CHARACTERIZATION OF ASPHALT BINDER EXTRACTED FROM RECLAIMED ASPHALT SHINGLES

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DISCLAIMER

1. Report No. NCAT Report 16-01	2. Government Accession No.	3. Recipient's Catalog No.

4. Title and Subtitle CHARACTERIZATION OF ASPHALT BINDER EXTRACTED FROM RECLAIMED ASPHALT SHINGLES	5. Report Date January 2016
	6. Performing Organization Code
7. Author(s) J. Richard Willis, Ph.D. and Pamela Turner	8. Performing Organization Report No.
9. Performing Organization Name and Address National Center for Asphalt Technology 277 Technology Parkway Auburn, AL 36849	10. Work Unit No. (TRAIS)
	11. Contract or Grant No. G00007328
12. Sponsoring Agency Name and Address Federal Highway Administration 1200 New Jersey Avenue, SE Washington, DC 20590	13. Type of Report and Period Covered Final Report 2013-2015
	14. Sponsoring Agency Code

15. Supplementary Notes

FHWA Contracting Officer's Representation: Christopher Wagner

16. Abstract

An 80% increase in the amount of RAS used in asphalt mixtures was reported from 2009 to 2012. Despite this increase, there is still little guidance given on the characterization of RAS binder in AASHTO MP 023 and PP 078. In addition to the lack of direction, many contractors and owner agencies do not have equipment capable of determining the actual high and low temperature performance grades of the RAS binder; therefore, work needs to be completed which can aid owner agencies and contractors in determining the true PG grades of RAS binder. While a common virgin binder is PG 64 – 22, RAS binders are much stiffer with critical high temperature grades between 140 and 180°C and critical low temperature grades between 0 and 40°C. If this stiffer binder is not considered in design, it can negatively influence fatigue and thermal cracking performance.

The objective of this research is to investigate methods of characterizing RAS asphalt binder for both the critical high and low temperatures. Binder was extracted from RAS and tested to determine the true (or measured) PG grade of the binder. In addition to direct measurement, extrapolation methods were assessed to determine appropriateness in case equipment was not available for direct measurement. Finally, within sample and between sample testing variability was quantified for RAS binders using conventional testing methodology. These tests were completed on RAS samples from across the U.S. and included both post-consumer (PC) and manufacturers' waste (MW) RAS.

Ultimately, it was determined that one could extrapolate the critical high temperature grade of RAS binders ensuring that variability and outliers were considered in the analysis; however, less repeatable and reliable results were discovered when assessing critical low temperatures.

17. Key Words SHINGLE, BINDER, VARAIBILITY, PERFORMANCE GRADE	18. Distribution Statement No restriction. This document is available at www.ncat.us.

19. Security Classif. (of this report) Unclassified	20. Security Classif. (of this page) Unclassified	21. No. of Pages 39	22. Price

Form DOT F 1700.7 (8-72) **Reproduction of completed page authorized**

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1 INTRODUCTION

1.1 Background

Engineers began using reclaimed asphalt shingles (RAS) in pavement mixtures as an alternate asphalt source in the 1980s and 1990s (1). When polymer modification of asphalt binders became more common in the Superpave Performance Grading (PG) specifications, practitioners began to search for ways to reduce asphalt binder cost while still receiving the desired level of performance. This encouraged the asphalt pavement industry to use recycled products such as reclaimed asphalt pavement (RAP), ground tire rubber (GTR), and RAS in their mixtures.

While products like GTR serve as an asphalt modifier by easing the industry's dependence on the supply of polymers (such as styrene-butadiene-styrene), RAP allows contractors to replace both aggregate and asphalt binder while RAS allows contractors to replace asphalt (primarily). Agency, industry, and academia have worked diligently and successfully to successfully increase RAP percentages on a national basis. Successful RAS utilization is still developing and is the primary reason for this report.

Nearly 11 million tons of waste shingles are produced each year, resulting in approximately 22 million cubic yards of waste material needing to be landfilled (2). Environmental standards have evolved since the 1980s, forcing disposal sites to either close or limit the amount of asphalt shingles they can accept. Between 1980 and 1997, more than 11,000 RAS disposal sites closed, causing tipping fees to escalate to near $100 per ton (1, 3, 4). Asphalt shingles account for approximately 8 to 10 percent of the annual building-related waste and construction debris produced in the U.S. annually and is the third largest building waste material in the world (5).

In addition to reducing the building waste, using just one ton of RAS to replace aggregate and asphalt in a new mixture can reduce the carbon footprint of the asphalt paving industry by 55 tons of carbon (6) Thus, using RAS in asphalt mixtures, in effect, reduces both the fiscal and environmental costs of asphalt pavement being produced. RAS has also been shown to improve the performance of asphalt mixtures in terms of rut resistance, stability, and high temperature susceptibility (7); however, the improper use of RAS (i.e. either using too much, too coarse a grind (size of shingle), or not using soft PG asphalt) has been shown to cause premature failures in some pavements.

An 80% increase in the amount of RAS used in asphalt mixtures was reported from 2009 to 2012 (8). Despite this increase, there is still little guidance given on the characterization of RAS binder in AASHTO MP 023 and PP 078. In addition to the lack of direction, many contractors and owner agencies do not have equipment capable of determining the actual high and low temperature performance grades of the RAS binder; therefore, work needs to be completed which can aid owner agencies and contractors in determining the true PG grades of RAS binder. While a common virgin binder is PG 64 – 22, RAS binders are much stiffer with critical high temperature grades between 140 and 180°C and critical low temperature grades between 0 and 40°C. If this stiffer binder is not considered in design, it can negatively influence fatigue and thermal cracking performance.

1.2 Objectives and Scope

The objective of this research is to investigate methods of characterizing RAS asphalt binder for both the critical high and low temperatures. Binder was extracted from RAS and tested to determine the true (or measured) PG grade of the binder. In addition to direct measurement, extrapolation methods were assessed to determine appropriateness in case equipment was not available for direct measurement. Finally, within sample and between sample testing variability was quantified for RAS binders using conventional testing methodology. These tests were completed on RAS samples from across the U.S. and included both post-consumer (PC) and manufacturers' waste (MW) RAS.

2 LITERATURE REVIEW

Asphalt roofing materials include composition shingles, built-up roofing, and torch down roofing (a polymer-modified asphalt membrane strengthened with fabrics and commonly used on flat roofs). The major components of asphalt roofing waste include asphalt, mineral filler and granules, glass fiber matting, organic paper felt, and nails. There are a number of potential end usages for asphalt roofing waste, including asphalt mixtures, which is currently the largest market for RAS (9).

Asphalt shingles contain at least two products needed in asphalt mixture production: asphalt binder and fine crushed aggregate. They also produce fibers (10-20% by weight) that may be useful in certain types of asphalt mixtures. RAS contains approximately 19 to 36% asphalt binder by weight. In addition, the granules in the shingles (approximately 20-38% by weight) are a source of aggregate used in asphalt pavement mixtures.

A number of laboratory and field-scale research studies have been conducted to evaluate the use of asphalt shingles in hot mix asphalt (HMA) and stone matrix asphalt (SMA). Some of these benefits include the following (10):

- Reduced demand for virgin asphalt binder and aggregate,
- Significant reduction to purchased MF for SMA mixtures,
- Improved resistance to rutting due to the reinforcement provided by fibers contained in shingles, and
- Reduced production cost of HMA.

The asphalt binder in RAS decreases the demand for virgin asphalt cement and provides several benefits to both the industry and state agencies. First, recycled binder from RAS reduces the cost of the asphalt binder needed depending on the state and/or source. Waste from shingle factories can be processed and immediately be added to the hot mix asphalt process or renewed with rejuvenating chemicals prior to the mix process. Secondly, asphalt mixtures require specific aggregate gradations with specific durability properties. The mineral or ceramic aggregate in RAS provides a source of fine aggregate and reduces the demand for virgin aggregate; however, this reduction of aggregate is small. Finally, certain properties of asphalt pavement have been shown to improve with the addition of recycled asphalt shingles. These include rutting resistance (11).

While the composition of shingles varies depending on the manufacturer and roofing application, most are composed of four basic materials: asphalt cement, felt or fiber, mineral or ceramic aggregate, and mineral filler. Organic or fiberglass felt backings form the basic structure for shingles. The organic felt is typically composed of either cellulose or wood fibers and is designed to support the asphalt and aggregate granules. Fiberglass backings are manufactured by mixing fine glass with water in the form of a glass pulp that is then formed into a fiberglass sheet (12, 13). The backing is then saturated with asphalt cement.

Shingle asphalts are “air blown,” which increases their stiffness when used in asphalt mixtures compared to conventional paving asphalt. They are further stabilized with a filler (70% passing the #200 sieve) (10, 14). A second application of “air blown” asphalt is applied as a covering for both sides of the shingle. The top side of the shingle is then covered with granules designed to protect the asphalt from both the sun’s ultraviolet rays and physical damage due to abrasion on rooftops. Most shingle manufacturers use a combination of crushed rocks coated with ceramic metal oxides as granules. Additional headlap granules can be used in this application. Both aggregate granules are ideal for roofing shingles due to their uniform size, toughness, and angular shapes (13). In some cases, chemicals are also added to the aggregate to prevent algae growth (14).

Shingles are finished with a dusting of fine sand to the back surface to prevent the agglomeration of the shingles that could occur during transportation. A schematic of the final product is shown in Figure 1. Table 1 presents estimates of the percentage of each material in organic and fiberglass shingles.

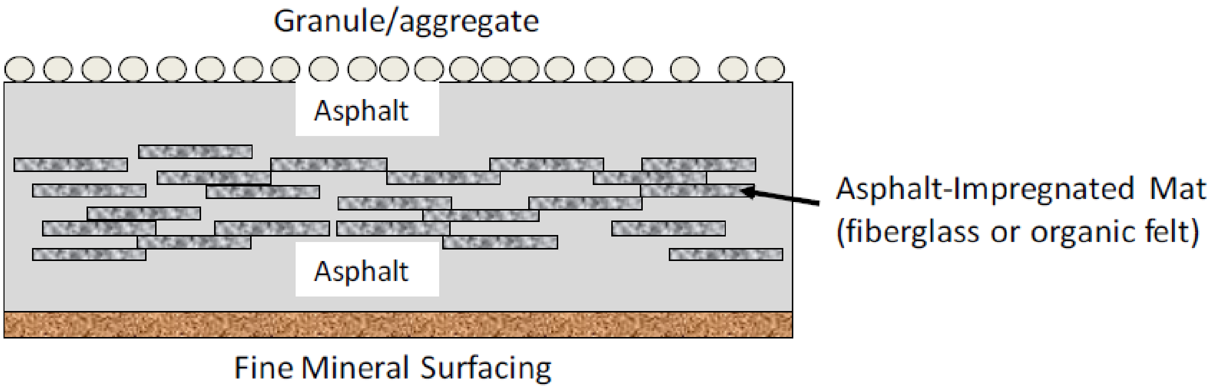


Figure 1 Schematic of asphalt shingle composition (10).

Table 1 Composition of Shingles (14, 15, 16)

Component	**Organic Felt**	**Fiberglass Mat**
Asphalt Cement	30-36%	19-22%
Felt (Fiber)	2-15%	2-15%
Mineral Aggregate	20-38%	20-38%
Mineral Filler	8-40%	8-40%

Just as there are differences between organic and fiberglass shingles, there are also differences in the material composition based on shingle source. Loss of aggregate particles in the PC shingles generally causes higher asphalt content than the MW shingles. Exposure to contaminants also causes PC shingles to contain more deleterious materials than MW shingles such as paper, wood, and nails. While many of these contaminants are removed during the grinding process, further removal of deleterious materials may be necessary before RAS can be used in asphalt mixtures (13).

An understanding of each component in RAS and how it can contribute to an asphalt mixture is essential. PC shingle stockpiles tend to exhibit more variability than MW shingles in size, aggregate gradation, and asphalt content as well as material properties such as specific gravity. Shingle type, manufacturer, and age can significantly influence these factors (17). Currently, PC RAS is used more frequently than MW RAS because there is 10 times the amount of PC shingles available for use.

2.1 RAS Asphalt Binder

The desire to use RAS in asphalt mixtures stems from the ability to substantially reduce the overall material cost by replacing virgin asphalt binder with reclaimed asphalt in a pavement mixture. In order for this to be cost-effective, the asphalt content of the RAS for mixture proportioning and its performance grade (PG) must be quantified to ensure mixture performance.

Since RAS asphalt have been air blown, they are inherently stiffer than virgin or modified asphalts and have different rheological properties. This stiffness causes many state agencies to be concerned about long-term durability of mixtures containing RAS in relationship to cracking and stripping. (3, 18, 19). One example of this was seen at the 2012 NCAT Test Track, in test sections designed for the Florida Department of Transportation. Two comparative sections were designed using PG 76-22 base binders. The first section had 20 percent RAP without any RAS, and the second section contained 20 percent RAP with an additional 5 percent RAS. The section which included RAS had significantly more low severity top-down cracking than the RAP only section (20).

The most common tool for assessing asphalt binders today is AASHTO M320, *Standard Specification for Performance Grade Binder Grading*. Many states do not require the RAS binder to be performance graded due to the difficulty in handling and testing. In order to use higher shingle or binder replacements, AASHTO PP 78, *Design Considerations When Using Reclaimed Asphalt Shingles (RAS) in Asphalt Mixtures*, requires users to know the PG of the RAS binder to complete blending charts used to determine the correct virgin binder grade to be used in the mix design. Despite potential handling and testing difficulties, it is important that blended RAS asphalt and virgin binder meets the same performance criterion as virgin binder in terms of binder grade, strength, and durability (21).

The Performance Grade of RAS binders is determined using AASHTO M320 on materials that have been extracted using methods previously mentioned and then recovered using ASTM D5404. In the current performance grading system, the stiffness and elasticity properties of the asphalt binder are evaluated at three aging levels. First, the original, unaged binder is assessed. Second, the asphalt binder undergoes a simulated short-term aging in the rolling thin film oven (RTFO) before being assessed again. The final assessment occurs after a simulated long-term aging in the pressure aging vessel (PAV). Current mix design and binder grading specifications do not require RAP binders to undergo long-term aging since they have already been aged in the field, but they do undergo short-term aging.

While this provision is given for RAP binders, no such guidance is provided for RAS binders. It is known that air blowing during shingle production increases the binder viscosity. The binder of PC shingles is further aged while the shingle is acting as a roofing material. Still, researchers have presented results using both the RTFO and PAV aging procedures (21).

Agencies have reported difficulty working with recovered RAS binders due to their extreme stiffness at normal binder handling temperatures and the small mold shapes required for some binder testing (21). Additionally, common DSR models using water baths for temperature control cannot be used to directly assess the high temperature grade of the RAS binder since many RAS binders have critical high temperatures above the boiling point of water (18).

In many cases, RAS binders also are difficult to test for critical low temperatures, as the binders must proceed below -36°C to reach the critical low temperature stiffness but have m-values that will only pass at temperatures greater than 0°C (18). This difference in low temperature failure values indicates that recovered RAS binders are extremely brittle, making the creation of test specimens for low temperature testing difficult. Agencies have reported using cheese graters to shred RAS binder so it could be PG graded (22). Example performance grades of shingle binders are presented in Table 2 to show the difficulties and extreme critical temperatures determined when assessing RAS binders. While this list of binder grades is not exhaustive, one commonly sees that PC RAS binder is much stiffer than MW RAS binder when reviewing low PG grades for the RAS.

Table 2 RAS PG Grades

Reference	Location	RAS Type	High PG	Low PG
23	Oregon	MW	134	NA
18	Wisconsin	MW	124-154	0>
22	Missouri	MW	143+	0>
24	Minnesota	MW	134-153.9	(-12.7) to (-6.1)
24	Minnesota	PC	121.2 – 133.1	(-6.9) to (10.6)
NCAT	Alabama	PC	175.4	41.7
NCAT	Alabama	MW	132.6 – 137.2	(-18.6) to (-13.0)

3 MATERIALS AND TEST METHODS

3.1 Materials

Six RAS samples were obtained to evaluate the high temperature performance of RAS binder, and four were supplied for low temperature evaluation (Table 3). These samples were sent to NCAT from contractors who volunteered to provide material. Some of the samples were not large enough to conduct the full suite of testing; therefore, the samples were either used for high temperature or low temperature testing. The research team tried to receive samples from different geographic regions of the country that commonly use RAS as well as samples from both PC and WM shingle types. The same of RAS from Oregon was a blend of both PC and MW RAS instead of just a single RAS source.

Table 3 RAS Samples

State	Shingle Type		Temperature Testing	
	MW	*PC*	*High*	*Low*
Michigan	✓		✓	
		✓	✓	
Oregon	✓	✓	✓	✓
New Hampshire		✓	✓	✓
Georgia		✓	✓	
Texas	✓		✓	✓
Wisconsin	✓			✓

3.2 Test Methods

3.2.1 Extraction and Recovery

Asphalt binder was extracted and recovered from each RAS source using the centrifuge extraction method (ASTM D2172-05 Method A) with Trichloroethylene (TCE) as the solvent. Other solvents are used by other agencies, and the Asphalt Institute is currently conducting some work to compare the effects of using different solvents on RAS binder testing results. Some states do not use TCE as a solvent because of the limited availability and environmental concerns associated with its use.

The rotary evaporator recovery procedure (ASTM D5404-03) was used to remove the solvent from the extracted binder. Due to the high viscosity of the recovered RAS binder, it was necessary to increase the drain time and temperature used for removing the recovered binder from the recovery flask. For this testing, a total drain time of 30 minutes at a temperature of approximately 190°C was used which might cause RAS stiffening at this point of testing. Three extraction/recovery procedures were performed for each RAS source with the recovered binders from each procedure being stored in separate containers for high temperature testing. A fourth extraction/recovery procedure was performed for each RAS source to obtain recovered binder for low temperature testing.

3.2.2 Binder Testing Methods

The recovered RAS binders were tested in accordance with the procedures described in AASHTO M320-10, AASHTO R29-08, and AASHTO D7643-10 to determine high and low performance grades and continuous grading temperatures. True grading temperatures as described in ASTM D7643 - 10 are defined as the temperature at which a test criterion is met (for example, the continuous grading temperature for RTFO-aged material tested in the DSR is the temperature at which $G^*/\sin(\delta)$ = 2.20 kPa) and are usually determined by interpolating between a passing and failing test result.

There is no official guidance given regarding aging of recovered binders except for AASHTO M323-13, Appendix X1, *Standard Specification for Superpave Volumetric Mix Design*, which states that the recovered binder from RAP should only be short-term aged in the Rolling Thin Film Oven (RTFO) prior to testing. An attempt at aging recovered RAS binder in the RTFO showed that the viscosity of the RAS binder was too high and that it did not move in the bottles during the procedure. Therefore, the RAS binders for this study were tested in their as-recovered state with no additional aging. Short term and long term aging of RAS binders in the RTFO or PAV is considered redundant due to the previous aging of in service PC shingles and the aging due to the air blown process of all shingles. These manufacturing and in-situ aging conditions are more severe than the RTFO and PAV.

The high temperature properties of the RAS binders were measured as described in AASHTO T315-08 using a TA Instruments AR 2000EX model Dynamic Shear Rheometer (DSR). This DSR model has the capability of testing at temperatures up to 150°C using a heated upper plate assembly. For temperatures higher than 150°C, an environmental temperature chamber assembly with a liquid nitrogen purge was used. The DSR procedure is performed using a 25-mm diameter parallel plate geometry with a 1000 micron sample gap. A sinusoidal shear load is applied to the test specimen at a frequency of 10 rad/sec and a target angular strain level of 10% to determine complex shear modulus (G^*) and phase angle (δ). For criteria purposes, the recovered RAS binder was tested as RTFO-aged material, even though no aging was performed.

The temperatures necessary to achieve a failing high temperature result for recovered RAS binder typically exceed the temperature range of many DSR models. This results in the need to extrapolate by 50 – 60°C or more to determine the continuous grading temperature. To address the issue of extrapolation over such a wide temperature range and its effect on continuous grade determination, two methods were used to calculate the high continuous grading temperature of the RAS binders. One set of samples from each individual extraction was tested at 82, 88, and 94°C, the highest standard test temperatures that a water controlled DSR should be able to maintain. The resulting values of $G^*/\sin(\delta)$ were plotted versus temperature on a log scale and extrapolated to a value of 2.20 kPa. The second set of samples was tested at elevated temperatures corresponding to passing and failing values of $G^*/\sin(\delta)$ and the continuous grading temperature calculated by interpolating between the two results using the equations provided in ASTM D 7643-10.

Low continuous grading temperatures were determined for the recovered RAS binder using two test procedures. The Bending Beam Rheometer (BBR) procedure as described in AASHTO T313-12 measures low temperature stiffness and relaxation properties using thin beams of asphalt binder to which a 980 mN creep load is applied for 240 seconds. The stiffness parameter, *S(t),* is calculated based on the measured deflection of the beam during the loading period. The relaxation parameter, *m(t),* is calculated as the slope of the *S(t)* curve at time *t*. Continuous grading temperatures for low temperature testing are defined as the temperatures at which *S(60 sec)* = 300 Mpa and *m(60 sec)* = 0.300, with the warmer of the two temperatures chosen as the continuous grade.

Testing recovered RAS binder in the BBR presented some challenges that made it difficult to obtain reliable results. The high viscosity of the recovered RAS binder in combination with the small dimensions of the BBR sample mold (6.35-mm x 12.70-mm x 127-mm) made preparing test specimens difficult, as the binder did not flow into the molds evenly. The RAS binder tended to cool and stiffen before reaching the bottom of the mold, leaving gaps in the test specimen. In addition to the difficulty with preparing the test specimens, the behavior of the recovered RAS binder at the test temperatures typically used in the BBR (below 0°C) prohibited testing at temperatures that bracket the low temperature pass / fail criteria. At low temperatures, recovered RAS binder has very poor relaxation properties and typically fails the *m(60 sec)* criteria at the warmest temperature that the BBR bath can reliably maintain (+6°C). The low temperature stiffness values require testing at much colder temperatures than what the *m(60 sec)* requires to achieve a failing test result based on the *S(60 sec)* criteria*.* The poor relaxation properties of the RAS binders make the test specimens brittle at colder temperatures and prone to breakage when the test load is applied. Since the m-value is the controlling criteria for RAS binders (its continuous grade temperature is higher than the continuous grade temperature for the stiffness criteria and is used to determine the low PG grade of the binder), testing was performed at the two warmest standard test temperatures possible: +6 and 0°C. The test results were then extrapolated to values of *S(60)* = 300 Mpa and *m(60)* = 0.300 to determine the low continuous grading temperatures based on the BBR criteria.

The low continuous grading temperatures of the recovered RAS binders were determined using a procedure developed by Western Research Institute (WRI). The 4-mm plate DSR procedure provides a method for estimating BBR *S(t)* and *m(t)* values that eliminates the need to mold test specimens and allows for testing at warmer temperatures than can be achieved in the BBR bath. The procedure uses 4-mm diameter DSR test specimen to determine values of storage modulus (*G'(t)*) over a range of frequencies from 0.1 – 100 rad/sec at 0.1% angular strain (chosen to be within the linear region for the binders being tested). The frequency sweeps were performed at two temperatures (4 and 14°C for this research) and the resulting isotherms were shifted using the Christensen-Anderson model to create a master curve at a chosen reference temperature. Once the master curve was created, *G'(t)* and the slope of the curve at 60 seconds, $m_{g'}$, were calculated by fitting a quadratic polynomial function to the master curve. Equations 1 and 2 (developed by WRI) were then used to convert the DSR data to BBR results at the reference temperature (25).

$$S(t) = 21.380 + 1.718*G'(t \qquad (1)$$

$$m = -0.115* + 0.708*m_{g'} \qquad (2)$$

The calculations were repeated at multiple reference temperatures and the resulting *S(60)* and *m(60)* values were used to determine the low continuous grading temperatures of the recovered RAS binders.

3.2.3 Testing Variability Analysis

For each RAS source, three types of variability were considered:

- Within sample – how repeatable are test results obtained from multiple test specimen taken from the same extraction / recovery procedure,
- Between sample – how reproducible are test results obtained from test specimen of the same RAS binder taken from different extraction / recovery procedures, and
- Test or calculation method – how do different methods of determining the same criteria affect the test results.

Table 4 shows a sample testing matrix for one RAS binder source.

Table 4 Sample Testing Matrix

	High Temperature		**Low Temperature**	
Extraction	*Measured*	*Extrapolated*	*BBR*	*4-mm*
1	Test 1	Test 1	Test 1	Test 1
	Test 2	Test 2	Test 2	Test 2
	Test 3	Test 3	Test 3	Test 3
2	Test 1	Test 1	Test 1	Test 1
	Test 2	Test 2	Test 2	Test 2
	Test 3	Test 3	Test 3	Test 3
3	Test 1	Test 1	Test 1	Test 1
	Test 2	Test 2	Test 2	Test 2
	Test 3	Test 3	Test 3	Test 3

Each cell in Table 4 represents three replicate test results to allow for determination of within sample variability. Statistical analysis was performed on the replicate results from each test procedure to determine whether or not the results obtained from a single sample of RAS binder were repeatable. If the results were not repeatable, this could imply that either the RAS binder itself was not a homogenous material or that it was sensitive to some portion of the handling or testing procedures.

Between sample variability for the high temperature tests was evaluated by repeating the test procedures on three separate samples of the same recovered RAS binder. This was done to evaluate if the extraction/recovery procedure could produce recovered RAS binder that had consistent, reproducible test results. Due to time and sample quantity limitations, between sample variability at cold temperatures was not evaluated.

Statistical comparisons were also performed to determine the effect of the different methods of determining high and low continuous grade temperatures. For the high temperature results, a comparison was made between the continuous grade temperatures determined by extrapolation and those obtained by testing at passing and failing temperatures. For the low temperature results, a comparison was made between the BBR continuous grading temperatures and those obtained from the 4-mm DSR procedure.

3.2.4 High Temperature Prediction

The temperatures necessary to achieve a failing high temperature result for RAS binders frequently exceed the temperature range of some DSR models, particularly those that use a water bath for temperature control. Because of this, it is often necessary to extrapolate by 50 – 60°C or more to determine the continuous grading temperature. Two methods were used to calculate the high continuous grading temperature of the RAS binders, and the results were compared to determine what effect, if any, the difference in test procedure had on the continuous grade temperature results.

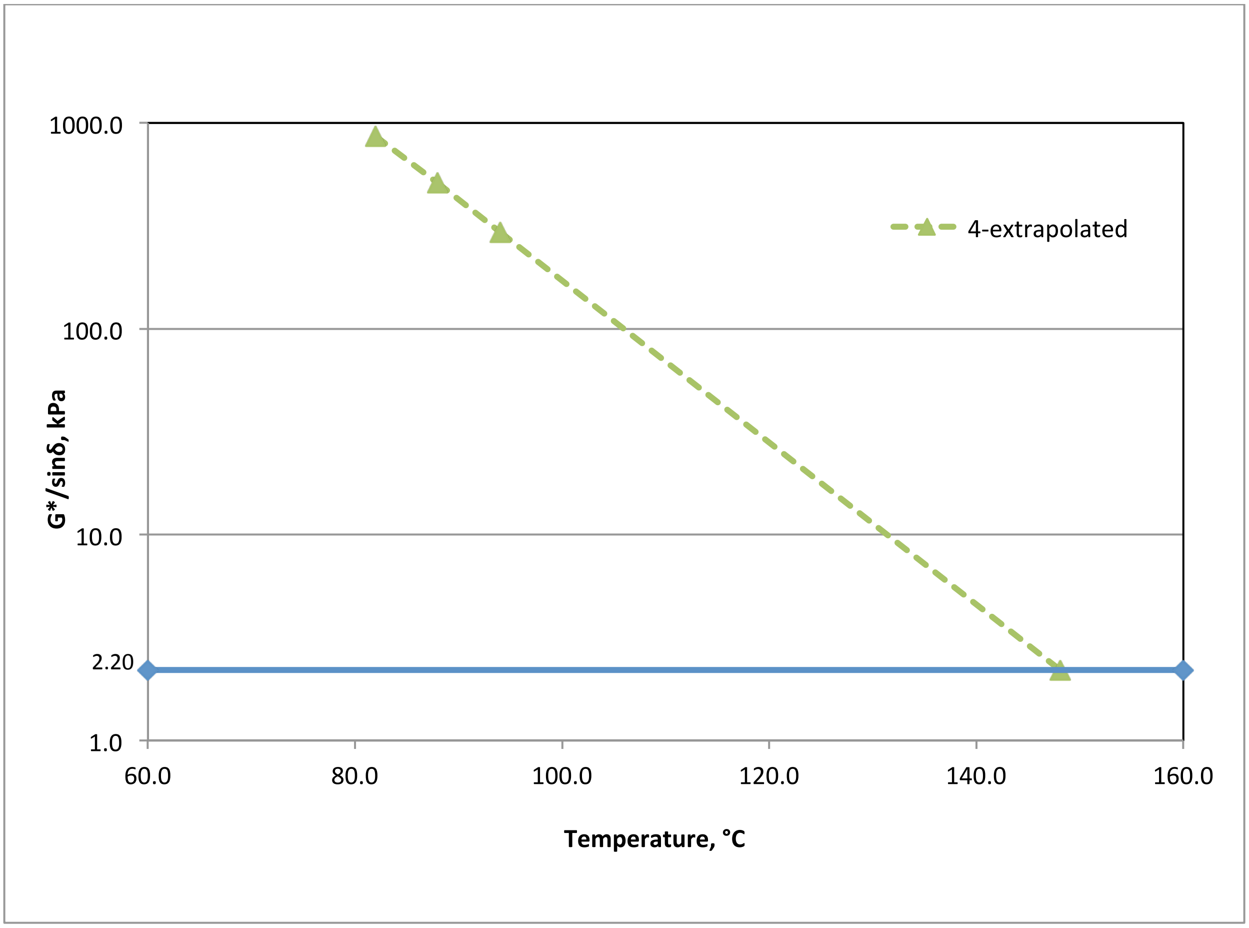


Figure 2 Extrapolated High Continuous Grade Extrapolation

The second set of samples was tested at elevated temperatures corresponding to passing and failing values of *G*/sin(δ)* and the continuous grading temperature calculated by interpolating between the two results.

4 TEST RESULTS

Seven RAS samples were delivered to NCAT for evaluation. This chapter describes the evaluation of those samples for both critical high and low temperatures.

4.1 RAS Binder Critical High Temperature Grade

4.1.1 Determining Critical High Temperature Grade and Variability

The critical high temperature grade of six RAS samples was determined using the previously described methodology. Three of the samples tested were PC RAS while two samples were from MW RAS stockpiles. The final sample was a blend of PC and MW RAS used in Oregon. Once the RAS binder had been extracted, the critical high temperature grade of each RAS sample was determined on three replicates from three separate extractions for each sample. This resulted in nine individual test points for each RAS sample. Table 5 provides the measured test results from each RAS sample. Individual test results are given in the Appendix A. It should be noted that these test results and conclusions are based on limited test results using very challenging testing procedures.

When comparing the variability of the high temperature test results, Table 5 shows that as expected, the within extraction variability was lower than the variability of the entire dataset. Eight of the 18 extractions had coefficients of variation (COV) less than 1%, while six had COVs less than 2%. When comparing the coefficients of variation for the entire data set of a singular RAS source, the COVs ranged from 3.2 to 7.7%.

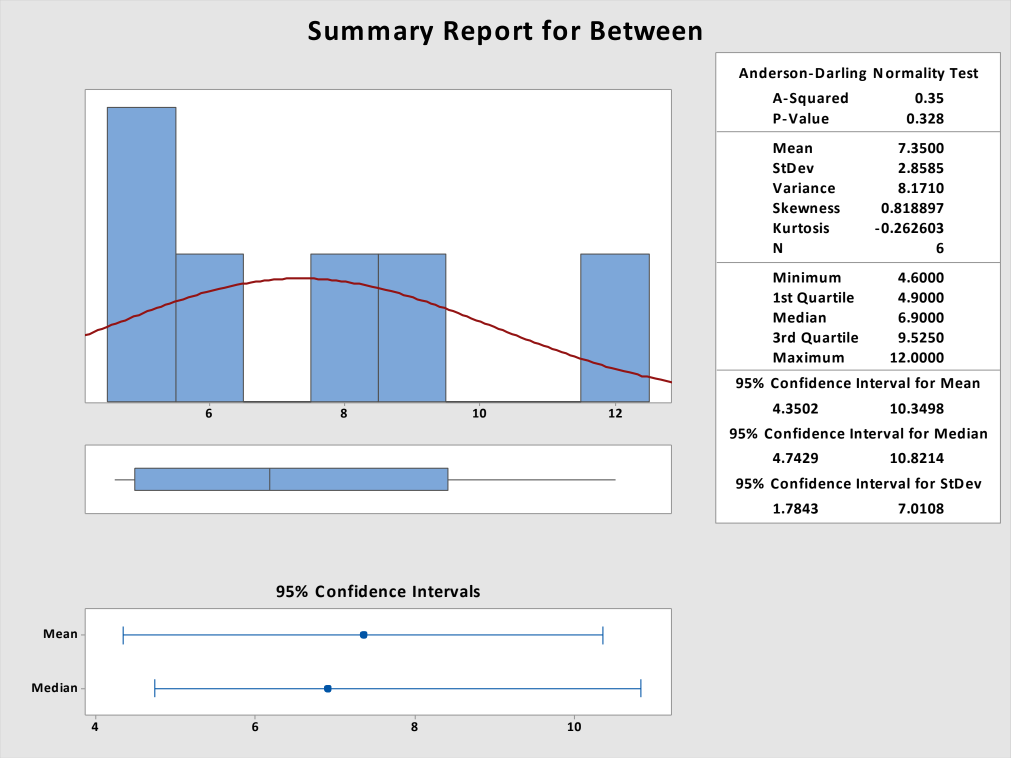
The standard deviations were similar. Ten of the extractions had standard deviations of triplicate measurements less than 2°C. Six extractions had standard deviations greater than 4°C. The single largest standard deviation for a single extraction was 11.7°C. However, when comparing the standard deviations of three replicates for three extractions, the data were more variable. Standard deviations ranged from 4.6 to 12°C.

The statistics show that MW RAS was commonly less variable than PC RAS; however, the one sample with the least variability between extractions was the MW/PC RAS blend from Oregon.

When trying to establish 95% confidence intervals for the standard deviations of the within extraction and between extraction datasets, Figures 3 and 4 show that while the between dataset is normal by the Anderson-Darling Normality Test (p = 0.328), the within extraction standard deviations are not normal. When transposed on a log scale (Figure 5), however, the data becomes normal, and one can ascertain a 95 percent confidence interval. Therefore, using these two 95 percent confidence intervals, one might expect the standard deviation for the testing of RAS binder between two different samples to be 4.4 and 10.3 °C. The expected range for testing the high temperature grade of RAS binder within single extractions would be 0.9 to 2.4°C.

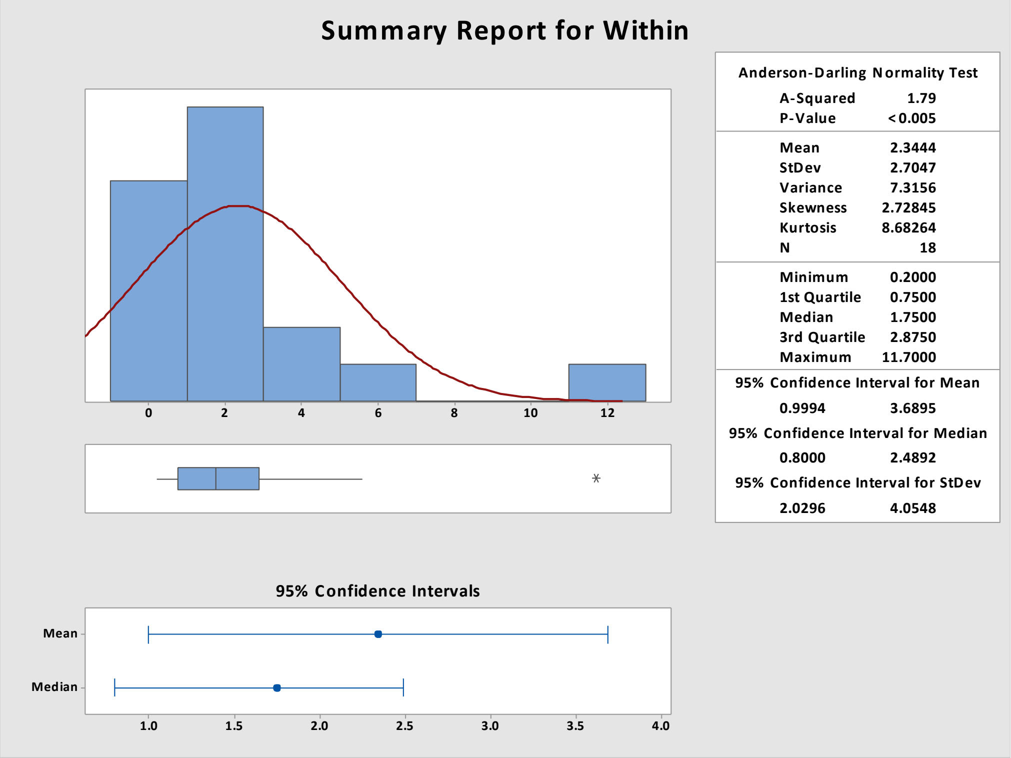
Table 5 High Temperature Test Results

State		*Michigan*	*Texas*	*Oregon*	*Michigan*	*New Hampshire*	*Georgia*
Source		*MW*	*MW*	*MW/PC Blend*	*PC*	*PC*	*PC*
Extract 1	*Average*	135.4	126.6	143.8	160	159.8	148.2
	Standard Deviation	2	0.2	1.2	1.7	3.7	2.8
	Coefficient of Variation	1.5	0.2	0.9	1	2.3	1.9
Extract 2	*Average*	133.1	137.4	146	161.7	163.8	151.1
	Standard Deviation	2.1	3.1	1	0.4	2.2	0.8
	Coefficient of Variation	1.6	2.3	0.7	0.2	1.3	0.6
Extract 3	*Average*	144.7	131.7	151.9	144.4	145.4	170.3
	Standard Deviation	0.5	1.8	5.6	0.8	0.6	11.7
	Coefficient of Variation	0.4	1.4	3.7	0.6	0.4	6.8
Set of 9 Tests	*Average*	137.7	131.9	147.2	155.4	156.3	156.5
	Standard Deviation	5.5	5	4.6	8.3	8.7	12
	Coefficient of Variation	4	3.8	3.2	5.4	5.6	7.7



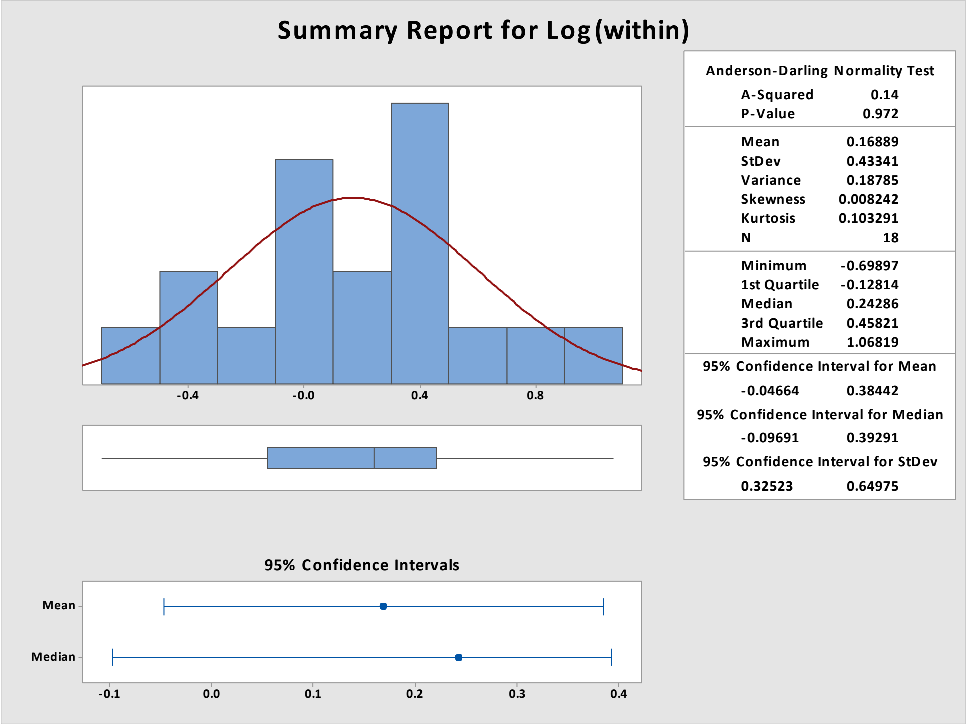
Anderson-Darling Normality Test	
A-Squared	0.35
P-Value	0.328
Mean	7.3500
StDev	2.8585
Variance	8.1710
Skewness	0.818897
Kurtosis	-0.262603
N	6
Minimum	4.6000
1st Quartile	4.9000
Median	6.9000
3rd Quartile	9.5250
Maximum	12.0000
95% Confidence Interval for Mean	
4.3502	10.3498
95% Confidence Interval for Median	
4.7429	10.8214
95% Confidence Interval for StDev	
1.7843	7.0108

Figure 3 Between Extraction Standard Deviation Normality Test Results



Anderson-Darling Normality Test	
A-Squared	1.79
P-Value	< 0.005
Mean	2.3444
StDev	2.7047
Variance	7.3156
Skewness	2.72845
Kurtosis	8.68264
N	18
Minimum	0.2000
1st Quartile	0.7500
Median	1.7500
3rd Quartile	2.8750
Maximum	11.7000
95% Confidence Interval for Mean	
0.9994	3.6895
95% Confidence Interval for Median	
0.8000	2.4892
95% Confidence Interval for StDev	
2.0296	4.0548

Figure 4 Within Extraction Standard Deviation Normality Results



Anderson-Darling Normality Test	
A-Squared	0.14
P-Value	0.972
Mean	0.16889
StDev	0.43341
Variance	0.18785
Skewness	0.008242
Kurtosis	0.103291
N	18
Minimum	-0.69897
1st Quartile	-0.12814
Median	0.24286
3rd Quartile	0.45821
Maximum	1.06819
95% Confidence Interval for Mean	
-0.04664	0.38442
95% Confidence Interval for Median	
-0.09691	0.39291
95% Confidence Interval for StDev	
0.32523	0.64975

Figure 5 Log(within) Standard Deviation Normality Tests

4.1.2 Extrapolating Critical High Temperature Grade

As shown in the previous tables, all six RAS sources had critical high temperature grades greater than 100°C. DSRs using water-controlled temperature baths cannot achieve reliable temperatures for testing greater than approximately 94°C before problems such as water boiling begin to occur. Therefore, in order for agencies and contractors using water-controlled temperature baths to determine critical high temperature grades of RAS binder, an extrapolation methodology needs to be determined. The simplest method of extrapolating these data would be to use a linear model to plot G*/sinδ at 82, 88, and 94°C, and then calculate the temperature where the data meet the critical high temperature criterion. This methodology is described previously in more detail. Table 6 provides the extrapolated high temperature results for all three replicate tests of each extraction. Appendix B shows the individual test results.

Table 6 High Temperature Extrapolations

State		*Michigan*	*Texas*	*Oregon*	*Michigan*	*New Hampshire*	*Georgia*
Source		*MW*	*MW*	*MW/PC Blend*	*PC*	*PC*	*PC*
Extract 1	*Average*	136.3	137.2	145.5	150.9	141.3	137.2
	Standard Deviation	6.2	0.9	6.5	23.5	25.4	0.9
	Coefficient of Variation	4.55	0.6	4.5	15.6	17.9	0.6
Extract 2	*Average*	131.1	137.5	139.3	192.0	146.1	137.5
	Standard Deviation	2.8	1.8	0.6	43.1	1.7	1.8
	Coefficient of Variation	2.15	1.3	0.5	22.4	1.2	1.3
Extract 3	*Average*	131.1	198.5	148.1	160.5	147.2	198.5
	Standard Deviation	2.2	53.1	1.1	23.8	4.0	53.1
	Coefficient of Variation	1.50	26.8	0.7	14.9	2.7	26.8
Set of 9 Tests	*Average*	132.9	157.7	144.3	167.8	144.9	157.7
	Standard Deviation	4.4	40.5	5.1	33	13.1	40.5
	Coefficient of Variation	3.29	25.68	3.56	19.68	9.08	25.68

It is noteworthy that when considering within extraction COV, five of the RAS samples had COVs for two extractions at less than 3 percent, while the third RAS extraction had a significantly larger COV. In most cases where the COV was significantly larger, only one test result caused the skew in the data. The only RAS sample that had within extraction COVs consistently greater than 14 percent was the Michigan PC RAS where there were consistent differences between all three extrapolations.

While one should consider variability in the extrapolations, one ultimately needs to know if the extrapolations are accurate compared to given measured data. To statistically assess these data, paired *t*-tests ($\alpha = 0.05$) were used validate the null hypothesis of no difference between the measured and extrapolated critical high temperatures. This test was conducted on the nine test results for each RAS sample as well as the entire population of tests. *P*-vales are presented in Table 7 and show no statistical differences between the measured and extrapolated data for any of the six samples as well as the entire population. These preliminary results were based on only six different RAS samples. Additional testing is taking place as part of National Cooperative Highway Research Project 9-55 to determine if these trends continue before more definitive conclusions are drawn.

Table 7 Statistical Analysis of Extrapolated to Measured Critical High Temperatures

Sample	Michigan MW	Michigan PC	Oregon Blend	New Hampshire PC	Georgia PC	Texas MW	Population
***p*-value**	0.078	0.194	0.178	0.077	0.906	0.095	0.766

Despite the lack of evidence showing statistical differences, scatterplots comparing the measured to extrapolated high temperatures show disagreement between the measured and extrapolated data (Figure 6). Further evidence is given by the histogram of the differences between the measured and extrapolated data (Figure 7). Though many of the results are within a few degrees of each other, numerous results still show errors in the extrapolation of more than 15°C on a point to point basis showing that single replicate extrapolation can lead to error in critical high temperature expectations.

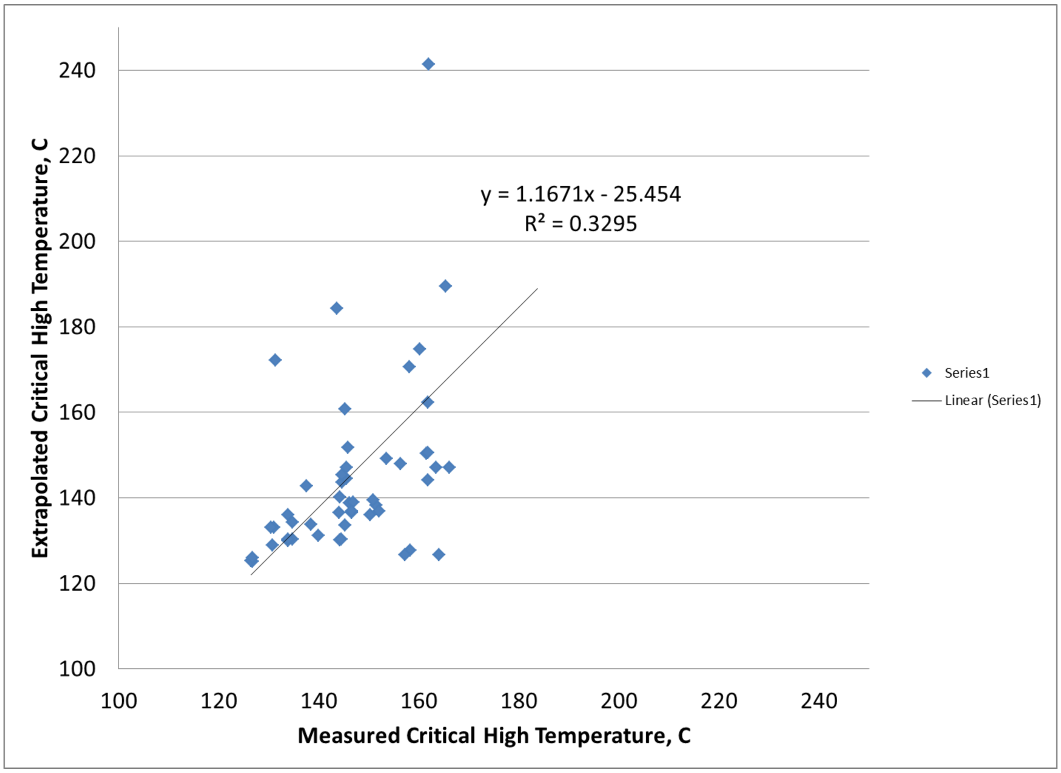


Figure 6 Differences between Measured and Extrapolated Data

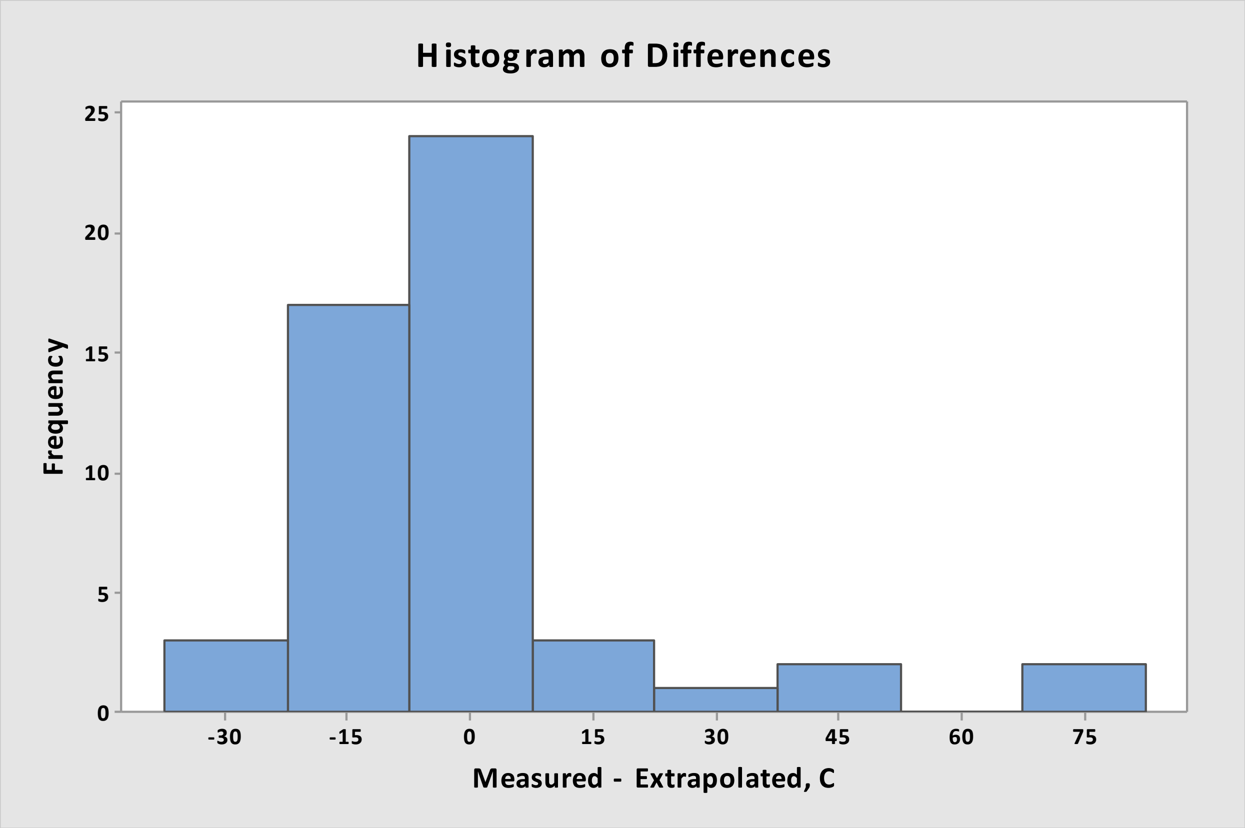


Figure 7 Histogram of Measured Minus Extrapolated Critical High Temperature

Better agreement is found when comparing the averages of triplicate testing on a single extraction. The scatterplot (Figure 8) shows that much of the variability seen from individual testing is removed, and the majority of the test results are more comparable between the data sets. However, when assessing the histogram of the differences (Figure 9), six of the eighteen data comparisons have differences greater than 10 °C, showing that further testing may be required to remove variability from the analysis. Some of this testing is currently underway. The statistical analyses may be slightly skewed by the limited test results.

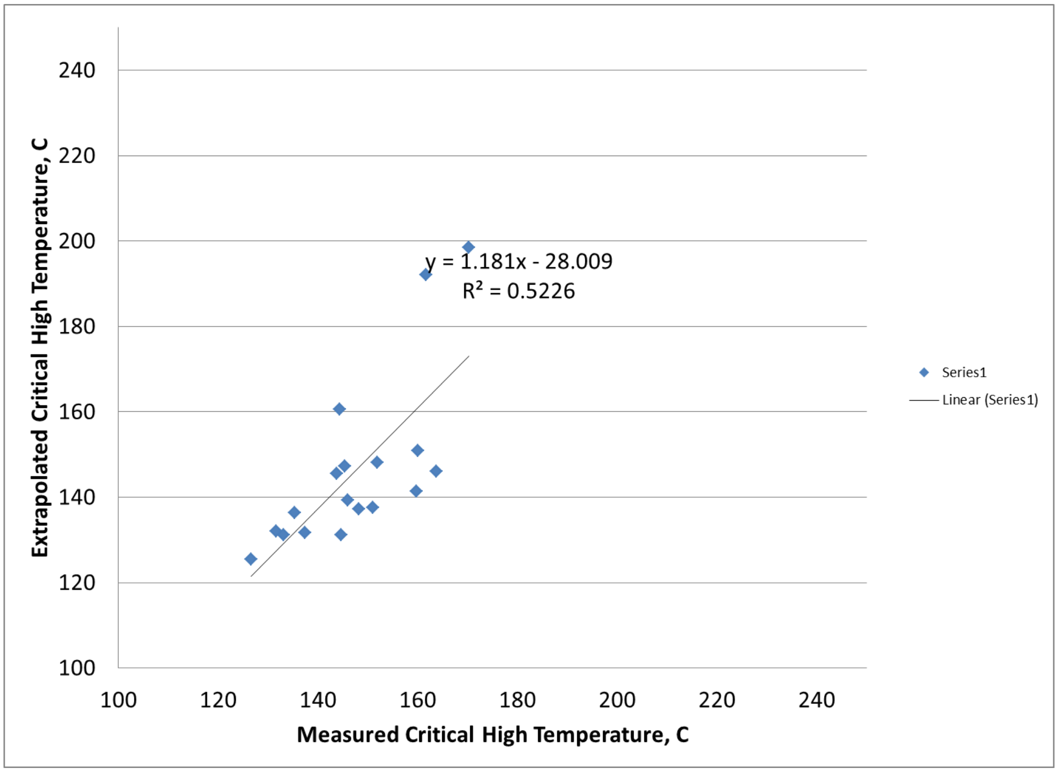


Figure 8 Comparison of Measured and Extrapolated Data of Triplicate Testing

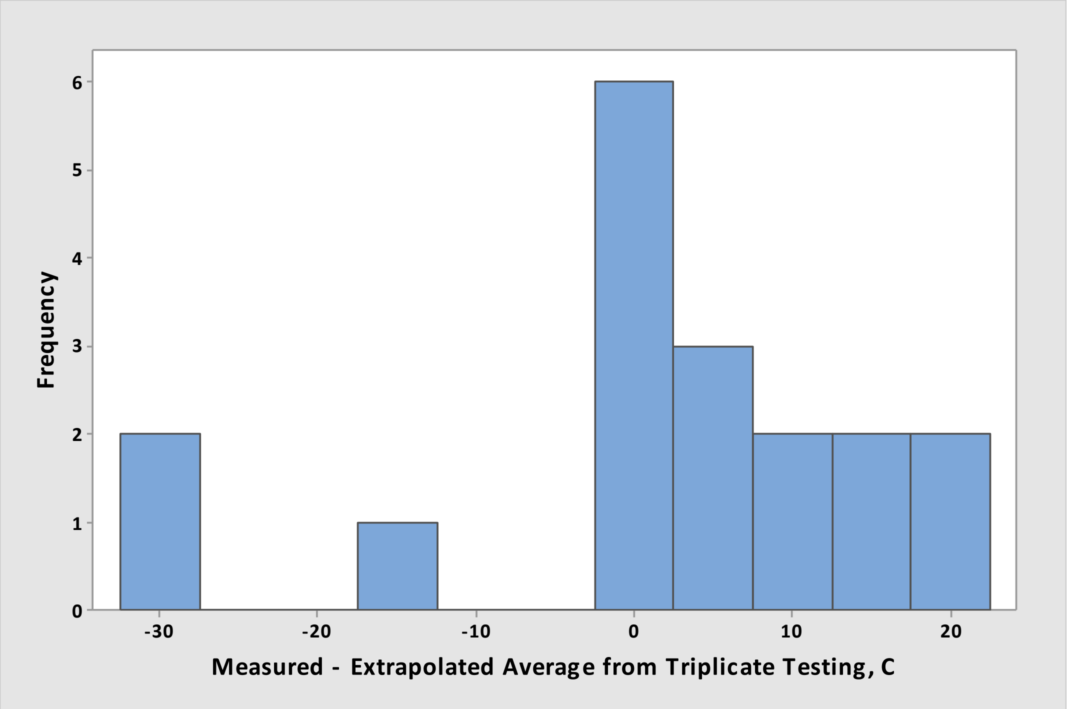


Figure 9 Histogram of Differences between Measured and Extrapolated Critical High Temperature Average of Triplicate Testing from Single Extraction

Similar results are seen when comparing the averages of triplicate data from three separate extractions. Approximately 1/3 of the variances have differences greater than 10°C. Therefore, using the results of this study, one should extrapolate data in order to ascertain a critical high temperature grade for RAS binder, running triplicate tests from a single extraction and removing obvious outliers would be the most appropriate method. The slight errors from the extrapolated results would have little effect on choosing a virgin binder. The RAS binders have a critical high temp around 120 C to 180°C. This is much hotter than a pavement will see in its life. The critical temperature is important because it will affect the final blended binder critical high and low temps.

4.2 RAS Binder Critical Low Temperature Grade

The two low temperature testing methods described in the methodology were used to determine critical low temperatures for four RAS sources (Texas MW, Wisconsin MW, Oregon Blend, and New Hampshire PC). The purpose of this portion of the testing was to determine if the DSR method could give critical low temperatures comparable to those obtained from the BBR method. A secondary reason for the testing was to determine if one method could provide better repeatability than the other.

Only one extraction procedure was performed for each RAS source due to material and time limitations. Using the recovered binder, three replicate tests were performed for both the BBR test and the 4-mm diameter DSR test. The results from each of these tests were used to calculate critical low temperatures based on the *S(60)* and *m(60)* criteria. This resulted in a dataset of twelve critical low temperatures for each RAS source (three per test/criteria combination).

It should be noted that these test results and conclusions are based on limited test results using very challenging testing procedures.

4.2.1 Determining Critical Low Temperature Grade Using Bending Beam Rheometer

Tables 8 and 9 show the BBR *S(60)* and *m(60)* critical low temperatures for each RAS source. As can be seen from the tables, the results based on the BBR test follow typical critical low temperature trends for the MW RAS sources with the values calculated using the BBR *m(60)* criteria being warmer than the values calculated using the BBR *S(60)* criteria. The Oregon Blended RAS also followed this trend for two of the three replicates, while the PC RAS only followed it for one of the three replicates.

Table 8 Critical Low Temperatures Based on BBR *S(60)* Criteria

Replicate	TX MW	WI MW	OR Blend	NH PC
1	-67.7	-31.5	-36.7	-42.6
2	-39.4	-26.0	-33.0	-72.2
3	-35.1	-32.8	-25.7	-26.0
Average	-47.4	-30.1	-31.8	-46.9
Standard Deviation	17.7	3.6	5.6	23.4
COV, %	37.3	12.1	17.6	49.9

Table 9 Critical Low Temperatures Based on BBR *m(60)* Criteria

Replicate	TX MW	WI MW	OR Blend	NH PC
1	-3.3	2.5	-28.9	-118.0
2	-9.3	-3.5	-44.5	-64.0
3	-9.1	-2.3	21.3	14.3
Average	-7.2	-1.1	-17.3	-55.9
Standard Deviation	3.4	3.2	34.4	66.5
COV, %	46.7	293.4	198.3	119.0

Both sets of BBR critical low temperatures had high variability with COV values ranging from 12.1 - 293%. Although the COV values for the BBR *S(60)* results appeared to be lower than the COV values for the BBR *m(60)* results, a paired *t*-test analysis (α=0.05) showed that there was no statistical difference between the variability for the two criteria (*p*=0.087). In this analysis the *m*(60) and *S*(60) criteria were paired from a singular RAS source.

Standard deviations ranged from 3.6 - 23.4°C for the BBR *S(60)* critical low temperatures and 3.2 - 66.5°C for the BBR *m(60)* critical low temperatures. A paired *t*-test analysis (α=0.05) showed that there was no statistical difference between the standard deviations for the two sets of data (*p*=0.401). This analysis was completed similarly to the previously mentioned COV analyses.

4.2.1 Determining Critical Low Temperature Grade Using Dynamic Shear Rheometer

Tables 10 and 11 show the *S(60)* and *m(60)* critical low temperatures calculated using the test results from the 4-mm diameter plate DSR procedure. The DSR critical low temperatures also followed typical trends with the DSR *m(60)* criteria having warmer critical temperatures than those calculated using the DSR *S(60)* criteria for all four RAS sources. COV values for the DSR critical low temperatures ranged from 40 - 163.7% for the DSR *S(60)* results and from 32.7 - 147.7% for the DSR *m(60)* results. Standard deviations for both criteria were high, ranging from 25.1°C - 70.4°C for the DSR *S(60)* critical low temperatures and from 10.5°C - 35.8°C for the DSR *m(60)* critical low temperatures. Paired *t*-test analysis (α=0.05) showed no statistical difference in either COV or standard deviation between the two criteria (COV *p*-value = 0.927, Standard deviation *p*-value = 0.133).

Table 10 Critical Low Temperatures Based on DSR *S(60)* Criteria

Replicate	TX MW	WI MW	OR Blend	NH PC
1	-58.9	-12.0	-61.5	-74.7
2	-87.4	-11.6	-102.3	-26.8
3	-132.7	-55.3	34.8	-18.8
Average	-93.0	-26.3	-43.0	-40.1
Standard Deviation	37.2	25.1	70.4	30.2
COV, %	40.0	95.5	163.7	75.4

Table 11 Critical Low Temperatures Based on DSR *m(60)* Criteria

Replicate	TX MW	WI MW	OR Blend	NH PC
1	-25.8	-14.0	-32.9	-50.0
2	-31.0	5.0	-68.6	-7.9
3	-47.6	-12.4	2.9	-12.3
Average	-34.8	-7.1	-32.9	-23.4
Standard Deviation	11.4	10.5	35.8	23.1
COV, %	32.7	147.7	108.8	98.9

4.2.3 Comparison of BBR and DSR Critical Low Temperature Results

To statistically compare the critical low temperatures results obtained from the BBR to those obtained using the DSR, paired *t*-tests ($\alpha = 0.05$) were used with the null hypothesis of no difference between the critical low temperature values. Separate analyses were done comparing the *S(60)* and *m(60)* critical low temperatures for each RAS sample. A comparison for each criterion was also completed using the entire population of tests. *P*-vales are presented in Tables 12 and 13.

Table 12 Statistical Analysis of BBR *S(60)* to DSR *S(60)* Critical Low Temperatures

Sample	Texas MW	Wisconsin MW	Oregon Blend	New Hampshire PC	Population
***p*-value**	0.128	0.820	0.809	0.774	0.425

Table 13 Statistical Analysis of BBR *m(60)* to DSR *m(60)* Critical Low Temperatures

Sample	Texas MW	Wisconsin MW	Oregon Blend	New Hampshire PC	Population
***p*-value**	0.016	0.396	0.617	0.469	0.752

The results show no statistical differences could be found between the measured and extrapolated results for almost all the RAS samples as well as the entire population. The only exception was the *m(60)* critical low temperatures for the Texas MW RAS source.

Despite the lack of evidence showing statistical differences, scatterplots comparing the BBR to DSR critical low temperatures show a lack of correlation between the two methods for either criteria (Figures 10-13). Further evidence of this lack of agreement is given by the histogram of the differences between the results. Though some results are within a few degrees of each other, most results show differences between the test methods of more than 25°C on a point to point basis which would make these differences be considered practical in nature. This indicates that there is not good agreement between the DSR and BBR methods for these RAS samples. Due to the complex nature RAS binders, it is difficult to determine which test result provides the most accurate test result.

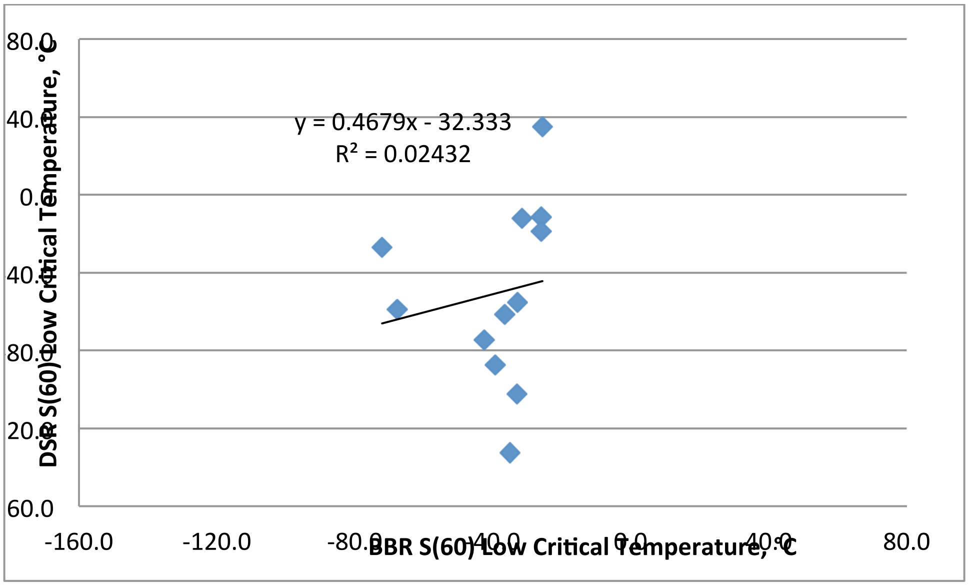


Figure 10 Comparison of BBR *S(60)* and DSR *S(60)*

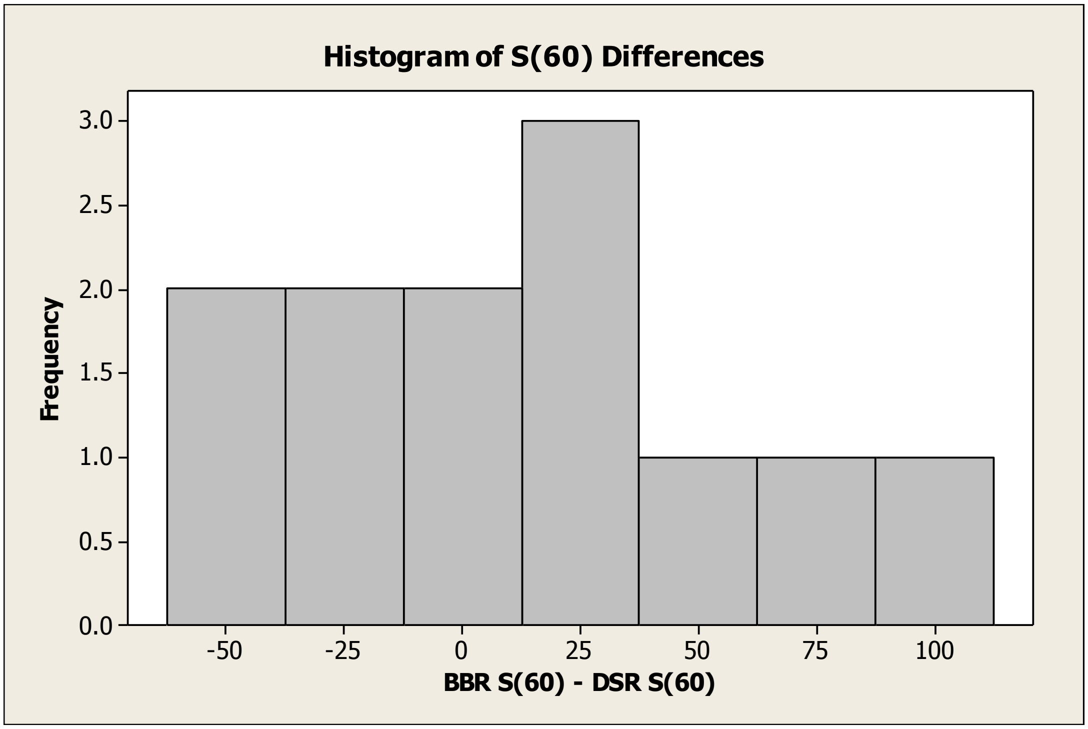


Figure 11 Histogram of *S(60)* Differences

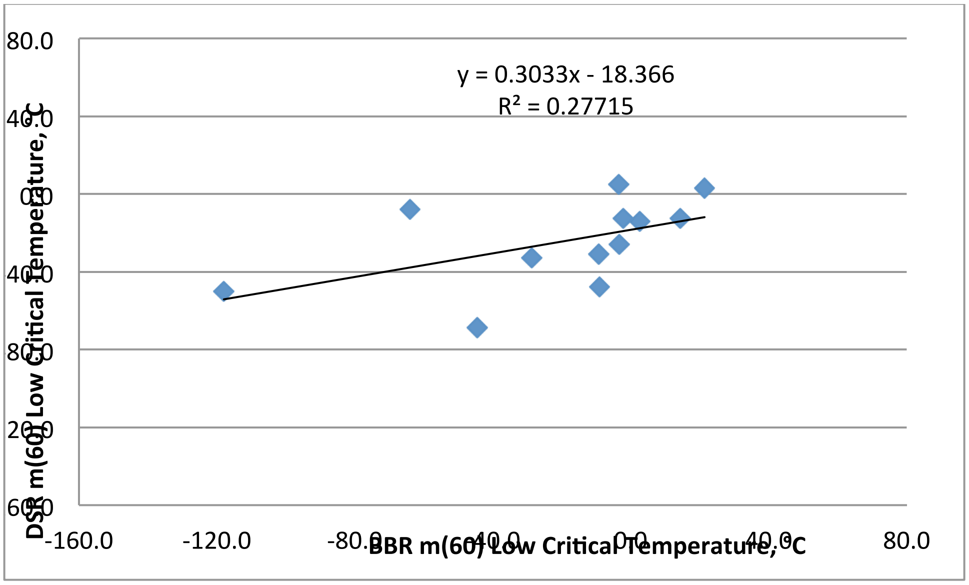


Figure 12 Comparison of BBR *m(60)* and DSR *m(60)*

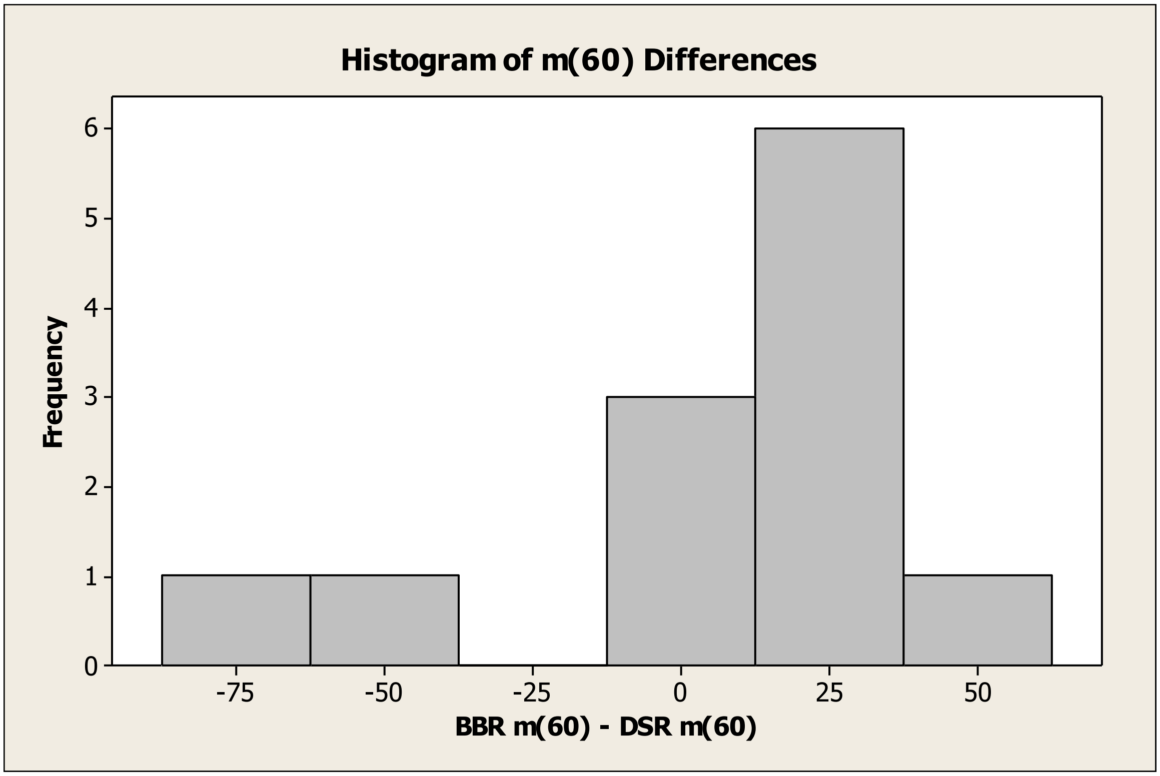


Figure 13 Comparison of BBR *m(60)* and DSR *m(60)*

Paired *t*-test analyses (α=0.05) were used to evaluate the COV values and the standard deviation between the two test methods. The DSR and BBR test methods were found to have statistically the same COV values (p = 0.971) and standard deviations (p=0.315).

Based on these results, it would appear that the 4-mm DSR test, while simpler to run may not provide critical low temperatures that are equivalent to those obtained from the BBR procedure for this set of RAS samples. The DSR procedure also does not appear to provide more repeatable results than the BBR procedure.

5 CONCLUSIONS AND RECOMMENDATIONS

Based on the limited study which evaluated various testing and extrapolation methods for determining RAS binder properties, the following conclusions were drawn:

- Within sample critical high temperature grades were less variable than between sample critical high temperature grades for a given RAS source. One would expect the results of three critical high temperatures to vary between 0.9 and 2.4°C for a given RAS source. The standard deviations for between sample tests ranged from 4.6 to 12°C.
- Linearly extrapolated critical binder high temperature grades were statistically the same as tested results; however, scatterplots showed practical differences were evident within the dataset. Using averages of triplicate test results reduced variability and disagreement.
- Both DSR and BBR low temperature testing provided variable and inconsistent critical low temperatures for RAS binder.

Based on these conclusions, the following recommendations can be made:

- One can extrapolate critical high temperatures of RAS binder; however, care should be taken to reduce variability. Remove outliers and take at least an average of triplicate results to ensure more precise and accurate results.
- Both the BBR and DSR are variable and provide results which may not be accurate for ascertaining the critical low temperature grade of RAS binder. Additional work needs to be completed on this subject. A new testing method may be needed for determining the low temperature properties of RAS binder.
- Other methods should be considered for extrapolation of critical high and low temperature grades for RAS binders. Currently, the Asphalt Institute is working on developing and quantifying the variability of other extrapolation/blending methods using the same materials used in this study.

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APPENDIX A HIGH TEMPERATURE TEST RESULTS

Table A-1 Michigan MW RAS Measured Critical High Temperature Grade

Replicate	**Measured Critical High Temperature, °C**		
	Extraction 1	Extraction 2	Extraction 3
1	137.6	130.7	144.3
2	133.8	133.8	144.5
3	134.7	134.7	145.3
Average	135.4	133.1	144.7
Standard Deviation	2.0	2.1	0.5
COV, %	1.5	1.6	0.4
Average of Nine Tests			137.7
Standard Deviation of Nine Tests			5.5
COV of Nine Tests, %			4.0

Table A-2 Michigan PC RAS Measured Critical High Temperature Grade

Replicate	**Measured Critical High Temperature, °C**		
	Extraction 1	Extraction 2	Extraction 3
1	161.6	161.8	144.1
2	158.3	162.0	145.3
3	160.2	161.3	143.7
Average	160.0	161.7	144.4
Standard Deviation	1.7	0.4	0.8
COV, %	1.0	0.2	0.6
Average of Nine Tests			155.4
Standard Deviation of Nine Tests			8.3
COV of Nine Tests, %			5.4

Table A-3 Oregon RAS Blend Measured Critical High Temperature Grade

Replicate	**Measured Critical High Temperature, °C**		
	Extraction 1	Extraction 2	Extraction 3
1	142.4	144.9	145.6
2	144.3	146.2	156.4
3	144.7	146.9	153.6
Average	143.8	146.0	151.9
Standard Deviation	1.2	1.0	5.6
COV, %	0.9	0.7	3.7
Average of Nine Tests			147.2
Standard Deviation of Nine Tests			4.6
COV of Nine Tests, %			3.2

Table A-4 New Hampshire PC RAS Measured Critical High Temperature Grade

Replicate	**Measured Critical High Temperature, °C**		
	Extraction 1	Extraction 2	Extraction 3
1	164.1	166.1	145.6
2	158.1	161.9	144.7
3	157.3	163.4	145.8
Average	159.8	163.8	145.4
Standard Deviation	3.7	2.2	0.6
COV, %	2.3	1.3	0.4
Average of Nine Tests			156.3
Standard Deviation of Nine Tests			8.7
COV of Nine Tests, %			5.6

Table A-5 Georgia PC RAS Measured Critical High Temperature Grade

Replicate	**Measured Critical High Temperature, °C**		
	Extraction 1	Extraction 2	Extraction 3
1	151.5	152.0	161.9
2	146.6	150.3	165.4
3	146.6	150.9	183.7
Average	148.2	151.1	170.3
Standard Deviation	2.8	0.8	11.7
COV, %	1.9	0.6	6.8
Average of Nine Tests			156.5
Standard Deviation of Nine Tests			12.0
COV of Nine Tests, %			7.7

Table A-6 Texas MW RAS Measured Critical High Temperature Grade

Replicate	**Measured Critical High Temperature, °C**		
	Extraction 1	Extraction 2	Extraction 3
1	126.7	133.9	133.8
2	126.4	138.4	131.0
3	126.8	139.9	130.4
Average	126.6	137.4	131.7
Standard Deviation	0.2	3.1	1.8
COV, %	0.2	2.3	1.4
Average of Nine Tests			131.9
Standard Deviation of Nine Tests			5.0
COV of Nine Tests, %			3.8

APPENDIX B HIGH TEMPERATURE EXTRAPOLATIONS

Table B1 Michigan MW RAS Extrapolated Critical High Temperature Grade

Replicate	**Measured Critical High Temperature, °C**		
	Extraction 1	Extraction 2	Extraction 3
1	142.7	128.9	130.1
2	136.0	130.2	130.3
3	130.3	134.3	131.3
Average	136.3	131.1	131.1
Standard Deviation	6.2	2.8	2.2
COV, %	4.55	2.15	1.50
Average of Nine Tests			132.9
Standard Deviation of Nine Tests			4.4
COV of Nine Tests, %			3.29

Table B2 Michigan PC RAS Extrapolated Critical High Temperature Grade

Replicate	**Measured Critical High Temperature, °C**		
	Extraction 1	Extraction 2	Extraction 3
1	150.3	162.4	136.6
2	127.7	241.4	160.8
3	174.8	172.1	184.3
Average	150.9	192.0	160.5
Standard Deviation	23.5	43.1	23.8
COV, %	15.6	22.4	14.9
Average of Nine Tests			167.8
Standard Deviation of Nine Tests			33.0
COV of Nine Tests, %			19.68

Table B3 Oregon Blended RAS Extrapolated Critical High Temperature Grade

Replicate	**Measured Critical High Temperature, °C**		
	Extraction 1	Extraction 2	Extraction 3
1	152.8	140.0	147.1
2	140.2	138.8	147.9
3	143.6	139.0	149.2
Average	145.5	139.3	148.1
Standard Deviation	6.5	0.6	1.1
COV, %	4.5	0.5	0.7
Average of Nine Tests			144.3
Standard Deviation of Nine Tests			5.1
COV of Nine Tests, %			3.56

Table B4 New Hampshire PC RAS Extrapolated Critical High Temperature Grade

Replicate	**Measured Critical High Temperature, °C**		
	Extraction 1	Extraction 2	Extraction 3
1	126.6	147.0	144.5
2	170.6	144.1	145.4
3	126.7	147.1	151.8
Average	141.3	146.1	147.2
Standard Deviation	25.4	1.7	4.0
COV, %	17.9	1.2	2.7
Average of Nine Tests			144.9
Standard Deviation of Nine Tests			13.1
COV of Nine Tests, %			9.08

Table B5 Georgia PC RAS Extrapolated Critical High Temperature Grade

Replicate	**Measured Critical High Temperature, °C**		
	Extraction 1	Extraction 2	Extraction 3
1	138.3	136.9	150.5
2	136.6	136.0	189.4
3	136.8	139.5	255.6
Average	137.2	137.5	198.5
Standard Deviation	0.9	1.8	53.1
COV, %	0.6	1.3	26.8
Average of Nine Tests			157.7
Standard Deviation of Nine Tests			40.5
COV of Nine Tests, %			25.68

Table B6 Texas MW RAS Extrapolated Critical High Temperature Grade

Replicate	**Measured Critical High Temperature, °C**		
	Extraction 1	Extraction 2	Extraction 3
1	138.3	136.9	150.5
2	136.6	136.0	189.4
3	136.8	139.5	255.6
Average	137.2	137.5	198.5
Standard Deviation	0.9	1.8	53.1
COV, %	0.6	1.3	26.8
Average of Nine Tests			157.7
Standard Deviation of Nine Tests			40.5
COV of Nine Tests, %			25.68