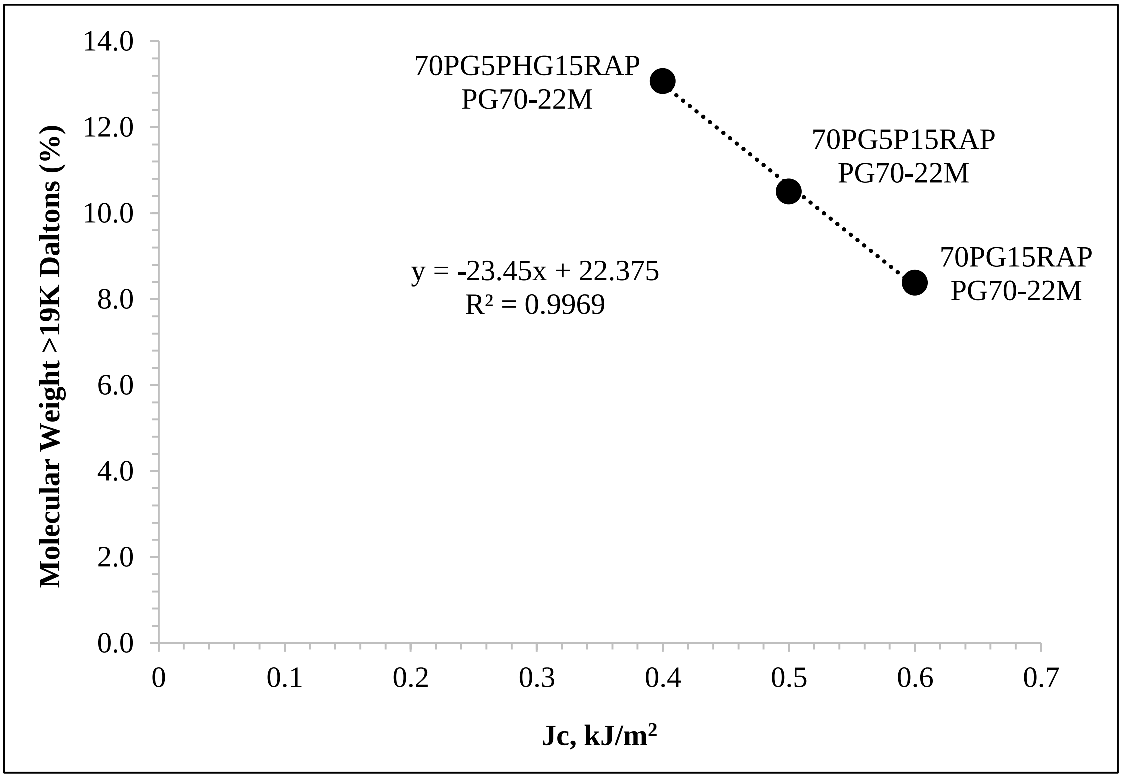


Figure 50
High MW contents for mixtures containing PG70, 5P, and 15RAP vs. J_c

One of the recycling agents chosen for the study was Hydrogreen (HG), which is an esterified derivative obtained from rosin, a by-product of the pulp and paper industry. This environmentally green rejuvenator is a low molecular weight product with the MW distribution shown in Figure 51. Its oxygen content is reflected by a significant carbonyl index (C=O = 0.04). Only 25% of its species matches the molecular weight of maltenes from an asphalt binder (MW ≈ 800-1500). The other recycling agents considered were naphthenic oil (Cyclogen-L), also a rejuvenator, and an asphalt binder meeting a PG 52-28, a softening agent. The anticipated role of the rejuvenators for RAS mixtures is to lower the association of high-end large MW asphaltenes present in RAS binders. However, the addition of 5% HG to a PG70-22M binder containing 5% PCWS (70PG5P5HG) does not seem to affect the distribution of high MW fractions derived from PCWS (Figure 51).

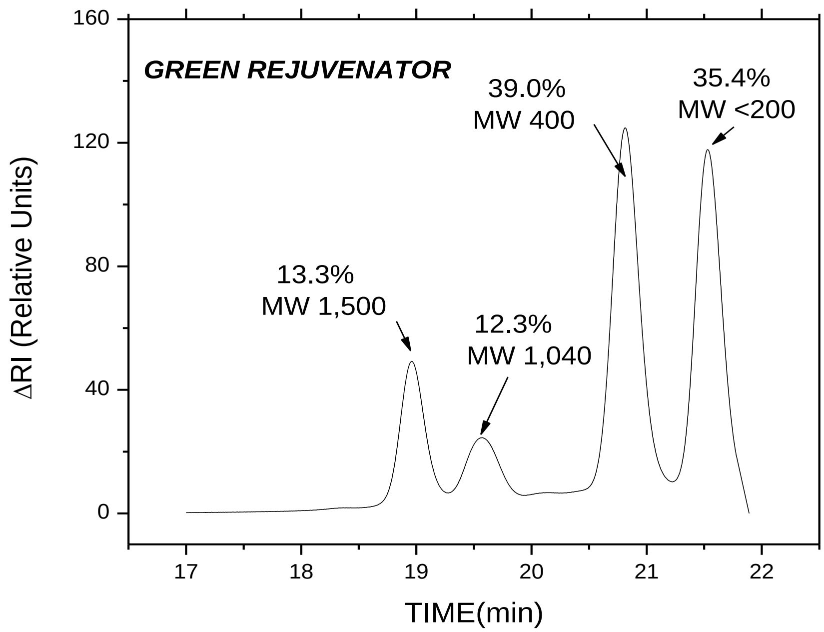


Figure 51
Molecular weight distribution of Hydrogreen green rejuvenator

At the same time, data listed in Table 22 and Figure 50 show that the addition of 5% HG to a PG70-22M binder containing 5% PCWS (70PG5P5HG) did not reduce the high MW asphaltenes and failed to retard the increase of high MW fraction due to the mixture aging for 5 days at 85ºC. This was clearly evident by the increase of asphaltenes from 9.59% to 11.50%, the corresponding reduction of the content of maltenes, and the increase in carbonyl index. These factors may have precluded the decrease of J_c from 0.37 to 0.26 kJ/m^2.

The blending and aging of 70PG/PCWS binders with other rejuvenating agents thought to improve the low temperature performance of the mixtures provided similar results. Adding softening agents instead of the Hydrogreen rejuvenator did not seem to alter the MW distribution of asphalt components (Table 22). GPC traces and MW distribution remained practically the same after SCB aging for both Cyclogen-L containing PG70-22M binder and the PG52-28 binder (Figures 52 and 53, respectively). While C=O index increased accordingly after aging, the cracking resistance expressed by critical strain energy, J_c, remained below the accepted limit (J_c<0.5 kJ/m^2), with decreased values for SCB aged mixtures, save for PG 70-22M containing 5% PCWS with 4.6% PG 52-28 by total weight of mix (Table 21).

Table 22
MW distribution, C=O, and J_c of related RAS binders containing RAs

Sample ID		Total VHMW (%) (Polymer &Highly Associated Asphaltenes) 1000K-19K	Total HMW (%) (Asphaltenes) 19K-3K	Maltene (%) < 3K	C=O $\times 10^2$	J_c (kJ/m^2)
70PG5P5HG	Unaged	9.59	28.06	62.34	13.9	0.37
	Aged 5days @85°C	11.50	29.06	59.43	17.1	0.26
70PG5P12CYCL	Unaged	8.30	25.98	65.38	9.9	0.13
	Aged 5days @85°C	8.77	27.17	64.06	13.0	0.36
52PG5P	Unaged	4.62	24.78	70.59	9.1	0.17
	Aged 5days @85°C	5.68	26.10	68.22	11.7	0.24

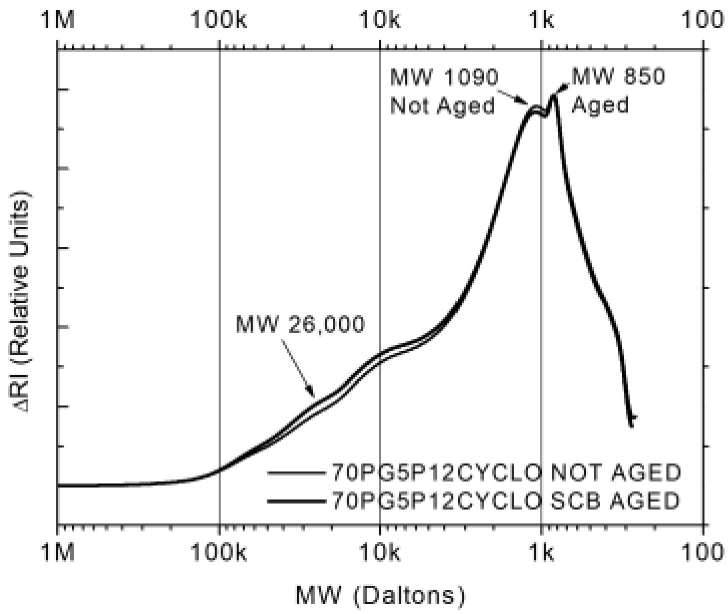


Figure 52
GPC traces of SCB aged and un-aged PG70-22M containing PCWS with Cyclogen-L

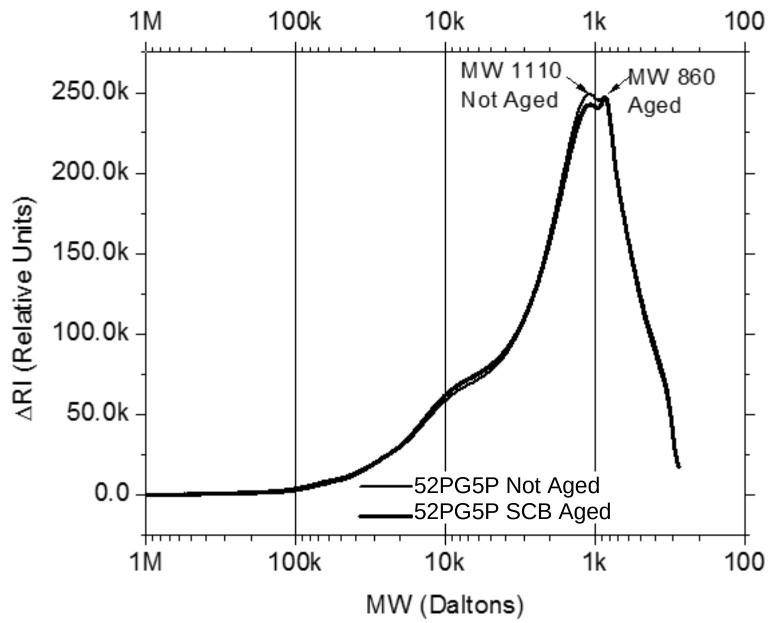


Figure 53
GPC traces of SCB aged and un-aged PG52-28 containing PCWS

Figure 54 presents the percentages of the HMW >3000 Daltons and LMW <3000 Daltons for the asphalt mixtures evaluated in this study. It is shown that the rejuvenating type recycling agents had the lowest percentage of HMW species and the highest percentages of LMW species followed by the neat asphalt binders, PG 52-28 and PG 70-22M. It is indicated that the PCWS followed by the MWS had the highest percentage of HMW species and the lowest percentages of LMW species. Figure 54 shows that as recycled materials (RAP and RAS) are added, regardless if a recycling agent is utilized in the asphalt mixture, there is an increase in the HMW species fraction and a decrease in the LMW species fraction. The increase in the HMW species fraction results in the mixtures being more brittle and more susceptible to fracture.

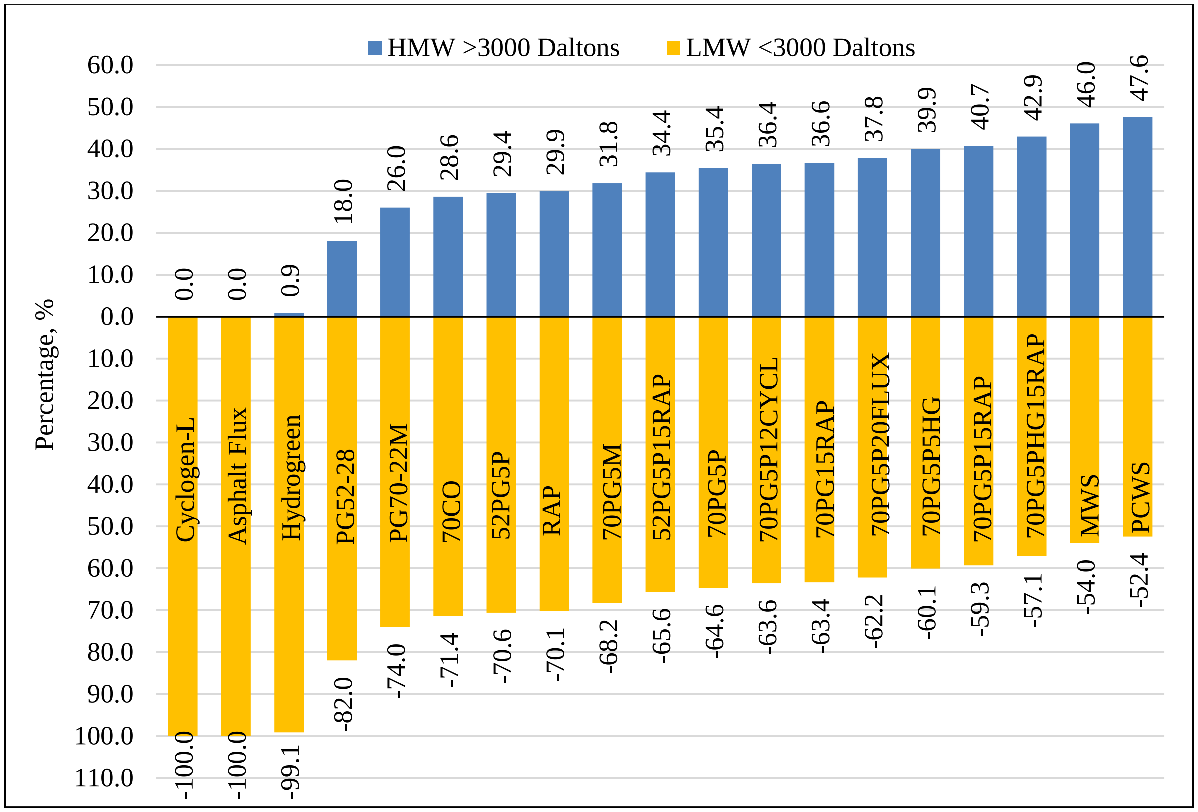


Figure 54
Percentages HMW >3000 daltons and LMW <3000 daltons per mixture type

Figure 55 shows the correlation between the Carbonyl Index, C=O, and the intermediate temperature performance parameter, J_c. The Carbonyl Index increases as an asphalt binder ages and increases in stiffness. As an asphalt binder oxidizes it becomes more brittle and stiffer. The complex shear modulus, G*, increases while the phase angle decreases. This results in a shift toward the loss modulus (plastic) and therefore, the binder is not as elastic before oxidation takes place. This figure indicates there is no correlation between the Carbonyl Index and J_c. However, it is noted that the trend shown is what would be expected. Figure 55 illustrates that as C=O increases the J_c decreases.

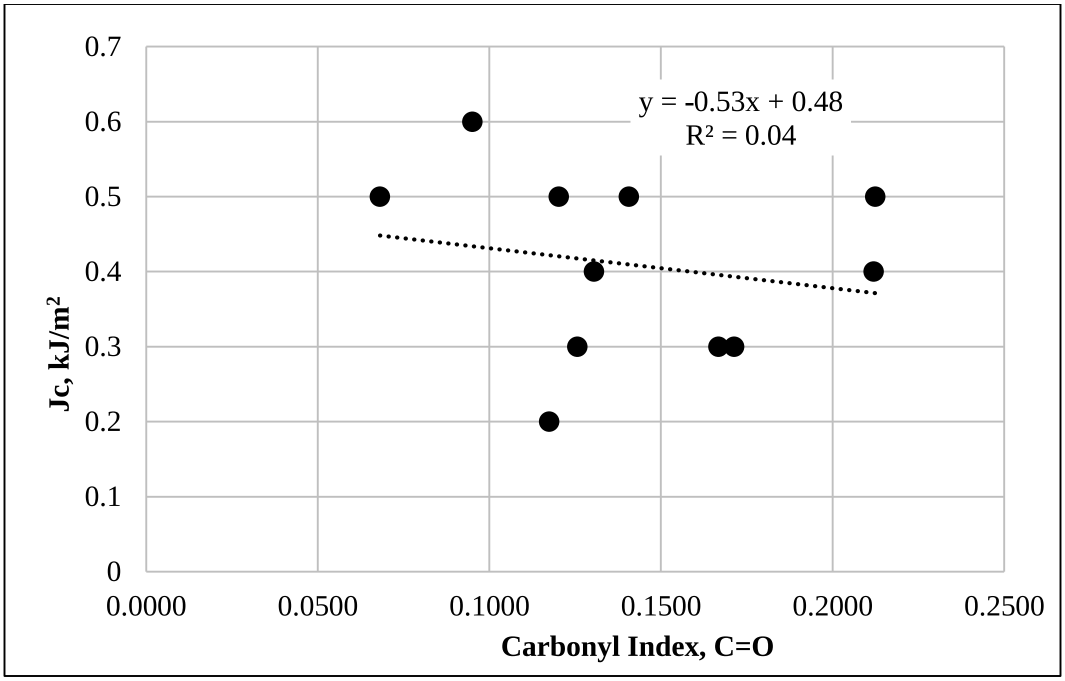


Figure 55
Carbonyl index correlation with J_c

Figure 56 indicates the carbonyl index as it relates to the critical strain energy release rate, J_c, from mixtures containing PG70-22M asphalt binder and 5% PCWS. It is shown that as the carbonyl index increases, the J_c increases. This is contrary to what is expected. It is anticipated that as the carbonyl increases, the J_c decreases. This expectation is because carbonyl index increases as an asphalt binder ages. As an asphalt binder ages the asphalt material becomes stiffer and brittle. This phenomenon results in a mixtures ability to resist intermediate temperature fracture.

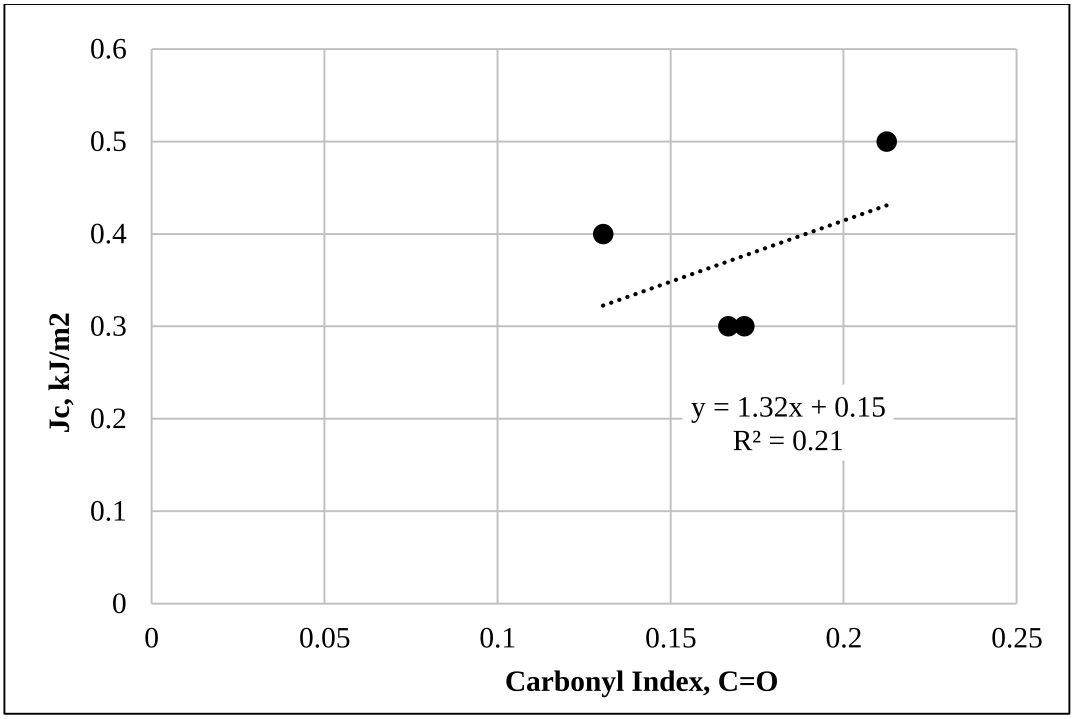


Figure 56
Carbonyl index correlation with J_c (mixtures containing PG70, and 5P)

Figure 57 illustrates the carbonyl index as it relates to the critical strain energy release rate, J_c, from mixtures containing PG70-22M asphalt binder, 5% PCWS, and 15% RAP. It is shown that there is a high correlation between the carbonyl index and the mixture J_c. Figure 57 indicates that as the carbonyl index increases the J_c decreases. This is the expected trend. When comparing Figure 56 and Figure 57, they indicate contrary results. Therefore, the use of carbonyl index as it relates to the critical strain energy release rate, J_c, is inconclusive for the mixtures evaluated. It is anticipated that the evaluation of additional mixtures should provide more conclusive results.

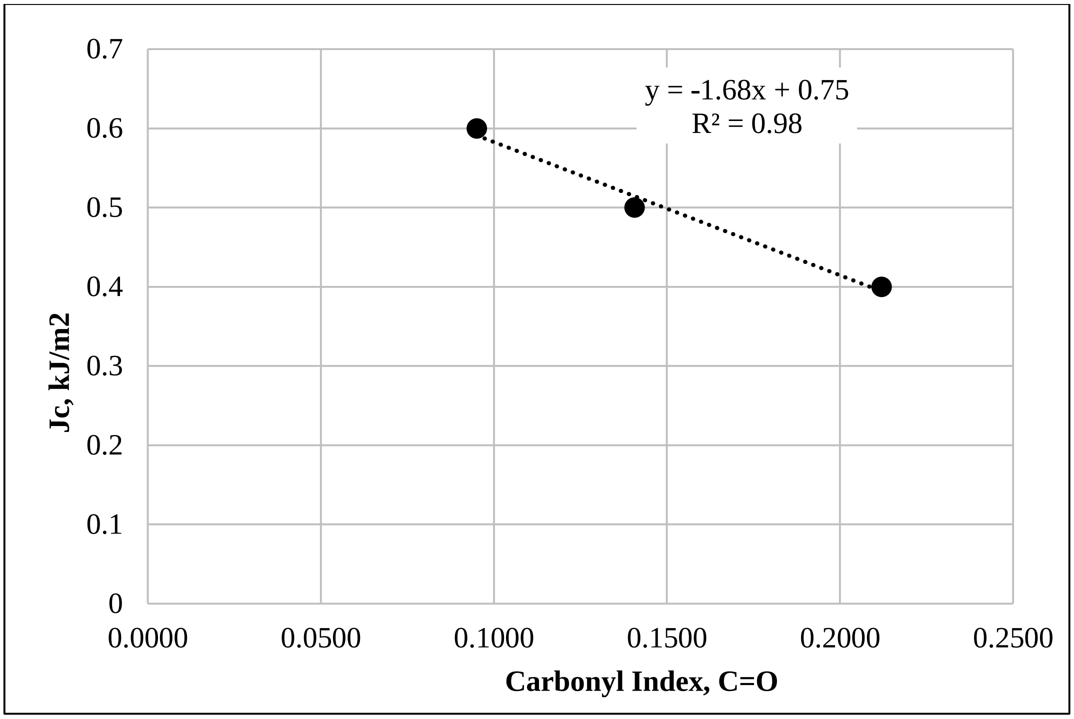


Figure 57
Carbonyl index correlation with J_c (mixtures containing PG70, 5P, and 15RAP)

Figure 58 indicates the GPC traces for mixtures 70PG15RAP, 70PG5P15RAP, and 70PG5PHG15RAP. Daly et al. indicated that the HMW threshold of larger than 19K Daltons relate to the stiffness of asphalt binders *[101]*. In comparing the molecular weights of mixtures 70PG5PHG15RAP and 70PG5P15RAP it is shown that mixture 70PG5PHG15RAP had 5.0% of its molecular structure greater than the 19K Dalton threshold. Whereas mixture 70PG5P15RAP had 1.3% greater than 19K Daltons. The corresponding J_c values for mixtures 70PG5PHG15RAP and 70PG5P15RAP were 0.4 and 0.5 kJ/m^2, respectively (Table 21). This would seem that the use of Hydrogreen did not improve the intermediate temperature performance even though Hydrogreen is considered a rejuvenator. It is however noted that the RBR for mixture 70PG5PHG15RAP is greater than mixture 70PG5P15RAP, 41.5 and 22.6 respectively (Table 15). In review of the MW at approximately 8000 Daltons as shown in Figure 58, it is indicated that mixture 70PG5P15RAP had the highest percentage of HMW species followed by 70PG5PHG15RAP and 70PG15RAP respectively. At the MW greater than 10K Daltons, it is shown that mixtures 70PG5PHG15RAP and 70PG15RAP have similar percentages. This would indicate that Hydrogreen did, in fact, rejuvenate portions of the higher molecular weight of RAS.

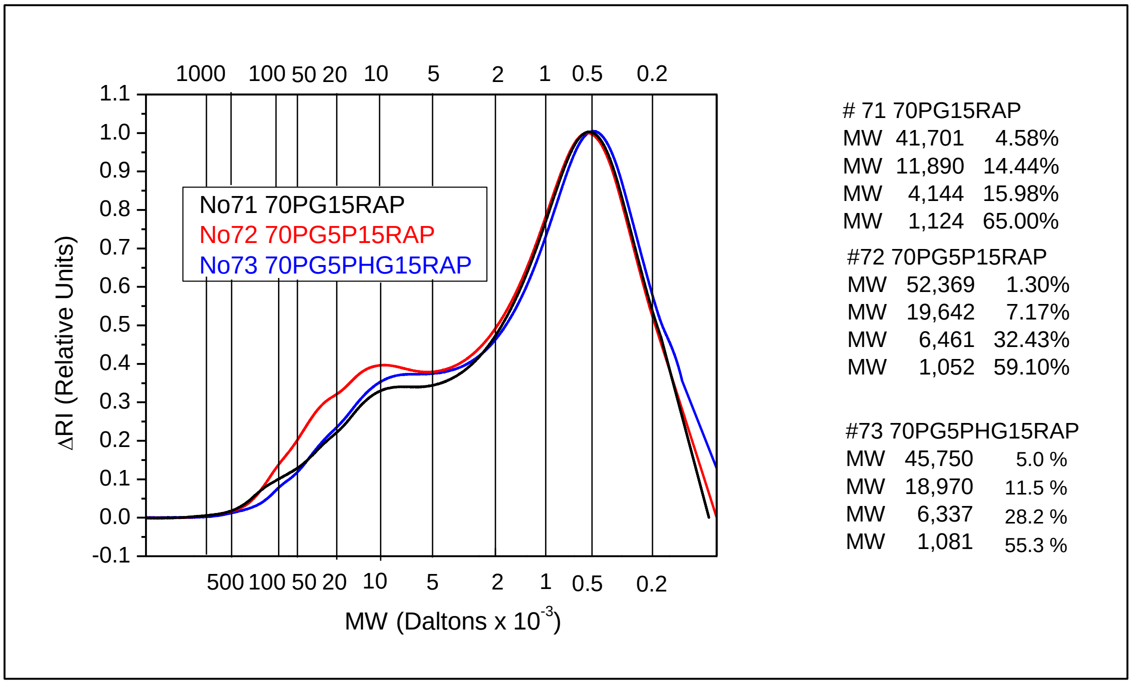


Figure 58
GPC traces of PG 70-22M containing RAP, RAS, RAP/RAS with or without HG

Low-Temperature Mixture Performance vs. Molecular Compositions

Figure 59 presents the relationships between the low-temperature cracking performance parameter and molecular composition of asphalt binders. It is generally indicated that as the asphaltenes content increases the TSRST fracture temperature rises and fracture work decreases. These trends are indicative of a more brittle asphalt mixture that would be more susceptible to low-temperature cracking development, Figures 59 (a) and (b). The superimposed linear trend lines show that the fracture temperature and fracture work done as measured and evaluated by TSRST have solid correlations with the asphaltenes content determined by GPC analysis, showing the R^2 values of 0.56 and 0.84, respectively. Similarly, correlations between each of the two mixture low temperature parameters (i.e., fracture temperature or fracture work) and various molecular compositional groups were determined. Table 23 summarizes the correlation analysis results.

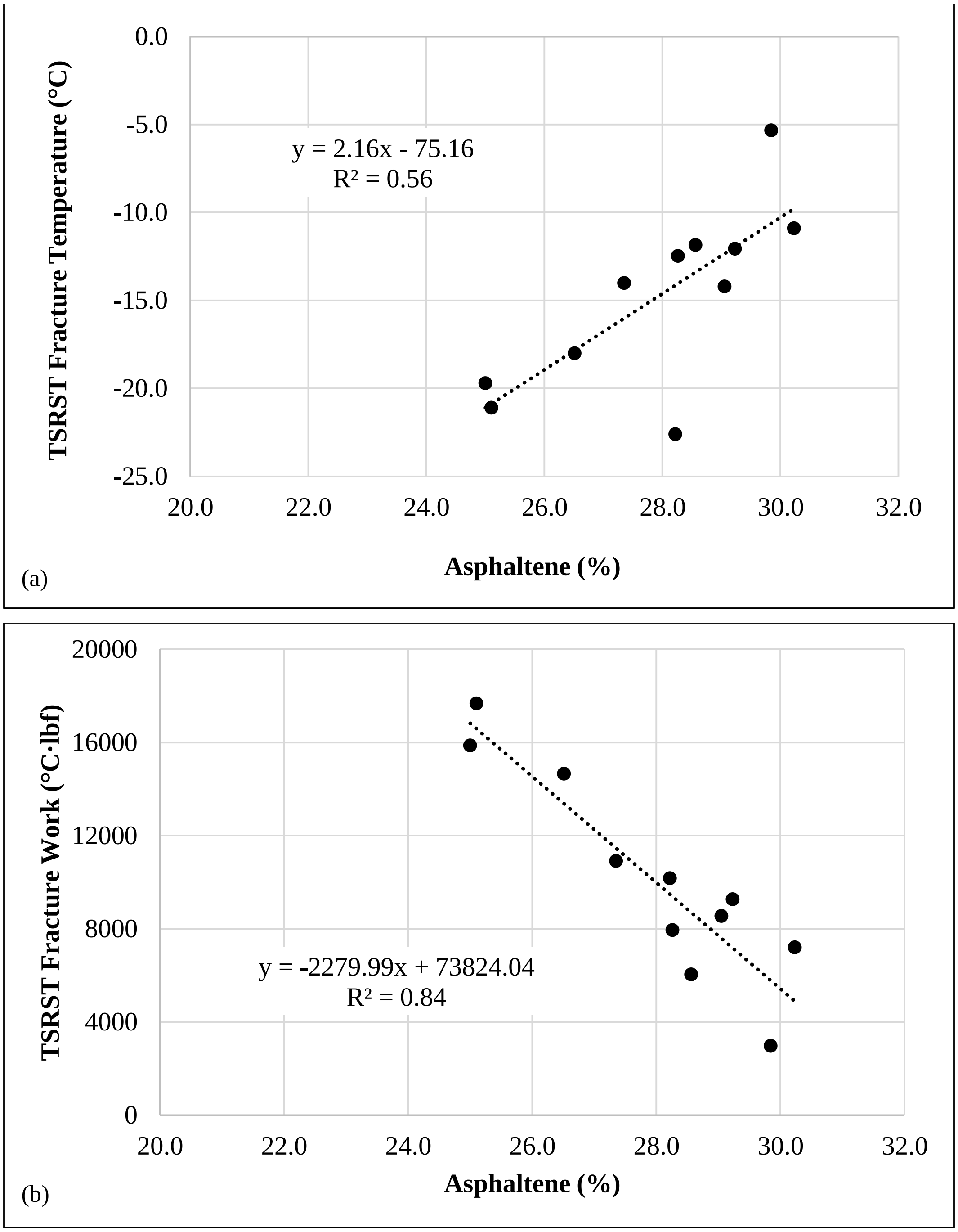


Figure 59
Mixtures' low-temperature cracking performance vs. %ssphaltenes: (a) fracture temperature and (b) fracture work

The total polymer content, asphaltenes, and maltenes determined from GPC analysis show good correlations with both fracture temperature and work done until fracture as compared to the four components (asphaltenes, resins, aromatics, and saturates) derived from SARA analysis. As discussed earlier, asphaltenes is generally regarded as the molecular component responsible for the stiffness and brittleness of asphalt binder and mixtures. Combining this result with an earlier observation from Table 14, it can be said that the addition of RAS and/or RAP into asphalt mixtures with and without RAs increases the formation of larger molecular weight species within the blended asphalt binder, which can considerably reduce the low-temperature cracking resistance of asphalt mixtures.

Table 23
R^2 values between dependent and independent variables

Dependent Variables	Independent Variables							
	Polymer (GPC)	Asphaltenes (GPC)	Maltenes (GPC)	Carbonyl Index	Asphaltenes (SARA)	Resins	Aromatics	Saturates
Fracture T (°C)	0.59	0.56	0.62	0.09	0.40	0.04	0.33	0.05
Work	0.64	0.84	0.77	0.39	0.40	0.11	0.17	0.02

SUMMARY AND CONCLUSIONS

Summary

The objective of this study was to assess the laboratory performance of conventional asphalt mixtures, mixtures containing RAP, RAS, RAP and RAS, with and without recycling agents (RAs), through laboratory measurements of mechanistic properties.

Comparative evaluations on the 11 asphalt mixtures were conducted, which include:

- a conventional Superpave asphalt mixture that contain PG 70-22M a, styrene-butadiene-styrene (SBS) modified asphalt binder, without RAS, RAP, and RAs as a control mixture;
- mixtures with PG70-22M binder vs. an unmodified PG 52-28 binder;
- mixtures with 5% MWS vs. with 5% PCWS;
- mixtures with 15% RAP vs. without RAP; and
- mixtures with RAs vs. without RAs.

A suite of laboratory mechanistic tests was conducted to characterize the high, intermediate, and low temperature properties for asphalt mixtures. Tests conducted include the dynamic modulus test for viscoelastic characterization, semi-circular bend (SCB) test for intermediate temperature fracture performance, and thermal stress restrained specimen tensile strength test (TSRST) for low temperature performance. A Hamburg type loaded wheel tracking (LWT) test was also performed to evaluate the mixtures' resistance to permanent deformation and moisture susceptibility. In addition, the molecular structure of asphalt binders of conventional asphalt mixtures as well as mixtures containing RAP, RAS, and both, with and without RAs were correlated with their intermediate and low temperature cracking potential using the SARA, GPC, and FTIR analyses results.

Conclusions

Based on the results of this study, the following conclusions can be drawn:

- With respect to the mixes without recycling agents, it is concluded that RAS binder does not fully blend with the virgin binder. The availability factor was found to range from 35 to 46%. Based on this fact, it was determined that the inclusion of RAS showed an improvement in rutting performance by resulting in a lower rut depth as compared to the control mixture without RAS. Further, because the RAS binder does not fully blend with the virgin binder, asphalt mixtures containing 5% recycled shingles showed no adverse effects to intermediate temperature properties (fatigue cracking) when compared to control mixture containing no RAS. In addition, the inclusion of 5% RAS did not adversely affect low temperature performance (thermal cracking) as compared to the control mixture. It was also determined that the addition of RAS did not adversely affect moisture susceptibility and no moisture susceptibility was predicted by LWT for the mixtures studied.

- In regards to the asphalt mixtures containing recycling agents, it was shown that RAS binder did blend with virgin binder when the mixtures were blended in accordance with the developed blending procedures. However, the availability factor was found to range from 50 to 100%. It was indicated that the addition of RAS with recycling agents generally showed an improvement in rutting performance by resulting in a lower rut depth as compared to the control mixture without RAS. The RAS mixture containing soft asphalt was the least resistant to permanent deformation. However, the inclusion of recycled shingles with recycling agents adversely affected the resistance to fracture at intermediate temperature even though the recycling agents are classified as rejuvenators. Further, the use of soft asphalt binders generally resulted in the least resistant to fracture at intermediate temperature. Also, RAS mixtures containing recycling agents adversely affected low temperature performance. It was also determined that asphalt mixtures containing RAS and recycling agents did not adversely affect moisture susceptibility and no moisture susceptibility were predicted by the LWT for the mixtures studied.
- In reference to the binder fractionation of the extracted asphalt binders from RAP/RAS mixtures with and without recycling agents, it was concluded that there were higher concentrations of high molecular weight species in the RAS binders as compared to the RAP binders. The concentration of the high molecular RAS species exceeds 40% in which 25% of these are highly aggregated with apparent molecular weights approaching 100K. In addition, the use of rejuvenating agents did not reduce the concentration of the very high molecular weight associated species, and thus they failed to improve the cracking resistance of the asphalt mixtures evaluated in this study. Also, it was shown that RAS is much more highly oxidized than RAP as indicated by FTIR spectroscopy. In addition, a relationship between the carbonyl index and fracture at intermediate temperature is inconclusive for the mixtures studied.
- The percentage of asphaltenes species fractionated from the SARA analysis was slightly less than that determined from the GPC analysis. The SARA asphaltenes analysis by precipitation did not capture the total amount of associated asphaltenes in the binder as measured by GPC. Some associated asphaltenes may remain in the resin fraction which is not captured by SARA analysis. The fracture temperature and fracture work measured by TSRST have good correlations with asphaltenes contents determined by GPC analysis. Similarly, the low-temperature cracking performance parameters have good correlations

with GPC determined polymer and maltenes contents, but showed considerably weaker correlations with SARA fractionated species. The addition of RAS and/or RAP, with and without RAs apparently increase the larger molecular weight species in the blended asphalt binder. In turn, the increased amount of larger molecular weight species adversely impacts the low-temperature fracture properties of asphalt mixtures and increase the likelihood of cracking in the asphalt pavement.

RECOMMENDATIONS

Based on the results, it is recommended that chemical analyses be performed on the asphalt binders containing recycled materials. It is recommended that SARA (Saturate, Aromatic, Resin and Asphaltenes) analysis be conducted to compliment GPC. While GPC has the capabilities to determine the molecular weight of species within a specimen, SARA analysis will divide the asphalt binder components according to polarity and polarizability. This will lend to a better understanding of the effects of RAS, RAP, and recycling agents on asphalt mixtures.

It is recommended that specifications for inclusion of RAS into mixtures be developed and experimental field projects be constructed. In doing so, the developed laboratory mixture design blending procedure can be validated. Also, asphalt mixtures from these field projects can be characterized to determine the effects of RAS on the high, intermediate, and low-temperature mixture properties. Furthermore, binder fractionation by molecular weight can be conducted on the extracted binders to further our understanding of the effects of RAS on mixture performance.

Also, it is recommended that an ALF (Accelerated Loading Facility) project be constructed. This will enable the evaluation of actual cracking and rutting under accelerated loading of mixtures containing RAS.

ACRONYMS, ABBREVIATIONS, AND SYMBOLS

ΔTc	Change in Critical Temperature
AASHTO	American Association of State Highway and Transportation Officials
BBR	Bending Beam Rheometer
C=O	Carbonyl Index
CAA	Coarse Aggregate Angularity
$\|E^*\|$	Dynamic Modulus
DOTD	Louisiana Department of Transportation and Development
DSR	Dynamic Shear Rheometer
FAA	Fine Aggregate Angularity
F&E	Flat and Elongated
FHWA	Federal Highway Administration
FTIR	Fourier Transform Infrared Spectroscopy
G*	Complex Shear Modulus
GPC	Gel Permeation Chromatography
G_{mb}	Bulk Specific Gravity of Mixture
G_{mm}	Maximum Specific Gravity of Mixture
G_{sb}	Bulk Specific Gravity of Aggregate
Gse	Effective Specific Gravity
Hz	Hertz
in.	Inch
ITS	Indirect Tensile Strength
J_c	Critical Strain Energy Release Rate
kJ/m^2	kilo-Joule per meter square
kPa	kilo Pascal
ksi	Kips per Square Inch
LAS	Linear Amplitude Sweep
LWT	Loaded Wheel Tracking
min	Minutes
MWS	Manufactured Waste Shingles
MPa	mega Pascal
mm	Millimeter
MSCR	Multiple Stress Creep Recovery
MW	Molecular Weight
NMAS	Nominal Maximum Aggregate Size
J_{nr}	Non-recoverable Creep Compliance

N_f	Number of Cycles to Failure
Pa·s	Pascal Second
%	Percent
PAV	Pressure Aging Vessel
PCWS	Post-consumer Waste Shingles
PG	Performance Grade
RA	Recycling Agents
RAP	Reclaimed Asphalt Pavement
RAS	Recycled Asphalt Shingles
RTFO	Rolling Thin-Film Oven
SARA	Saturate, Aromatic, Resin, and Asphaltene
SE	Sand Equivalency
sec.	Second
Tc(m)	Critical Low Temperature – m-value
Tc(S)	Critical Stiffness Temperature
TSR	Tensile Strength Ratio
TSRST	Thermal Stress Restrained Specimen Tensile Strength Test
U.S.	United States
Va	Air Voids
VFA	Voids Filled with Asphalt
VMA	Voids in the Mineral Aggregate

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APPENDIX

GPC Curves

COMPARATIVE DATA

71 70PG15RAP

MW 16,961 8.30%
MW 5,066 33.52%
MW 1,069 58.18%

#72 70PG5P15RAP

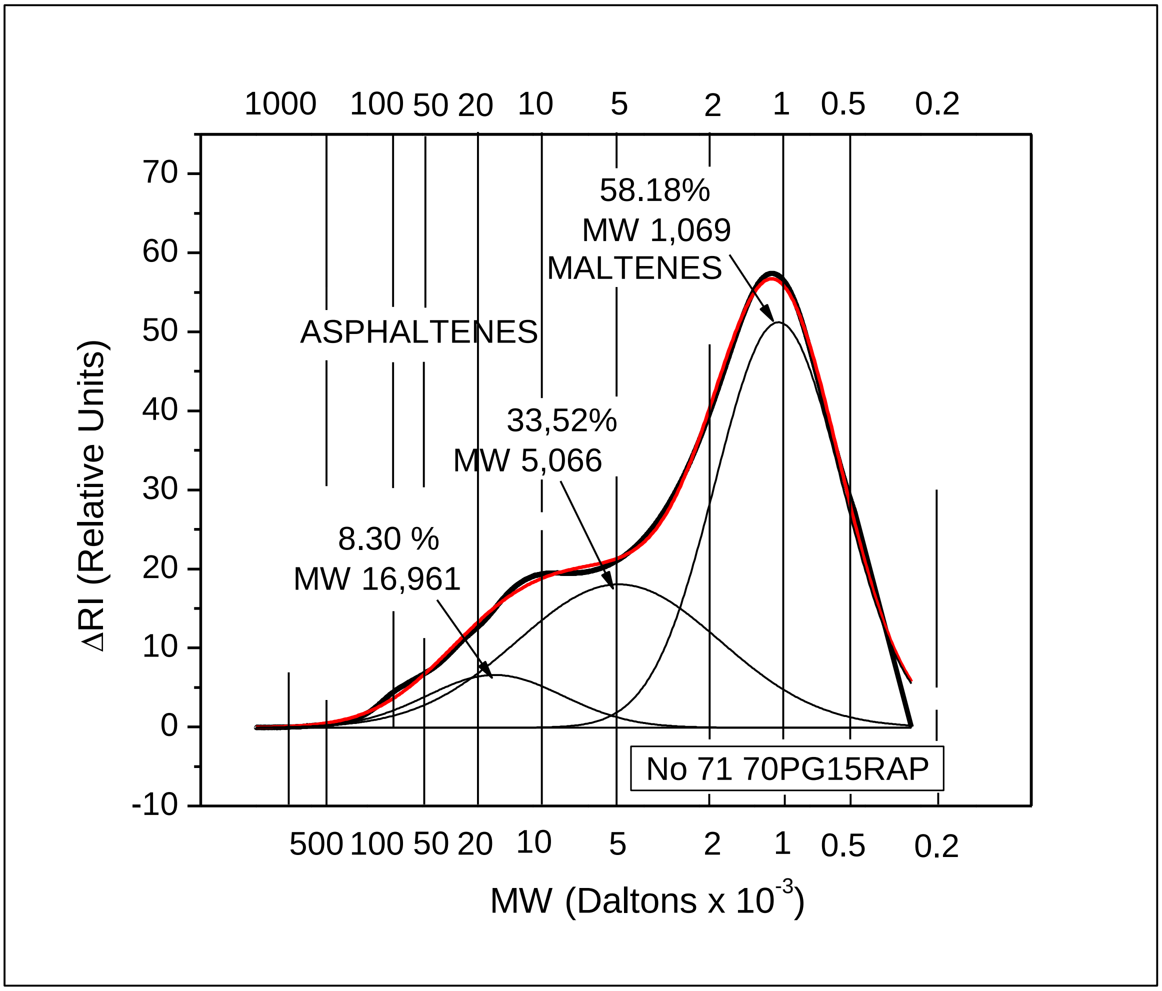
MW 32,571 5.70%
MW 12,945 7.17%
MW 3,691 41.88%
MW 1,052 45.25%

#73 70PG5PHG15RAP

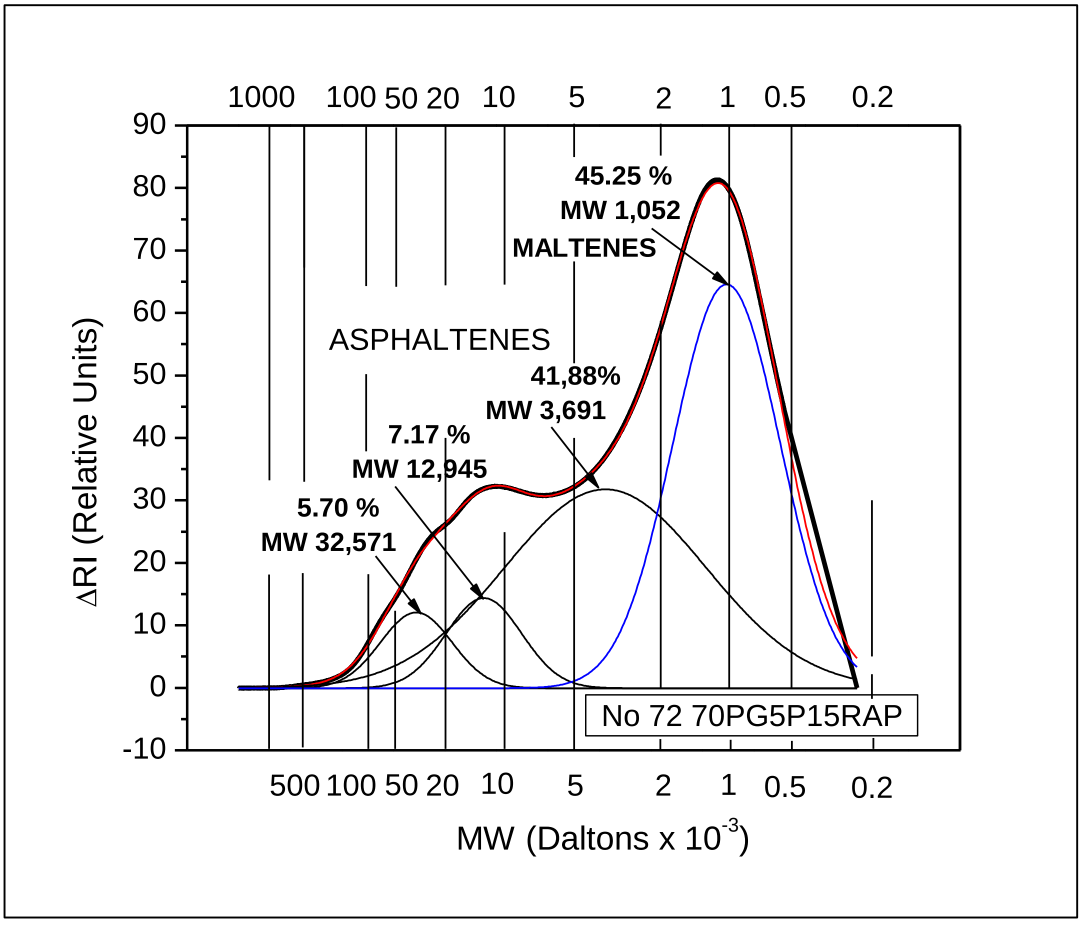
MW 45,750 5.0%
MW 18,970 11.5%
MW 6,337 28.2%
MW 1,081 55.3%

#74 52PG5P15RAP

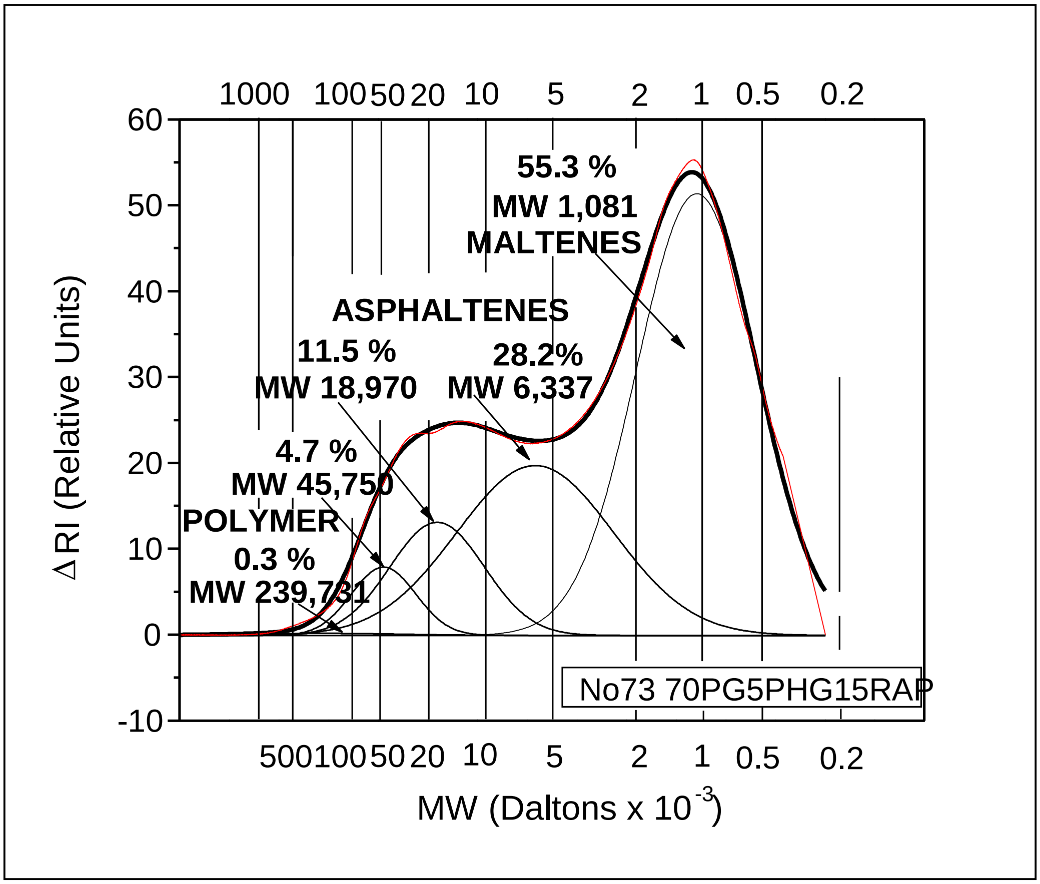
MW 93,814 0.06%
MW 21,137 3.85%
MW 7,815 29.27%
MW 1,069 66.82%



71 70PG15RAP
MW 16,961 8.30%
MW 5,066 33.52%
MW 1,069 58.18%

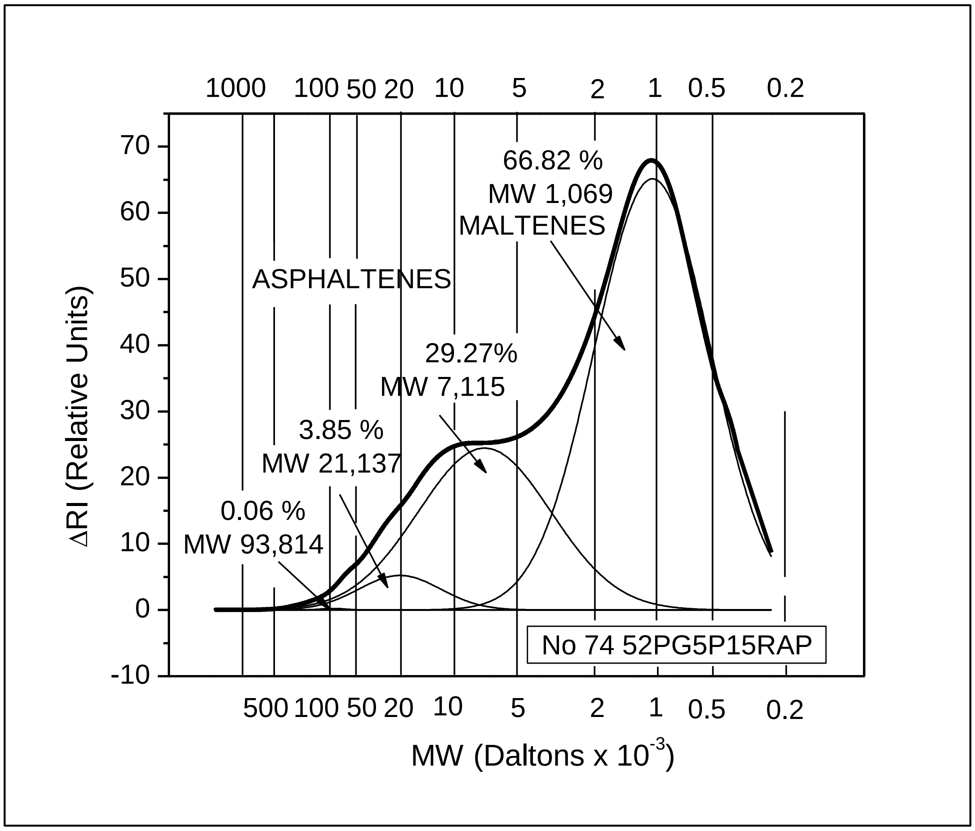


<u>#72 70PG5P15RAP</u>
MW 32,571 5.70%
MW 12,945 7.17%
MW 3,691 41.88%
MW 1,052 45.25%



<u>#73 70PG5PHG15RAP</u>

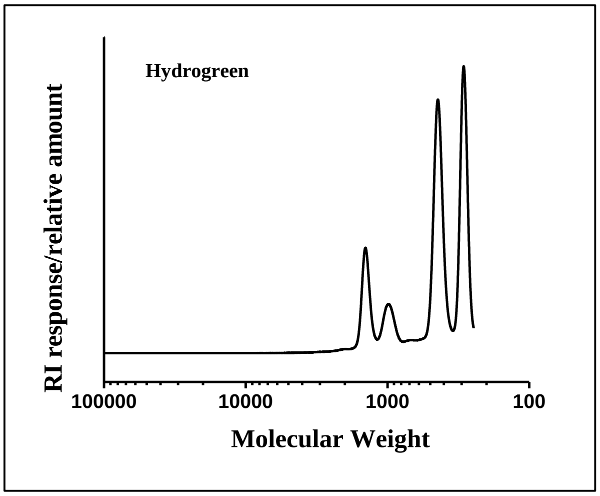
MW	45,750	5.0%
MW	18,970	11.5%
MW	6,337	28.2%
MW	1,081	55.3%



#74 52PG5P15RAP

MW 93,814 0.06%
MW 21 ,137 3.85%
MW 7,115 29.27%
MW 1,069 66.82%

#74 52PG5P15RAP	#72 70PG5P15RAP
MW 93,814 0.06%	--------
MW 21,137 3.85%	MW 32,571 5.70%
MW 7,815 29.27%	MW 12,945 7.17%
------------	MW 3,691 41.88%
MW 1,069 66.82%	MW 1,052 45.25%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RI response/relative amount
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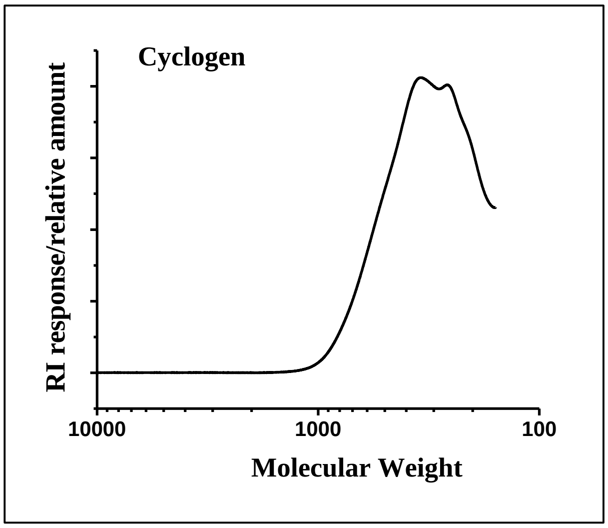
10000
1000
100
Molecular Weight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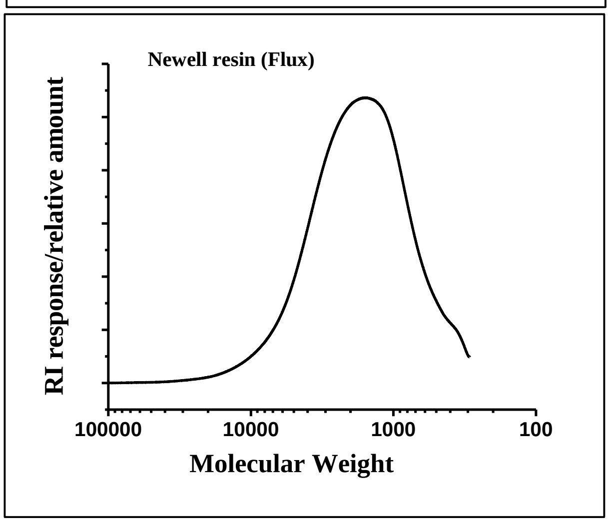
Newell resin (Flux)
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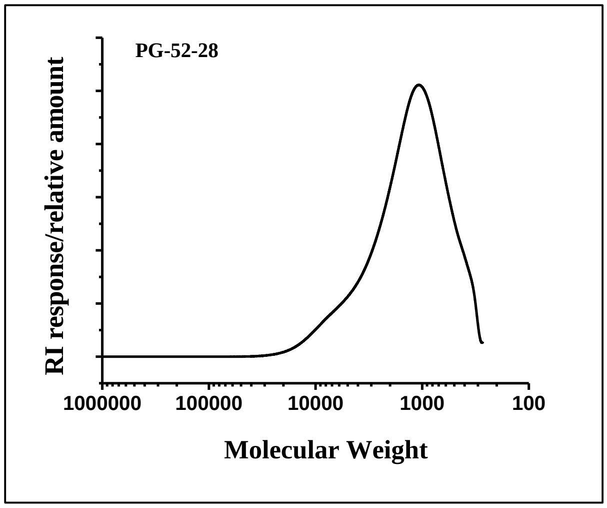
Molecular Weight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RI response/relative amount
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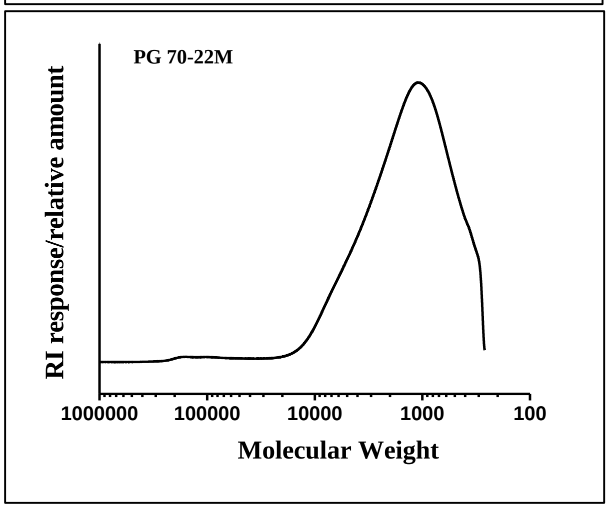
Molecular Weight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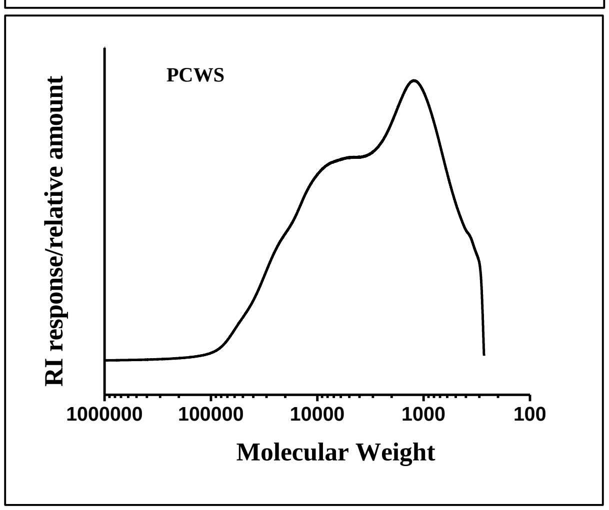
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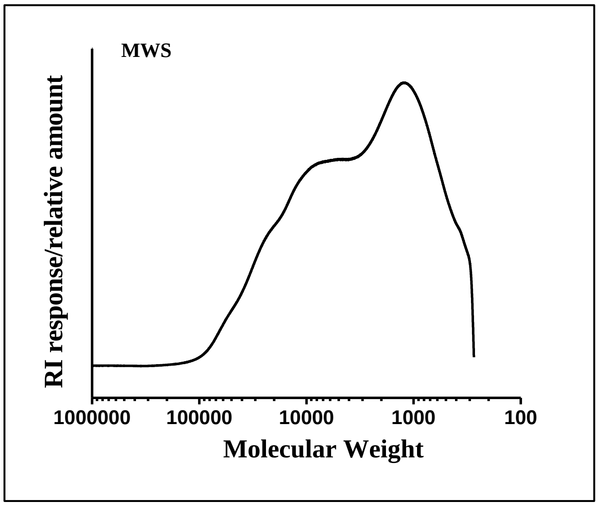
Molecular Weight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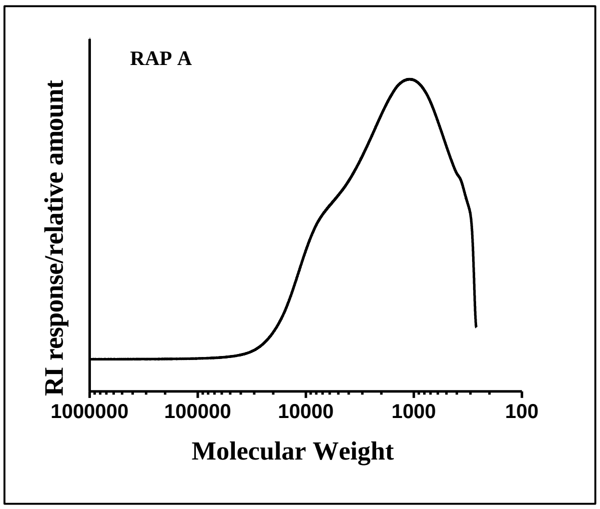
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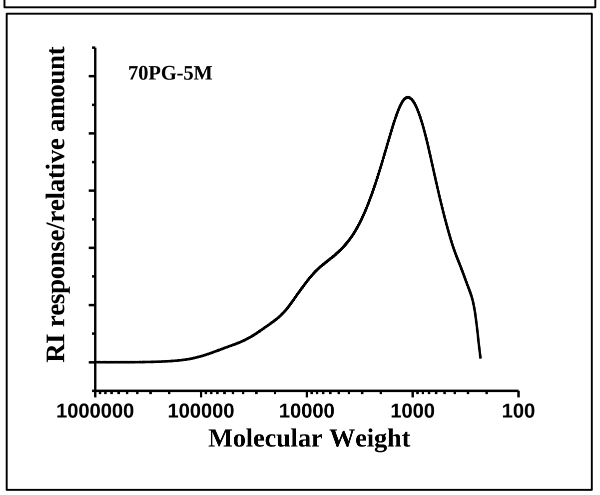
Molecular Weight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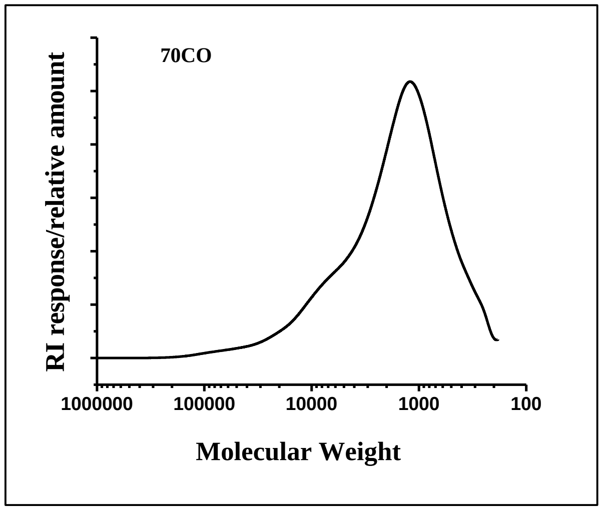
Molecular Weight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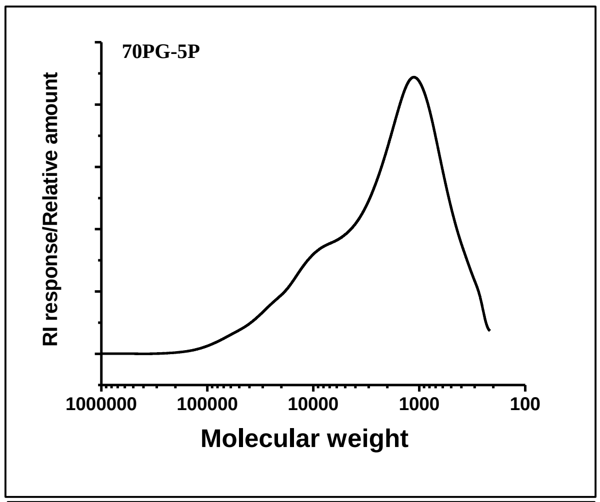
Molecular Weight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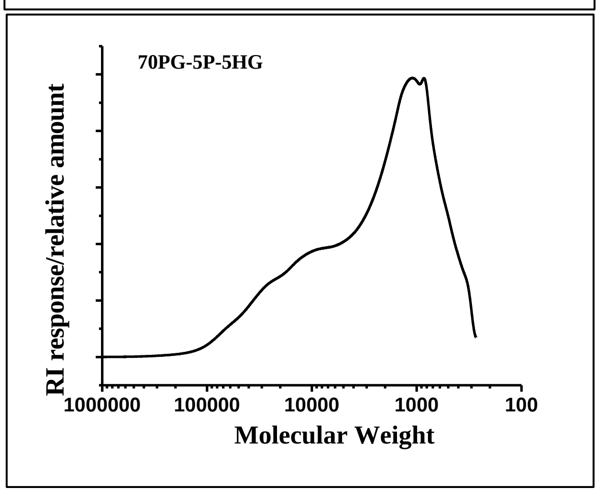
Molecular weight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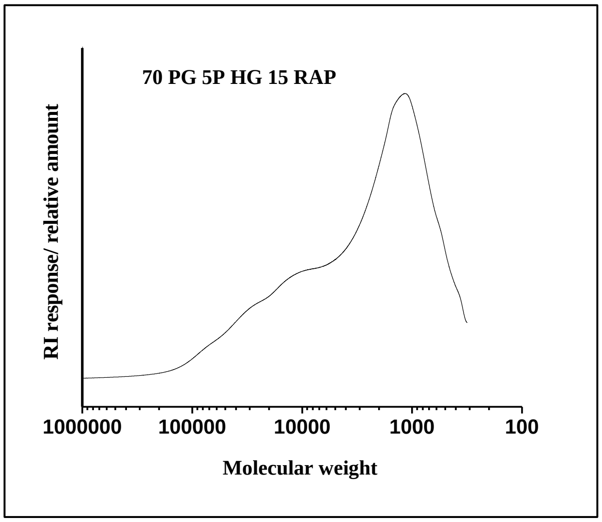
Molecular Weight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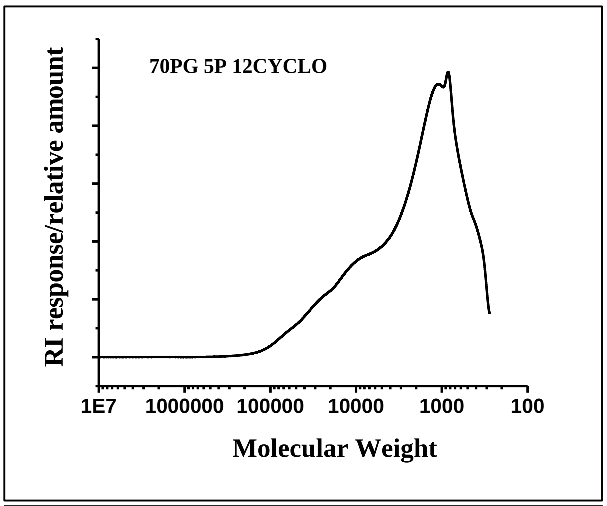
Molecular weight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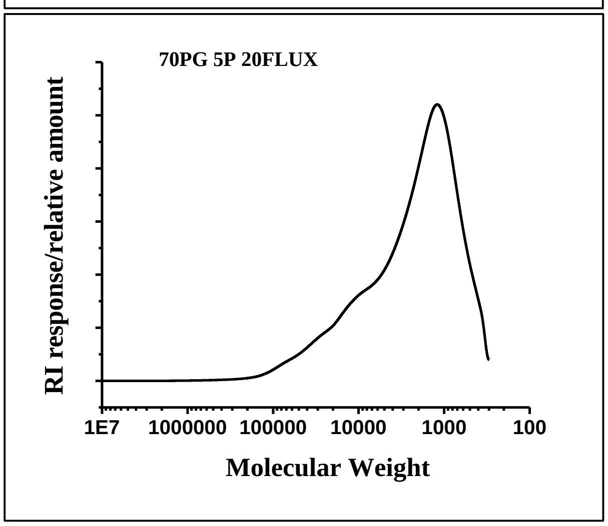
Molecular Weight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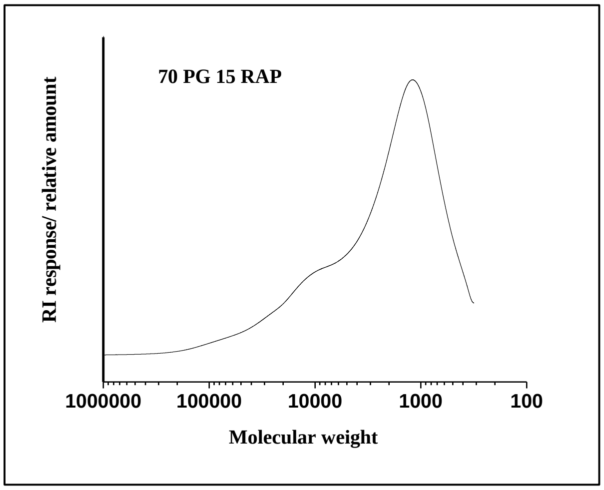
Molecular Weight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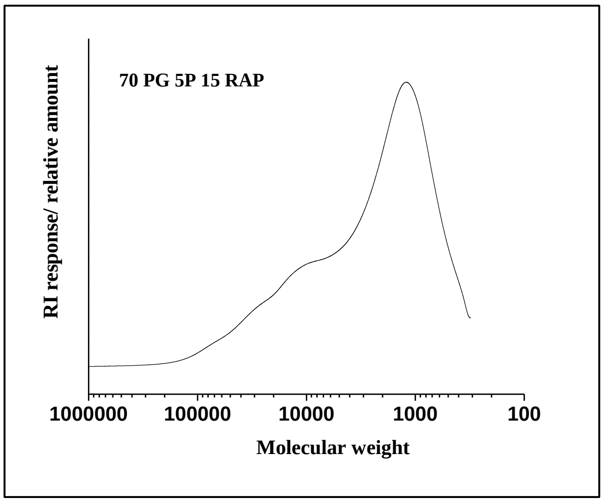
Molecular weight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RI response/ relative amount
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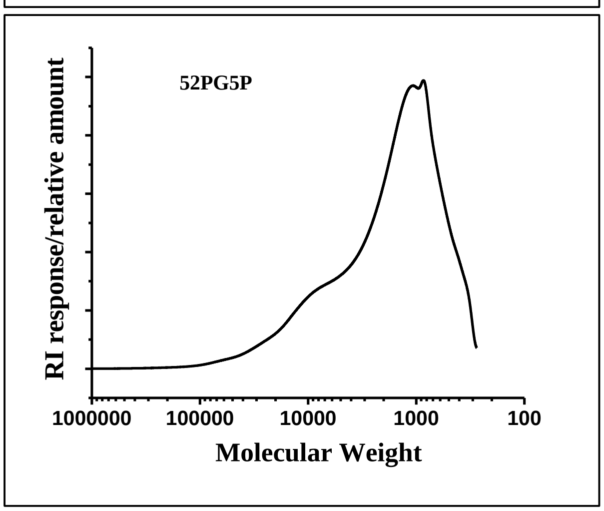
Molecular weight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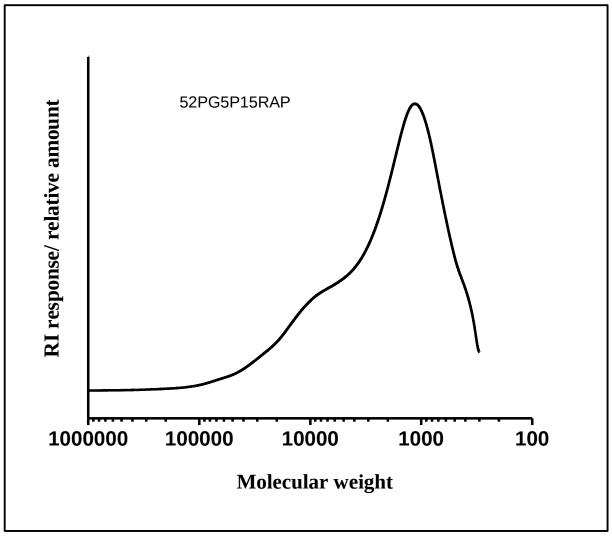
Molecular Weight



52PG5P15RAP
RI response/ relative amount
1000000
100000
10000
1000
100
Molecular weight

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