Asphalt Binder Extraction Protocol for Determining Amount & PG Characteristics of Binders Recovered from Asphalt Mixtures

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16. Abstract

This research project evaluated test procedures to quantify the amount of binder in asphalt mixtures. Although there are different well established AASHTO procedures to determine the asphalt content of an asphalt mix, the accuracy of these test procedures may be sensitive to local materials. In addition, as the use of high recycled binder content mixes increases, there is a need to evaluate the within-lab and between-lab variability. A second goal for this project was to evaluate the variability of the PG properties of the extracted binder after recovery.

The experimental plan included the evaluation of AASHTO T164 Methods A (centrifuge extraction) and method B (reflux extraction), asphalt analyzer and AASHTO T 308-ignition test. To accomplish this, several laboratories from Wisconsin along with the NCAT lab participated in the study. A total of eight mixes that included virgin mixes and mixes with various contents of recycled binder were evaluated to quantify the variability in the determination of their asphalt content. Two sources of RAP and one RAS were also evaluated. Within-lab and between-lab variability for each test procedure were developed for Wisconsin materials and compared to the current AASHTO standards. Recommendations were provided to revise and adjust current WisDOT JMF tolerance for asphalt content specification when these test procedures are used. In addition, PG characteristics of the recovered asphalt binders were also assessed.

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Executive Summary

This research evaluated several test procedure to quantify the amount of asphalt in asphalt mixtures. Although there are various well-established American Association of State Highway and Transportation Officials (AASHTO) procedures to determine the asphalt content of a mix, the accuracy of these procedures may be sensitive to local materials. In addition, as the use of high recycled binder content increases, there is a need to evaluate the variability of test results within-lab but also between different labs. To accomplish this, several laboratories from Wisconsin along with the National Center of Asphalt Technology (NCAT) lab participated in the study. A second goal for this project was to evaluate the variability of the Performance Grading (PG) properties of the extracted binder after recoveries.

The experimental plan included the evaluation of AASHTO T164 methods A (centrifuge extraction), and method B (reflux extraction), asphalt analyzer as well as AASHTO T 308. A total of eight mixes, including virgin mixes and mixes with various contents of recycled binder, were evaluated to quantify the variability in the determination of their asphalt content. In addition, two sources of recycled asphalt pavements (RAP) and one source of recycled asphalt shingles (RAS) were also evaluated. All of the materials evaluated represent those commonly used in Wisconsin.

Within-lab and between-lab variability for each test procedure were developed for Wisconsin materials and compared to the current AASHTO standards. Recommendations were provided to revise and adjust current Wisconsin Department of Transportation (WisDOT) job mix formula (JMF) tolerance for asphalt content specification when these test procedures are used. In addition, PG characteristics of the recovered asphalt binders were also assessed.

Summary of Findings

Centrifuge Extraction

- Within-lab and between-lab variability for asphalt content does not seem to be affected by the use of recycled mixes.
- The average difference between the actual and measured asphalt content for all mixes was found to be equal to 0.21%. This indicates that the solvent extraction may not able to remove all the binder from the mix.
- Standard deviations and acceptable range of two test results found for this test method are within the current precision statement of AASHTO T 164.
- AASHTO T 164 includes one precision statement regardless of the extraction method that is used (e.g centrifuge, reflux). Within-lab and between-lab variability for this test method was found to be lower than the precision statistic included in AASHTO T 164.

Asphalt Analyzer

- The asphalt analyzer had the lowest standard deviations of the test procedures evaluated for tests conducted within a lab and between labs.
- Similar to the results from centrifuge test, within-lab and between-lab variability for asphalt content does not seem to be affected by the use of recycled mixes.
- The average difference between the actual and measured asphalt content was 0.17%. Once again, solvent may not be able to remove all the binder from the mix.
- Although there is currently no precision statement for this test method, the within-lab and between lab variability developed in this study was found to be within the current precision statement of AASHTO T 164 and similar to the variability for centrifuge testing.

Reflux

- Within-lab and between-lab variability for this test procedure were developed with test results from two labs only. The results show higher variability than the centrifuge and asphalt analyzer but were still within AASHTO T 164 precision statement. The average difference between the actual and measured asphalt content was 0.17%.

Ignition

- The aggregates used in this study had a relatively high mass loss even at the reduced temperature of 800°F. Higher mass loss translates into higher correction factors (CFs). Conducting the tests at higher temperatures would yield even higher CFs and more variability in the test results.
- Using the corrected asphalt content test results, standard deviations and acceptable range of two test results found for this test were slightly higher than the ones currently specified in the test procedure. This may be attributed to the aggregates used in this study, which generally showed high mass loss even at the reduced temperature of 800°F.
- For a few of the recycled mixes, within-lab and between-lab variability for individual test results by mix type were higher than those for virgin and low recycled mixes.
- The average difference between the actual and measured asphalt content for all mixes was found to be equal to 0.05% (with CFs). This indicates that if correction factors are properly applied, ignition tests can yield measured asphalt contents closer to the actual asphalt contents.
- For ignition tests conducted on RAS material only, it was observed that at 800°F, there was some unburned asphalt left in the residue. Testing conducted at 900°F didn't show any signs of unburned asphalt. Hence, it is recommended that the test temperature for RAS only be set at 900°F.

PG Characteristics of Recovered Asphalt Binders

- Continuous grading properties for the recovered binders for virgin and recycled mixes were found to be similar for the different labs with the exception of one lab, which indicated lower values for high continuous grades for all mixes.
- For the materials evaluated in this study, extraction method and type of solvent used does not have a significant effect on PG properties of recovered binders.
- Regarding PG characteristics of the recovered binders, more recycled binder leads to stiffer materials. The results for the mixes with no or low percentages of recycled materials (Mix 1, 4, 5, and 6) showed that the recovered binder was a PG 64-28. Prior to mixing, the base PG grade was a PG 58-28. The results for Mix 7 with high recycled binder content (RAP only) shows that the binder classifies as a PG 70-22. For Mix 8 with high recycled binder content from a RAP and RAS combination, the recovered binder classifies as a PG 76-22. This indicates that although the amount of recycled binders for Mixes 7 and 8 are similar, the RAP and RAS combination change the grade of a binder differently, yielding a higher increment in the high PG grade of the binder.
- Multiple stress creep recovery (MSCR) test results showed that all recovered binders met J_{nr} requirement as follows: Mixes 1, 4, and 5, heavy traffic "H", Mixes 6 and 7 very heavy traffic "V", and the binder recovered for Mix 8, extremely heavy traffic "V". However, none of the recovered binders met the percent recovery requirement for their respective grade.

Recommendations

- It is recommended that WisDOT specifications allow the ignition test and the asphalt analyzer to be used. Section 460.2.6 for recovered asphaltic binders in the current specifications only allow for AASHTO T 164.
- If the ignition method is incorporated into WisDOT specifications, it is recommended to conduct tests at 800°F. This is particularly important considering the high mass loss for the aggregates used in this study.
- All the solvent extraction methods underestimated the actual asphalt content by approximately 0.2%. The JMF limit for asphalt content in WisDOT specifications is currently set at -0.3%. This indicates that if any of these solvent extraction methods are used, very small error due to test procedure variability is allowed. When high recycled content mixes were evaluated, the measured asphalt content was found to be more than 0.3% lower than the actual asphalt content. Therefore, the tolerances may need to be increased when high recycled content mixes are used.
- For the ignition method, the actual asphalt content was underestimated by approximately 0.1%. If the ignition oven method is used and CFs are properly applied, the current JMF limit for asphalt content may be adequate.

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1. INTRODUCTION

This report compiles the work completed in Wisconsin Highway Research Program (WHRP) Project 0092-16-02, *Asphalt Binder Extraction Protocol for Determining Amount and PG Characteristics of Binders Recovered from Asphalt Mixtures*. The objective of this project was to assess the variability and accuracy of different procedures for determining the asphalt content of mixtures and mixture components. This evaluation included virgin mixes, mixes with various content of recycled binder, and mixtures containing reclaimed asphalt pavement (RAP) and recycled asphalt shingles (RAS). The procedures include AASHTO T 164 (methods A, B, and E only) and AASHTO T 308. For each of these procedures, within-lab and between-lab variability were evaluated. In addition, a limited evaluation was conducted with a subset of the extracted binder to evaluate the variability of the PG characteristics of the recovered binders. The findings in this report are based on laboratory test results conducted at the National Center for Asphalt Technology (NCAT) and at various laboratories in Wisconsin. This project included the following tasks:

Task1. Literature Review and State of Practice. This task encompassed a literature review on different test procedures for determination of asphalt content and interviews of states to obtain their current practices and concerns regarding asphalt content determination. This information helped the researchers to refine the experimental plan.

Task 2. Work Plan and Laboratory Testing. In this task, a laboratory experiment was designed and conducted to evaluate the within-lab and between-lab variability of different test procedures for asphalt content determination using materials local to Wisconsin. In addition, the variability of PG characteristics of the recovered binders was assessed.

Task 3. Specification/Procedure Modifications and Recommendations. Based on the results of Task 2, recommendations related to test procedures, specifications and testing tolerances for WisDOT implementation were provided.

Task 4. Final Report. This task includes a final report addressing the findings of the study and Project Closeout Activities.

2. LITERATURE REVIEW

2.1 Background

Accurate determination of asphalt content is critical in the control of quality of asphalt mixtures during construction. Historically, the most common method for determining asphalt content was extraction using different methods and solvents such as trichloroethylene, methylene chloride or n-propyl bromide. This method does not require calibration factors, and the aggregate and binder properties can be determined post-extraction.

In the 1980s, the use of chlorinated solvents for asphalt extractions began to be questioned because of potential health and safety impacts and disposal difficulties. The costs of the solvents and disposal also generated additional costs required to determine asphalt content. Biodegradable solvents were evaluated by several agencies to replace the more hazardous chlorinated solvents; however, despite modifications to the extraction procedure to accommodate these new solvents, testing time increased and the accuracy of the test decreased *(1).* Nuclear gauge methods were also used to measure asphalt content. This is an indirect method that uses radiation to determine the amount of asphalt by measuring the amount of hydrogen in the asphalt mix. This method can measure the asphalt content rapidly and is reasonably accurate but doesn't allow the determination of aggregate gradation and properties of the asphalt binder.

Because of the aforementioned issues with existing tests (extraction and nuclear gage), alternative procedures to determine asphalt content were needed. This led to the development of the ignition oven method at the National Center for Asphalt Technology in the mid-1990s. Since then, the ignition oven has been adopted by laboratories around the world to eliminate the use of hazardous solvents needed for extraction testing methods. The ignition oven method is more accurate than the solvent extraction test and has benefited contractors and agencies by reducing costs while producing a safer work environment *(1, 2).*

Today, the two most common methods to determine asphalt content are solvent extraction and ignition method, as described in AASHTO T 164 "Standard Test Method for Quantitative Extraction of Asphalt Binder from Hot Mix Asphalt (HMA)" and AASHTO T 308 "Determining the Asphalt Binder Content of Hot Mix Asphalt (HMA) by the Ignition Method" *(3, 4).*

The AASHTO T 164 procedure describes different methods to extract asphalt binder from aggregate with three different solvents: trichloroethylene (TCE), normal-propyl bromide (nPB), or methylene chloride. The procedure includes the four different methods: method A (centrifuge extraction), method B (reflux extraction), method D (extraction kettle), and method E (vacuum extractor). The asphalt binder content is calculated by differences from the mass of the extracted aggregate, moisture content, and mineral matter in the extract. The asphalt binder content is expressed as a mass percent of moisture-free mixtures.

The AASHTO T 308 procedure consists of burning the asphalt from laboratory mix samples in an ignition furnace at a high temperature (1000°F). The test is complete when the change in mass does not exceed 0.01 percent for three consecutive minutes. The asphalt content is calculated as the percent mass loss of the mix minus a correction factor during the ignition process.

The precision criteria to accept test results for asphalt content for single operator and multi-laboratory evaluation for both procedures are presented in Table 1. As it can be observed, the precision requirements indicate that the ignition procedure is more precise than the extraction method. It is also interesting to see that all five methods in AASHTO T164 are assumed to have the same precision.

Table 1 AASHTO T 164 and AASHTO T 308 -precision estimate *(3, 4)*

Condition	**Standard Deviation**		**Acceptable Range of Two Tests**	
	AASHTO T 164	**AASHTO T 308**	**AASHTO T 164**	**AASHTO T 308**
Single Operator Precision: AC (%)	0.18/0.21[1]	0.069	0.52/0.58[1]	0.196
Multilaboratory Precision: AC (%)	0.29	0.117	0.81/0.83[1]	0.33

[1] Precision estimate when extractant containing 85% terpene is used.

The asphalt analyzer has recently gained popularity among some agencies to determine the asphalt content of asphalt mixtures. This new machine is an automatic closed-loop binder extractor that can be used to determine binder content and recover binder using a rotary evaporator. Work is currently underway to incorporate this unit as test method F in the American Society for Testing Materials (ASTM) D 2172 "Standard Test Method for Quantitative Extraction of Asphalt Binder from Asphalt Mixtures." The test procedures described in ASTM D 2172 are similar to the ones included in AASHTO T 164. Although there are some variations between the two standards, AASHTO T 164 will be used as the reference standard for asphalt determination by extraction for the purpose of this report.

Each standard and/or test procedure has its own advantages and disadvantages. Although precision estimates are available, the individual materials being tested or the equipment used may affect test procedure accuracy. This report provides a literature review of the current knowledge on asphalt content determination using AASHTO T 164 and AASHTO T 308.

2.2 Solvent Extraction

AASHTO T 164 involves removing the binder from the aggregate using solvents specified in the test procedure. The general methods are the centrifuge extraction, reflux extraction, and vacuum extractions. Centrifuge extraction is most commonly used, followed by reflux. Vacuum extraction is not commonly used, and not much information about its effectiveness is available. The asphalt content is determined by calculating the differences from the mass of the extracted aggregate, moisture content, and mineral matter in the extract. The extracted asphalt can be recovered by the Rotavapor method or Abson method following AASHTO T 319 "Quantitative Extraction and Recovery of Asphalt Binder from Asphalt Mixes" or AASHTO T 170 "Recovery of Asphalt Binder from Solution by Abson Method." Once the asphalt is recovered, binder characterization tests can be conducted. The size of the test sample is governed by the nominal maximum aggregate size (NMAS) of the asphalt mix as presented in Table 2.

Table 2 AASHTO T 164 minimum mass requirements by NMAS *(3)*

NMAS (mm)	Min. Mass of Specimen (g)
4.75	500
9.5	1000
12.5	1500
19.0	2000
25.0	3000
37.5	4000

In 2012, Mehta summarized the advantages, disadvantages, and differences of determining asphalt content using the methods included in AASHTO T 164 *(5)*. This information is presented Table 3. The centrifuge uses cold solvent compared to hot solvent in the reflux method. In general, cold solvent extraction method is preferred over hot because it minimizes heat hardening of the asphalt that may have an effect on binder stiffness.

Table 3 Advantages and disadvantages of AASHTO T 164 Extraction Methods *(5)*

Method	Advantages	Disadvantages
Centrifuge	Simple test and widely used It can be used for binder properties	May leave up to 4% of the total binder
Reflux	Commonly used	Aging effect from high temperature Causes hardening of binder Does not remove all the binder Should not be used for binder properties
Vacuum	Not aging from high temperature	Not much is known

Studies have shown that the asphalt is not completely removed from the aggregate when solvent extraction methods are used. In addition, the absorbed asphalt seems more difficult to remove for highway cores than for laboratory mixes *(6)*. This may lead to underestimating the asphalt content from 0.1 to 0.5 percent asphalt *(7, 8)*. For aggregates that had water absorption of more than 2.5 percent, the average asphalt content retained by the aggregate was reported to be approximately 0.75 percent *(9)*.

2.2.1 Solvents Used for Extractions

There has been some debate about the types of solvents used for extractions. When the test was first utilized, carbon disulfide was used until it was later replaced by benzene; however, in the 1950s and 1960s, the use of chlorinated solvents began to increase. At this time, solvents such as trichloroethylene (TCE) and methylene chloride became more popular. Early work conducted in 1990 by Abson and Burton, showed that TCE could be just as effective as benzene and, therefore, benzene was phased out of use *(10)*.

Additional work has been conducted to compare the impact of solvent type on asphalt content determination. In 1991, Cipione et al conducted an experiment to determine the impact of TCE versus toluene on asphalt extraction *(6)*. The study determined that TCE mixed with 15 percent ethanol proved to be the most effective solvent in the experiment. Most of the solvents were able to remove the bulk of the asphalt from the aggregate; however, the research suggested that new methods may be needed to remove all the asphalt.

Like benzene, agencies and labs have attempted to find viable substitutes for TCE due to its classification as a carcinogen *(11)*. The Florida Department of Transportation (FDOT) conducted a study to assess the possibility of using n-propyl bromide (currently allowed in AASHTO T 164) without changing testing methodologies. Preliminary research had shown this solvent to be safer; however, its effectiveness was still unknown. FDOT compared the asphalt contents from asphalt recovery tests as well as testing the recovered binders for penetration, viscosity, and time required for three different mixtures. The first mixture was a Marshall mix, the second mixture was a Superpave mix, and the third was a ground tire rubber mix. The experiment was then repeated using n-propyl bromide that had been recovered from previous extraction and recoveries. The results showed no statistical differences between the asphalt contents, penetration results, or viscosity results for any of the mixtures when comparing the TCE to the n-propyl bromide or recovered solvent. The only statistical differences occurred when determining the time required to recover the binder. For the Marshall mix and the ground tire rubber mixture, the TCE took longer than the n-propyl bromide or the recovered n-propyl bromide *(12)*.

A similar study used five different solvents (one TCE and four n-propyl bromides) to extract and recover the asphalt of six different mixtures using both the centrifuge and Rotavapor methods. The solvent type did not influence the amount of asphalt recovered nor the time of recovery. Additionally, there was little dependency seen between the PG binder tests performed on the recovered binder. A n-propyl bromide solvent reacted negatively with polymer-modified binders potentially due to incompatibilities issues with the stabilizer additives used with the solvent. One area that showed some differences was PG binder tests post-aging, where there seemed to be some interaction between test results and solvent type. Additionally, the authors noted that the variability of the test results was a concern since the within-laboratory coefficient of variation was between 23 and 30 percent and the between-laboratory was between 38 and 45 percent. Despite a lack of statistical analysis, the TCE extracted binders were numerically stiffer than those extracted with n-propyl bromide solvents *(13).*

The Asphalt Institute warns that extraction and recovery of asphalt binders containing (PPA) as a modifier or as a catalyst are affected when some commercial grades of n-propyl bromide and TCE containing a stabilizer (such as 1, 2 epoxy butane) are used. The stabilizer reacts with acid in the binder including PPA and causes the modifier to be ineffective. Therefore, it is recommended that PPA modified asphalt binders are extracted with solvents that do not contain an acid scavenger such lab-grade TCE and n-propyl bromide, toluene, or tetrahydrofuran (THF) *(14).*

The Minnesota Department of Transportation (MnDOT) conducted a cost benefit analysis of switching from d-limonene to n-propyl bromide after flashpoint testing showed that the extraction waste could be considered hazardous. D-limonene is a biodegradable solvent extracted from citrus rinds and is commonly used by MnDOT. The cost benefit analysis included on-site solvent recycling and a user extraction study. The results showed that using n-propyl bromide would result in cost savings for disposal fees, shorter extraction times, and lower solvent costs when compared to other solvents *(15).* In addition, although d-limonene is a safer alternative to the hazardous chemicals found in chlorinated solvents such as TCE, according to AASHTO T 319, biodegradable solvents are not suitable when the recovered binder is required for additional testing.

2.3 Ignition Oven Test for Asphalt Content Determination

This test method was developed at NCAT in the 1990s to address the limitations of asphalt content determination by extraction methods in which solvents are used to remove binder from the mix. Some of these limitations include potential health and safety risks, difficulty of disposal, and length of test time. The method has shown to be more accurate than the solvent extraction test and also reduces overall cost, especially when the environmental, health, and safety benefits are considered *(16, 17).*

The test procedure is described in AASHTO T 308. It consists of burning the asphalt from laboratory mix samples in an ignition oven at a high temperature (1000°F). The test is complete when the change in mass does not exceed 0.01 percent for three consecutive minutes. The asphalt content is calculated from the mass of the mix sample prior to ignition and the mass of the aggregate remaining after the ignition using equation 1.

$$P_b\ (\%) = \left[\frac{(M_i - M_f)}{M_i} \times 100\right] - C_f - MC \qquad (1)$$

Where:

P_b= measured (corrected) asphalt binder content, percent;
M_i=total mass of the HMA sample before ignition, g;
M_f= total mass of aggregate remaining after ignition, g;
C_f= the correction factor as a percent of mass of HMA sample; and
MC=moisture content of the HMA if not oven dried prior to testing**.**

The procedure establishes minimum mass requirements for the test specimen that depend on the NMAS of the mixture. This is important because large specimens of fine mixtures may result in incomplete burning of the asphalt binder *(3*). The minimum mass requirements by NMAS are presented in Table 4. The standard also indicates that the specimen sizes should not be more than 500g greater than the minimum recommended specimen mass.

Table 4 Minimum mass requirements by NMAS *(3)*

NMAS (mm)	Min. Mass of Specimen (g)
4.75	1200
9.5	1200
12.5	1500
19.0	2000
25.0	3000
37.5	4000

The procedure to obtain a correction factor involves the preparation of two samples of the asphalt mixture at the design asphalt content. The mix calibration samples are tested and the calibration factor is calculated as the difference between the actual and measured asphalt binder contents expressed as a percentage of the HMA mass.

The AASHTO T 308 procedure requires that an asphalt correction factor be determined by testing a set of specimens with known asphalt content for each job mix formula (JMF). A correction factor needs to be obtained each time a change greater than 5% in stockpiled aggregate proportions occurs. The test method also requires that additional testing be conducted at a lower temperature (900 ±8°F) if the measured asphalt correction factor exceeds 1.0%. This will occur with “problematic aggregates” such as dolomitic aggregates.

The AASHTO procedure describes two methods for the ignition test. Method A is the procedure when an ignition oven with an internal balance is used. For these units the test is conducted automatically to determine the asphalt content. This is by far, the most common method. Method B is the procedure when units with no internal balance are used. For these units, the specimen basket with the asphalt mixture sample needs to be removed from the oven and weighted a number of times until the change in measured mass of the specimen does not exceed 0.01 percent of the initial specimen mass.

There are basically two ignition oven units based on their heating mechanism: convection and infrared units. Both of them are included in the AASHTO standard. For convection units, the user can select the test temperature. For infrared units the user only has the option to select between 3 profiles: default profile that is recommended for most materials, option 1 designed for soft aggregates such as dolomites (or any mixture with a large correction factor) and option 2 recommended for some mixes with higher asphalt content, such as stone matrix asphalt and special modifiers.

2.3.1 Effect of Mixture Components in Asphalt Content Determination by Ignition

In 2000, Prowell and Youtcheff conducted a study in Virginia to evaluate mixture components that might affect the ignition oven correction factor *(18)*. The experiment was designed to investigate the effects of the amounts of lime, sulfur, calcium carboxylates, and fines. A 9.5 mm NMAS Superpave mix and asphalt binders with different chemistries were included in each experiment. They found that the variability in the percentage of hydrated lime added to the mixture had a significant effect on the ignition furnace correction factor. The results are summarized in Table 9.

The correction factor varied from 0.64 with no hydrated lime to 0.13 with 2% hydrated lime. The variability reported was large enough to cause non-compliance with quality control tests according to Virginia DOT's specifications. They also reported that the amount of sulphur present in the asphalt binder significantly affected the mass loss occurring during the ignition process, but to a lesser degree than the lime content. The sulfur content ranged from 1.2% to 6.4%. An accurate calibration of the ignition furnace is also dependent on relatively constant sulfur content in the asphalt when lime is present. This should not be a problem for a given binder, but it may be an issue if crudes with variable sulfur contents are used. The other two components, calcium carboxylates and fines, didn't have a significant effect on the ignition furnace correction factors, but they recommended that additional testing should be conducted to assess the effect of fines variability with other aggregates, particularly dolomites.

Table 5 Effect of hydrated lime content on ignition furnace factors *(18)*

Description	Measured Asphalt Content (%)			Average	Standard Deviation	Correction Factor
	1	2	3			
Control	5.84	5.80	5.89	5.84	0.045	0.64
+0.5% hydrated lime	5.63	5.65	NA	5.64	NA	0.44
+1 % hydrated lime	5.47	5.50	5.45	5.47	0.025	0.27
+1% hydrated lime aged 2 hr	5.46	5.59	5.44	5.50	0.081	0.30
+2% hydrated lime	5.32	5.31	5.35	5.33	0.021	0.13

2.3.2 Asphalt Content Determination for Plant Produced Mixes

In 2002, a round robin study was conducted in Florida and documented by Sholar et al. to determine precision values for asphalt content and gradation for plant-produced mixtures *(19).* They indicated that plant-produced mixtures have different possible sources of variability compared to lab prepared samples. These additional sources of variation include differences in an asphalt mixture within the truck box, collection of samples from the truck, splitting of the mixture into samples for testing, and differences in equipment and operation. The study included twelve laboratories and nine different mixtures including six superpave mixtures (four coarse mixtures and two fine mixtures) and three open graded friction courses (OGFCs). The major aggregate types used were limestone and granite. RAP was included in some of the mixtures, but no information about the percentages used was reported. Each mix was sampled out of the truck box at the asphalt plant. The study recommended new precision values presented in Table 6 to be used for plant-produced mixtures only. Florida Method FN 5-563 for the determination of asphalt content of asphalt mixtures by the ignition method recommends these values *(20).*

Table 6 Precision statement for asphalt content for plant produced mixtures-Florida *(20)*

Condition	Standard Deviation	Acceptable Range of Two Test Results
Within-Laboratory: Asphalt Content (%)	0.1138	0.32
Between-Laboratories: Asphalt Content (%)	0.1563	0.44

2.3.3 Ignition Testing with High Loss Aggregates

For problematic aggregates such as dolomitic aggregates, loss of some aggregate weight during ignition, and breakdown of the aggregate particles due to high temperatures, can cause the measured asphalt content to be greater than the actual content and also can cause changes in the gradation of the recovered aggregate. Some samples may continue to burn for longer periods of time because the weight does not stabilize.

Some states, such as Indiana, have reported problems with the use of ignition ovens with this type of aggregate. A study conducted by Kowalski et al. in 2010 evaluated dolomites with the ignition method *(21).* They indicated that the high temperature required during ignition testing produced decomposition/chemical change of aggregates, which caused mass loss to continue after the binder was burned off. As a result, the mass at the end of the test was higher and more variable. As part of this study, the research team investigated different variables that can influence mass loss during the tests. These variables included test temperature, test time, and temperature distribution inside the oven.

In their study, the influence of test temperature on the mass loss during the ignition test was investigated using three temperatures: 538°C, 482°C, and 427°C (1000°F, 900°F and 800°F). A 25.0 mm NMAS mix with a PG 64-22 binder was used in this evaluation. The design binder content was 4.6%. They found that the observed mass losses were a function of the test temperature, with higher losses as temperature increased. They reported mass losses of approximately 8.5% at 538°C (1000°F), 6.5% at 482°C (900°F), and 5.8% at 427°C (800°F). They concluded that since the asphalt content of this mix was 4.6%, the difference in mass losses must represent the mass loss due to thermal decomposition of the dolomite; these increases were 3.9%, 1.5%, and 1.2% respectively. A strong relationship between test temperature and sample mass loss was found as shown in Figure 1. They also found that the oven temperature exceeded the target temperature in all cases and that the higher the test temperature, the faster the temperature was exceeded. This indicates that by decreasing the temperature, significant effect on the mass loss and rate of mass loss can be achieved.

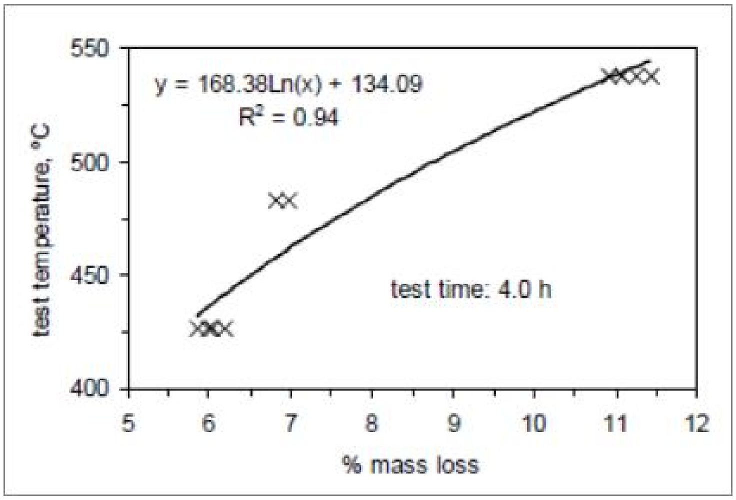

Figure 1 Mass loss as a function of test temperature *(21)*

The influence of test time on mass loss was also investigated as part of this study. Ignition tests between 90 and 240 minutes were conducted at 538°C (1000°F), with the results presented in Figure 2. This linear relationship shows that the mass loss was 0.2% for each 10-minute period.

Ignition tests were also conducted on the dolomite aggregate blend with no binder, and the results showed that the mass loss was 0.1% for each 10-minute period. This indicates that test time at high temperature impacts the mass loss, which confirmed that the binder in the mix, as it ignited, increased the mass loss rate.

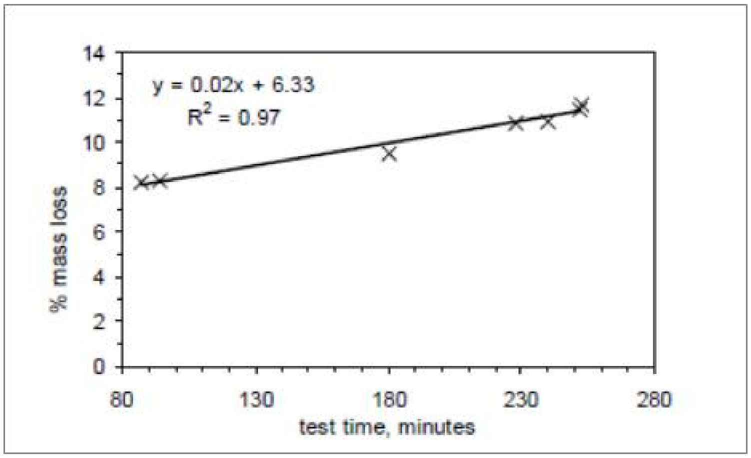

Figure 2 Mass loss as a function of test time *(21)*

The temperature inside the ignition oven was monitored with the oven thermistor and two additional thermocouples (TC) installed in the top (TC1) and bottom (TC2) of the sample baskets. Figure 3 summarizes the results of tests conducted using a mixture containing dolomite (HM-1) and a dolomite aggregate sample (DBL-1without binder) at 483°C (900°F) (similar trends were obtained at 427°C (800°F) and 538°C (1000°F)). This figure shows that the temperature inside the oven was not constant and that the temperature in the top basket was higher than the one in the lower basket. When the temperature was stabilized, the differences became relatively constant. The temperature differences between the bottom and top baskets was 58°C (136°F), between top basket and oven was 35°C (95°F), and between oven and bottom basket was 24°C (75°F). These differences were very similar at different target temperatures as shown in Figure 4. They concluded that this temperature difference might cause an increase in mass loss and decomposition of the aggregate and that placing the mix sample only in the bottom basket, would result in a more uniform temperature, hence limiting the decomposition of high loss aggregates.

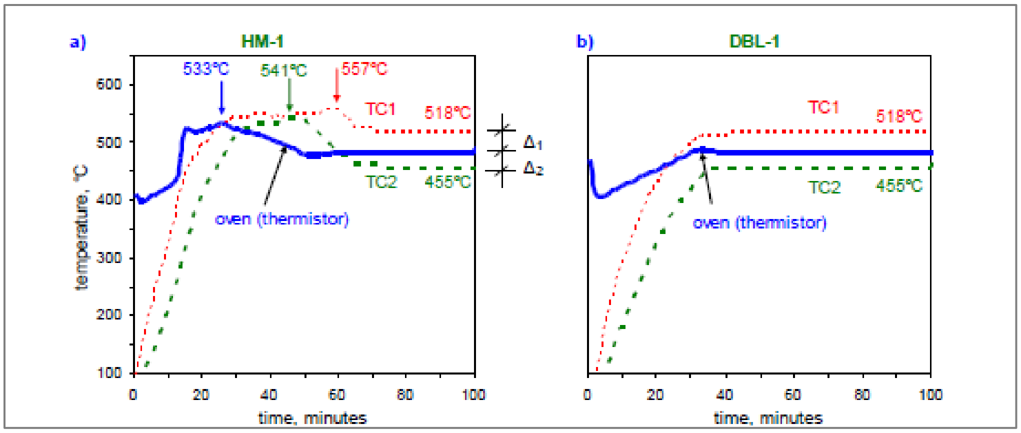

Figure 3 Temperature distribution inside ignition oven, TC1, TC2 and oven thermistor (HMA mix and dolomite aggregate sample) *(21)*

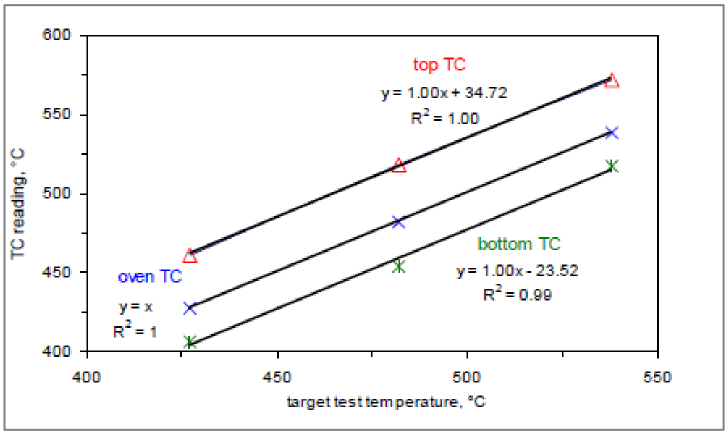

Figure 4 Relationship between oven and thermocouples in the top and bottom basket when temperature stabilized (target temperatures, 427°C (800°F), 483°C (900°F) and 538°C (1000°F)) *(21)*

Aggregate properties before and after ignition tests were also evaluated in this study. A 25.0 mm NMAS with 4.6% binder was used to compare the results of bulk and apparent specific gravities and absorption of the aggregate before and after ignition. They found that the ignition test affects the results significantly. After ignition, specific gravities decrease and absorption increases. The results are summarized in Table 7.

.

Table 7 Specific gravities and absorption of dolomite before and after ignition *(21)*

Mix at 538°C (1000°F)	Before Ignition	After Ignition
Bulk specific gravity	2.710	2.423
Apparent Specific gravity	2.773	2.642
% absorption	0.8	3.4

Based on the findings of this study, modifications to the ignition oven test procedure were suggested for the high weight loss materials in Indiana. In order to reduce the mass loss, it was recommended to reduce the total mass by one-half, using only the bottom basket and running the test at 800°F. The modified protocol was recommended for problematic aggregates or aggregates with correction factors greater than or equal to 1, or when the test was not completed in less than 90 minutes. The modified procedure was verified using six plant produced mixes with problematic aggregates. Comparing these ignition tests to solvent extraction results showed very similar results. The current Indiana DOT test method to determine asphalt content by ignition allows the test to be conducted at 800°F if dolomite is used in the mix *(22).*

2.3.4 Variability of Ignition Correction Factors

As part of a project sponsored by the National Cooperative Highway Research Program (NCHRP) 9-56, a study was conducted to assess the variability of ignition oven correction factors (CFs) for different brands of ignition ovens and mixes to better understand the implications of sharing CFs *(23).* Twenty-three laboratories used various brands of ovens to test four mixes containing aggregates with varying CFs. Table 8 summarizes the average asphalt content (AC), average CF, within-lab and between-lab standard deviations for each mix, and the acceptable range of two test results from the same laboratory and from different laboratories. For Mixes 1 and 2, within-lab and between-lab standard deviations were relatively similar: 0.089 and 0.074 for within-lab, and 0.131 and 0.111 for between-lab. These numbers are close to the values recommended in AASHTO T 308, 0.069 and 0.117 respectively. As the correction factors increased for Mixes 3 and 4, the standard deviations also increased; the within-lab results for Mixes 3 and 4 were 0.112 and 0.178, and the between-lab standard deviations were 0.264 and 0.403.

Mixes 1 and 2 used the same mix design and the only difference was that mix 2 included 1% lime. From the results presented in Table 9 the addition of lime caused a significant variation in the CF from 0.12 with no lime to -0.23 with 1% lime resulting in a difference of 0.35. Table 8 shows that the within-lab and between-lab statistics are very similar for mixes 1 and 2. The primary difference between these two mixtures is that lime was used in mix 2. The data showed that mix 2 actually had slightly less variability in test results than mix 1. The authors indicated that if lime is accurately added to a mixture at a consistent content, then the ignition test should be satisfactory for use, at least for low weight loss aggregates. In addition, since no testing was performed with lime for high weight loss aggregates, they indicated that it is not certain what the effect may be for high weight loss aggregate. If the amount of lime varies during construction, this will affect the CF and possibly result in an error in asphalt content measurement.

The study concluded that CFs were significantly different for the different mixes even when the same unit brand was used. The within-lab and between-lab precision developed in the study suggests that different precision statements are necessary for aggregates with high breakdown potential and that the current precision included in AASHTO T 308 was likely developed for low mass loss aggregate making it unacceptable to use for aggregates with higher CFs *(24).*

Table 8 Precision statement for asphalt binder content by mix *(24)*

Mix	Actual AC %	Measured AC%	CF	s_r	s_R	r	R
1	5.2	5.32	0.12	0.089	0.131	0.250	0.367
2	5.2	4.97	-0.23	0.074	0.111	0.207	0.311
3	6.2	7.08	0.90	0.112	0.264	0.314	0.740
4	6.1	7.31	1.21	0.178	0.403	0.499	1.128
AASHTO T 308				0.069	0.117	0.196	0.330

s_r=repeatability standard deviation
s_R=reproducibility standard deviation
r=repeatability acceptable range of two test results
R=reproducibility acceptable range of two test results

Ways to minimize variability in asphalt CFs were also evaluated as part of the study. Samples for the different mixes used in the inter-laboratory study were prepared at optimum -1% and optimum +1% asphalt content. Tests were conducted at 800°F and 1000°F. The results are presented in and clearly show that decreasing the test temperature decreases the aggregate weight loss for all mixes not containing lime. In fact, the mix containing lime had less weight loss for lower temperature than for higher temperature. The use of a temperature of 800°F effectively removed the binder from the mixes without substantial changes in testing time. It was determined that ignition tests conducted at a lower temperature (800°F) proved to be effective in reducing the variability in measured asphalt content since the lower temperature reduced the asphalt correction factors for asphalt mixes.

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Table 9 Asphalt content CFs at different test temperatures *(24)*

Mix	Temperature (°F)	AC (%)	Average CF	Average Test Time(min)
1	800	4.2	0.00	59
1	800	6.2	-0.04	63
1	1000	4.2	0.16	44
1	1000	6.2	0.13	61
2	800	4.2	-0.27	51
2	800	6.2	-0.30	57
2	1000	4.2	-0.18	36
2	1000	6.2	-0.22	60
3	800	5.2	0.29	67
3	800	7.2	0.20	65
3	1000	5.2	0.91	47
3	1000	7.2	0.83	61
4	800	5.1	0.72	70
4	800	7.1	0.80	57
4	1000	5.1	3.05	116
4	1000	7.1	3.08	111

2.3.5 RAP/RAS Asphalt Content Determination with Ignition

When using RAP, test procedures need to be adjusted for measuring asphalt content. The AASHTO T 308 procedure does not provide adequate guidance when using RAP or RAS materials.

One of the problems with using the ignition test to determine the asphalt content for RAP is the determination of the correction factor for asphalt content. Since the raw materials are not available, it is not possible to determine the asphalt correction factor. The normal procedure with virgin mixtures is to mix a known gradation with a known asphalt content and to conduct the ignition test. The difference between the actual gradation and actual asphalt content between the measured gradation and measured asphalt content is identified as the correction factor. This procedure works well for virgin mixtures, but becomes more difficult when RAP is used. If the amount of RAP is relatively small, the CF of the mixture can be determined without significant issues. However, as the amount of these recycled materials is increased, the need to have a more specific procedure for these mixtures increases.

One approach for handling RAP is to determine the asphalt content and gradation of these materials before being mixed with the virgin materials. These test results for the RAP along with the known asphalt content and gradation of the virgin materials can be used to calculate the actual asphalt content and gradation of the recycled mixture. The correction factor can then be established by conducting the ignition test on the recycled mixture and subtracting the measured asphalt content and gradation from the actual asphalt content and gradation.

One caution that should be considered when determining the asphalt content of RAS is the sample size used for testing. The RAS typically contains 20 to 30 percent asphalt binder which is much more than the 5 to 6 percent that normally exists in HMA. If the sample size is too large the equipment may be damaged as combustion begins due to the large amount of asphalt binder *(25).*

States like Texas have incorporated RAP and RAS in their specifications for asphalt content by ignition. TEX-236-F procedure indicates that for RAS materials, the asphalt binder may not be removed during combustion if the sample size is too large and recommends a sample size of 500-700 grams *(26).* Other states have also incorporated ignition oven procedures or modifications to their specification to account for the use of RAP/RAS, including Minnesota, North Carolina, South Carolina, and Virginia.

2.4 Comparison of Ignition and Extraction Methods for Asphalt Content Determination

In 1998, a study was conducted by Prowell to evaluate three different methods for asphalt content determination: ignition oven, solvent extractions and nuclear gauge *(27).* A Thermolyne convection unit was used for the ignition method. A total of three surface mix designs with different gradations and aggregate sources from Virginia were used. A statistical comparison was conducted to evaluate the asphalt content test results using the three methods. This comparison is presented in Table 10. The standard deviations were calculated on the difference between the measured and actual asphalt content. They concluded that the most accurate method was the ignition method.

Table 10 Summary of statistics by test method *(27)*

Test Method	No. Observations	Standard Deviation % AC	Bias %AC
Extraction	23	0.29	-0.113
Ignition	24	0.11	-0.033
Nuclear Gauge	108	0.14	0.070

A survey was conducted by NCAT in 2009 to gather information from contractors on how they managed and processed RAP materials *(28).* The test methods used for determining asphalt content are presented in Figure 5. According to the survey, approximately 85 percent of the contractors preferred the ignition oven compared to solvent extraction.

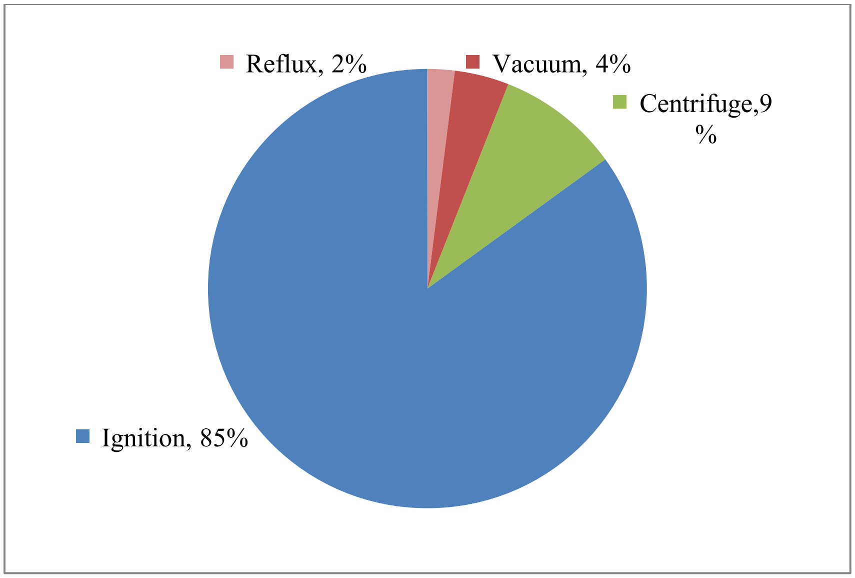

Figure 5 Methods used to determine asphalt content of RAP stockpiles *(28)*

A study conducted in 2012 by Hajj et al. evaluated the impact of extraction methods (i.e. centrifuge, reflux, and ignition oven) on the binder content and extracted aggregate properties of laboratory produced RAP mixtures with four aggregate sources: Alabama (hard limestone), California (granodiorite), Florida (soft limestone), and Nevada (rhyolite) *(29).* The results indicate that the true asphalt binder contents were consistently higher than the asphalt binder contents obtained from all of the extraction methods. The centrifuge method yielded the lowest asphalt binder content and the ignition method appears to yield the most accurate results for the asphalt content of the RAP mixes. Figure 6 shows the asphalt binder contents obtained from each extraction method.

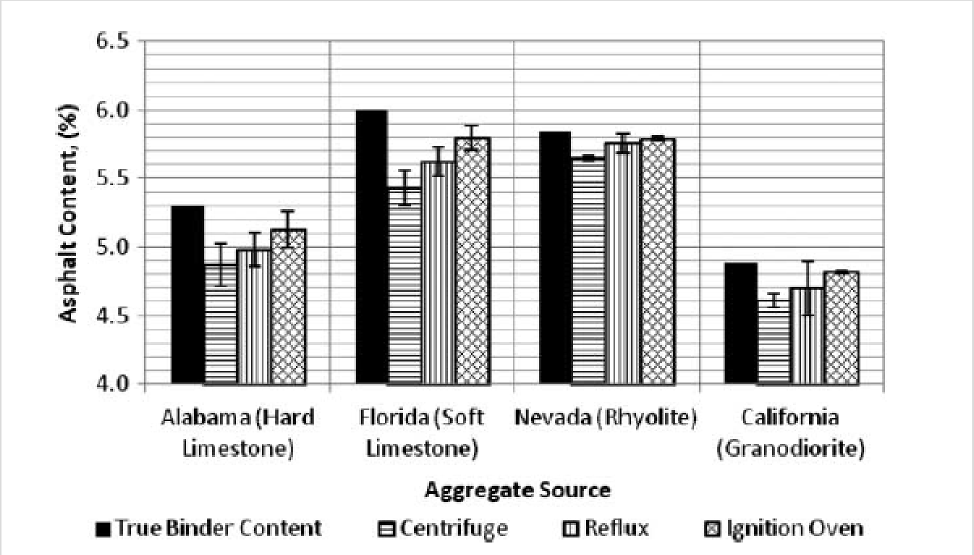

Figure 6 Comparison of Binder Contents for Different Extraction Methods *(29)*

The authors indicated that no correction factors were used for the ignition method results since development of a correction factor is not possible with most RAP sources in the field. They also indicated that the solvent extraction methods do not appear to remove all of the aged binder from RAP, and therefore measured RAP asphalt contents using these methods tend to be lower than they actually are.

The study also evaluated the properties of the extracted aggregates. Table 11 shows the combined statistical significance for all four aggregate sources by extraction method. The results in this table show that the binder contents measured by the different extraction methods were statistically significantly lower than the true asphalt binder content except for two aggregates using the reflux method, but this was attributed to the large amount of variability observed in the reflux measurements for these two aggregates. For the aggregate properties, the authors concluded that overall, none of the extraction methods consistently impacted the properties of the aggregates. Although none of the extraction methods had a significant impact on the size distribution of the coarse aggregates, the effect for the fine aggregates was aggregate source dependent. It was also concluded that coarse and fine specific gravities was method dependent.

Table 11 Comparison of binder content and aggregate properties for RAP mixes with different aggregate sources *(29)*

Properties	**Centrifuge**			**Reflux**			**Ignition**		
	SL	**NS**	**SH**	**SL**	**NS**	**SH**	**SL**	**NS**	**SH**
Asphalt Binder Content	4	-	-	2	2	-	4	-	-
Sieve Analysis									
12.5 mm sieve	-	4	-	-	4	-	-	4	-
4.75 mm sieve	-	4	-	1	2	1	-	3	1
2.36 mm sieve	1	2	1	2	1	1	1	2	1
0.30 mm sieve	1	1	2	1	2	1	-	2	2
0.075 mm sieve	1	2	1	1	2	1	2	-	2
Coarse bulk specific gravity, dry	2	1	1	-	2	2	3	1	-
Fine bulk specific gravity, dry	-	2	2	-	1	3	1	1	2

SL: significantly lower; NS: not significant; SH: significantly higher

In 1999, Beheres et al. conducted a study for the Nebraska Department of Roads Bituminous Laboratory to assess the hidden environmental and liability costs associated with the different methodologies for asphalt content determination *(30)*. In this study, five potential asphalt extraction methods were compared using a total cost analysis: solvent extraction using trichloroethylene (TCE), solvent extraction using an alternative solvent, solvent extraction using TCE and a solvent recovering reclaimer, ignition oven, and an ignition oven and solvent combination. This analysis included capital costs, operation and maintenance costs, and environmental, health, and safety costs. Fuzzy set theory was used to address uncertainty in the model. When a traditional model that did not include the hidden costs was conducted, the three best options were the ignition oven, ignition oven with solvents, and solvent extraction with a reclaimer. These three options were basically equivalent; however, when all the costs were considered, the ignition oven was by far the option with the least cost.

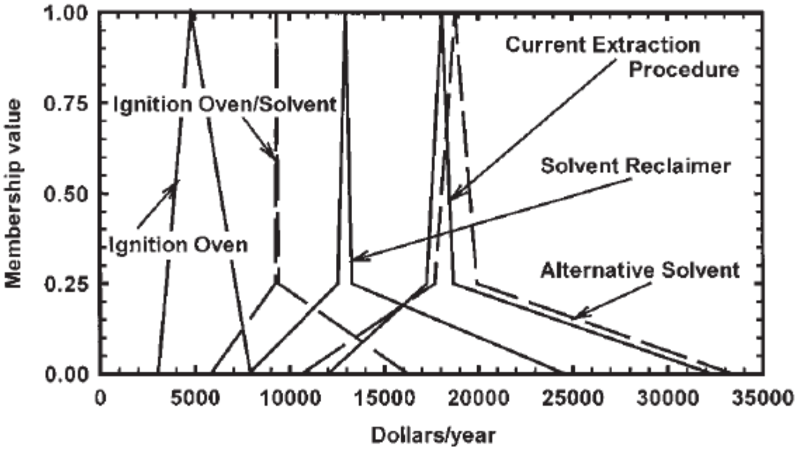

Figure 7 Annual costs of each asphalt extraction option including hidden costs (extraction constant at 970 per year) ***(30)***

2.5 State Interviews Regarding Asphalt Binder Determination

The research team conducted phone interviews with Iowa, Minnesota, and Illinois to find out how they determined asphalt content in mixtures. There were no set questions for the interviews; however, they generally began by asking the state engineer about determining asphalt content. The notes from these interviews are attached in Appendix A. It is interesting to note that none of the states actually measure asphalt content for quality control and quality assurance. Iowa measures the asphalt content of recycled materials for mix design using solvent extraction. They currently use n-propyl bromide due to lower costs and ease of disposal. Minnesota uses d-limonene, and Illinois uses TCE as the preferred solvent.

Another interesting note is that Illinois does not measure the asphalt content of recycled materials. Instead, the state tells the contractor what to use as the asphalt content for the RAP or RAS.

Iowa and Minnesota do not measure asphalt content for quality control because they assess film thickness of the binder and determine asphalt content with different methods. Iowa, for example, pays for asphalt from the tank stick or a calibrated flow meter. Guidance from these states will be limited, as they approach the issue of asphalt content from a very different methodology.

3. EXPERIMENTAL PLAN DESCRIPTION

The work conducted for this study is presented in two phases: tests conducted at the NCAT laboratory and tests conducted at various laboratories in the Wisconsin area. With the support of the project oversight committee (POC) and information gathered in the literature review and interviews, the test plan originally presented in the project proposal was modified and finalized. The following information summarizes the key requirements in terms of materials and test procedures that the research team and POC agreed upon.

- *Asphalt content determination procedures to be evaluated*: ignition (per AASHTO T 308), centrifuge and reflux (per AASHTO T 164, methods A and B), and asphalt analyzer (per manufacturer recommendations).
- *Aggregates*: four aggregates from different areas of the state that are commonly used in asphalt mixes. It was considered important that the aggregates had a wide range of LA abrasion values because that would significantly impact the correction factor used in ignition testing.
- *RAP:* two RAP sources from different parts of the state.
- *RAS*: one post-consumer (PC) source.
- *Asphalt binder:* one PG grade (PG 58-28) for all mixes.
- *Asphalt mixes*: four virgin and four recycled mixes with various contents of recycled binder (one low: 5-10%, one medium: 15-20%, and two high: 30-35%). One of the high recycled content mixtures had to be a RAP plus RAS combination.
- *Asphalt mix design*: since all of the mix designs submitted by the POC included RAP at percentages different than the one selected for this study, the designs had to be used as a baseline and adjusted to achieve virgin and recycled mix requirements.

Table 12 provides the testing matrix of the testing completed at NCAT (Lab 1). Four different aggregate sources were used to prepare four virgin mixes. In addition, four mixes which include various contents of recycled binder were tested for variability. The recycled mixtures used aggregate #4. The recycled binder contents for these four mixes are also presented in Table 12**.** Two sources of RAP and one RAS material were also evaluated. Three replicates of each mixtures, RAP and RAS materials were tested using the centrifuge extraction, reflux extraction, and ignition oven methods. Triplicates were required to determine within sample and between sample variability on the same material set for a singular lab.

Table 12 NCAT testing plan

Procedure	Aggregate Source				RAP 1	RAP 2	RAS	Mixtures with			
	1	2	3	4				5-10% Recycled Binder	15-20% Recycled Binder	30-35% Recycled Binder(RAP Only)	30-35% Recycled Binder(RAP +RAS)
Centrifuge	3	3	3	3	3	3	3	3	3	3	3
Reflux	3	3	3	3	3	3	3	3	3	3	3
Ignition	3	3	3	3	3	3	3	3	3	3	3
PG Grade	NA			1	NA			1	1	1	1

NCAT also determined the variability of the PG properties of a select sample of the testing matrix as shown in Table 12. One virgin aggregate with a known binder and the four mixtures with recycled materials were PG characterized for variability. Extracted and recovered binders were PG graded to determine the true high, intermediate, and low grades of the binder.

In order to assess the between-lab variability, the following five Wisconsin contractors/agencies participated in the study: Bitumix (Lab 2), Mathy Construction Co. (Lab 3), Payne and Dolan (Lab 4), WisDOT-Bureau of Technical Services (Lab 5), and Benhke Materials Engineering (BME) (Lab 6). The laboratory testing plan that was conducted by Wisconsin partners' to assess the within-lab and between-lab variability of the different test methods is presented in Table 13. To develop this matrix, it was required that, as a minimum, two laboratories (including NCAT) would conduct tests for each test method.

Table 13 Wisconsin labs testing plan

Test Method	Agg. 1 (Mix 1)	Agg. 4 (Mix 4)	5-10% Recycled Binder (Mix 5)	15-20% Recycled Binder (Mix 6)	30-35% Recycled Binder (Mix 7)	30-35% Recycled Binder, RAP +RAS (Mix 8)
Centrifuge	Bitumix Mathy					
Asphalt analyzer	Payne and Dolan, Behnke Materials Engineering (BME) Mathy					
Reflux	Payne and Dolan					
Ignition	WisDOT Payne and Dolan					
PG Grade	N/A	Mathy (after centrifuge and analyzer) Bitumix (after centrifuge) Payne and Dolan (after analyzer)				

The participant labs used a variety of solvents to extract binder for centrifuge, asphalt analyzer, and reflux extractions. For the study, it was decided to have the labs use their normal solvents as they are all allowed by current standards. Furthermore, additional sources of variability could be introduced if a lab was asked to handle a solvent they were not used to working with. The solvents that were used are as follows:

- NCAT: trichloroethylene (TCE) for centrifuge and reflux;
- Mathy: toluene for centrifuge, TCE for asphalt analyzer;
- Bitumix: n-propyl bromide (nPB) for centrifuge;
- BME: TCE for asphalt analyzer; and
- Payne and Dolan: TCE for asphalt analyzer.

3.1 Materials and Mix Designs

The aggregate selected by the POC to be used in this study are shown in Table 14. The table includes Los Angeles (LA) abrasion wear results for each material for 100 and 500 revolutions. The LA abrasion results were used to select "hard" and "soft" aggregates. Higher LA abrasion results indicated a softer material.

Table 14 Aggregate types used for mix designs

Aggregate	Source	LA Abrasion Wear (100)	LA Abrasion Wear (500)
#1	Kings Bluff	10.9	40.9
#2	Merrillan	2.5	15.8
#3	Vienna	6.8	30.1
#4	Larsen	5	23.1

Once the materials were received, each aggregate type was oven dried, and then washed-gradations and aggregate specific gravity testing was performed on each material.

A total of eight mix designs were completed. The first four were virgin designs created by altering the JMFs sent by the different Wisconsin contractors by removing the recycled materials and replacing with virgin materials that allowed for all volumetric criteria to be met. The other four mix designs corresponded to recycled mix designs using Aggregate #4. One of these designs (15-20% recycled binder) was simply verified based on the JMF sent. The other three had to be created by adding and/or removing recycled materials to yield the desired recycled binder content. Table 15 summarizes the design aggregate gradation, design volumetrics and asphalt content for the four virgin mix designs used in this study. All these mixes are 12.5mm NMAS designed to 4% air voids. Asphalt content for these mixes ranged from 5.6 to 6.0%

Table 15 Virgin mix designs

Sieve Size (mm)	% Passing			
	Mix #1	Mix #2	Mix #3	Mix #4
19	100	100	100	100
12.5	95.4	94.5	97.1	96.5
9.5	89.7	83.1	88.8	89.7
4.75	69.2	69.1	71.4	77.1
2.36	53.4	52.6	51.7	54.9
1.18	42.7	39.7	39.5	39.4
0.6	29.2	25.3	30.7	28.7
0.3	17.2	11.4	15.5	15.6
0.15	8.7	6.3	7.4	8.2
0.075	4.1	4.2	5.0	5.6
Opt. AC, %	5.8	5.9	6.0	5.6
VMA, %	14.8	15.8	14.6	14.8
VFA, %	72.4	74.7	72.5	73.1
D/B	0.9	0.81	1.1	1.2
G_{mm}	2.494	2.426	2.49	2.53
G_{mb}	2.395	2.329	2.39	2.429
G_{sa}	2.748	2.657	2.768	2.777
G_{se}	2.733	2.652	2.741	2.768
G_{sb}	2.645	2.602	2.628	2.693
P_{ba}, %	1.26	0.74	1.61	1.03
P_{be}, %	4.6	5.21	4.53	4.59

Four mix designs were completed using Aggregate #4 and different amounts of recycled materials. Table 16 shows the target recycled binders for these mixes. The same RAP was used for all four recycled mixes. The gradations and asphalt contents obtained from TCE extractions for the RAP and RAS are shown in Table 17. To calculate the amount of virgin binder to add during mix design for the recycled mixes, the asphalt content from TCE extractions was used for the recycled materials. The gradation obtained after TCE extraction was also used for mix designs. Table 18 shows a summary of the four recycled mix designs including aggregate gradation, design volumetrics and asphalt content. The table also includes the actual recycled binder ratio (RBR) for each mix. These ratios were limited to the maximum allowable ratio recommended in Wisconsin specifications, section 460.2.5. The recycled binder contents were calculated using the measured binder content of the RAP and RAS obtained from solvent extraction.

Table 16 Target recycled binder contents

Mix	Source	Designation	% Recycled Binder
#5	Larsen	Low Recycle (RAP)	5 - 10
#6	Larsen	Moderate Recycle (RAP)	15 - 20
#7	Larsen	High Recycle (RAP)	30 - 35
#8	Larsen	High Recycle (RAP and RAS)	30 - 35

Table 17 RAP and RAS properties

Sieve Size (mm)	**% Passing**	
	RAP	**RAS**
12.5	100.0	100.0
9.5	97.6	99.8
4.75	80.1	98.6
2.36	61.2	98.2
1.18	48.4	81.9
0.6	38.5	60.7
0.3	25.2	51.2
0.15	15.8	40.5
0.075	10.9	30.4
Source	Larsen	Vienna
% AC (Centrifuge)	4.33	23.90

Table 18 Recycled mix designs

Sieve Size (mm)	**% Passing**			
	Mix #5	**Mix #6**	**Mix #7**	**Mix #8**
19.0	100.0	100.0	100.0	100.0
12.5	96.5	96.8	96.8	96.5
9.5	89.4	89.9	89.8	89.1
4.75	76.1	74.9	73.8	74.6
2.36	56.9	55.7	54.8	55.6
1.18	41.5	40.8	40.4	40.0
0.6	30.2	29.8	29.8	28.4
0.3	15.0	15.3	16.4	15.6
0.15	6.9	7.1	8.4	7.9
0.075	5.0	4.8	5.6	5.2
Optimum AC, %	5.7	5.6	5.4	5.5
VMA, %	15.9	15.3	14.6	14.8
VFA, %	75.5	74.1	72.8	73.4
D/B	1.03	1.00	1.20	1.14
G_{mm}	2.513	2.523	2.542	2.541
G_{mb}	2.412	2.422	2.440	2.439
G_{sa}	2.771	2.771	2.778	2.781
G_{se}	2.754	2.758	2.776	2.780
G_{sb}	2.707	2.703	2.704	2.709
P_{ba}, %	0.66	0.76	0.99	0.97
P_{be}, %	5.10	4.83	4.49	4.62
RAP, %	10	24	39	25
RAS, %	--	--	--	3
Rec. Binder Ratio (RBR)	0.091	0.187	0.312	0.325

3.2 Mix Sample Preparation for Testing

Once the mix designs were completed, samples for each mix were batched to provide mixes with a minimum of 1500 g (sample size requirement for 12.5 mm NMAS mixes) and placed in wax-lined boxes. These samples were then randomized and tested both at NCAT and Wisconsin labs. A total of 72 samples were prepared for NCAT testing and 144 for the Wisconsin labs. Each participant lab received specific instructions for the samples and tests to be conducted. Three samples per mix were provided. For PG grading, one full set of tests was required per mix. Labs were asked to have one technician perform all of the tests to reduce variability associated with multi-operator testing.

Each participant laboratory was asked to follow the procedure set forth by AASHTO T 164 and/or AASHTO T 308. For ignition samples, it was required that all samples be tested at 800°F. This required modification in the AASHTO test procedure was based on findings from NCHRP 9-56, which indicated that tests conducted at this lower temperature proved to be effective in reducing the variability in measured asphalt content. This may be particularly important for recycled mixes since the CF of recycled materials cannot be determined.

For PG grading, laboratories were required to recover the asphalt binders using the rotary evaporator per ASTM D5404 and to treat the recovered residue as un-aged binder.

4. RESULTS AND ANALYSIS

The following sections present the results of the laboratory testing conducted at NCAT and at the different partner labs.

4.1 RAP and RAS Evaluation

As mentioned before, the recommendation was to run ignition tests at 800°F for the different mixes under evaluation. Since this approach has not been evaluated for recycled materials, a preliminary evaluation was conducted to assess how temperature affects the ignition test results for recycled materials. The approach was to run tests at three different temperatures: 1000°F (current procedure), 900°F and 800°F.

Based on current recommendations from other state agencies such as Texas and based on NCAT experience, the sample size for RAS materials was limited to 500 grams.

Table 19 summarizes the results of the asphalt content determination for the RAP and RAS with ignition at different temperatures. From the results, it is evident that decreasing the test temperature decreases the average and variability of the test results. For RAP 1 material (Larsen source) the standard deviation was reduced from 0.35 to 0.09 for tests conducted at 1000°F and 800°F respectively. For RAP 2 (Kings Bluff) the standard deviation was reduced from 0.21 to 0.11 for tests at 1000°F and 800°F, respectively. For the RAS material, the standard deviation was reduced from 1.37 to 0.34 for tests conducted at 1000°F and 800°F respectively. Despite this fact, for the RAS material it was observed that for tests conducted at 800°F, there was some unburned asphalt left in the residue. Figure 8 shows the RAS material after ignition test. No residue was observed for tests conducted at 900°F. Based on these results, it is recommended to conduct ignition tests for asphalt content determination of RAS materials at 900°F.

Table 19 RAP and RAS asphalt content test results by ignition at different temperatures

Material	Test Temp.	Sample 1	Sample 2	Sample 3	Avg.	Std. Dev.	CV (%)
RAP 1 (Larsen)	1000	6.22	5.52	5.83	5.86	0.35	5.99
	900	5.26	5.46	5.03	5.25	0.22	4.10
	800	5.30	5.20	5.1	5.21	0.09	1.64
RAP 2 (Kings Bluff)	1000	6.29	5.87	6.04	6.07	0.21	3.48
	900	5.85	5.56	5.58	5.66	0.16	2.86
	800	5.28	5.09	5.29	5.22	0.11	2.16
RAS	1000	29.26	28.6	26.63	28.16	1.37	4.86
	900	26.82	27.00	26.43	26.75	0.29	1.09
	800	26.42	26.09	25.75	26.09	0.34	1.28

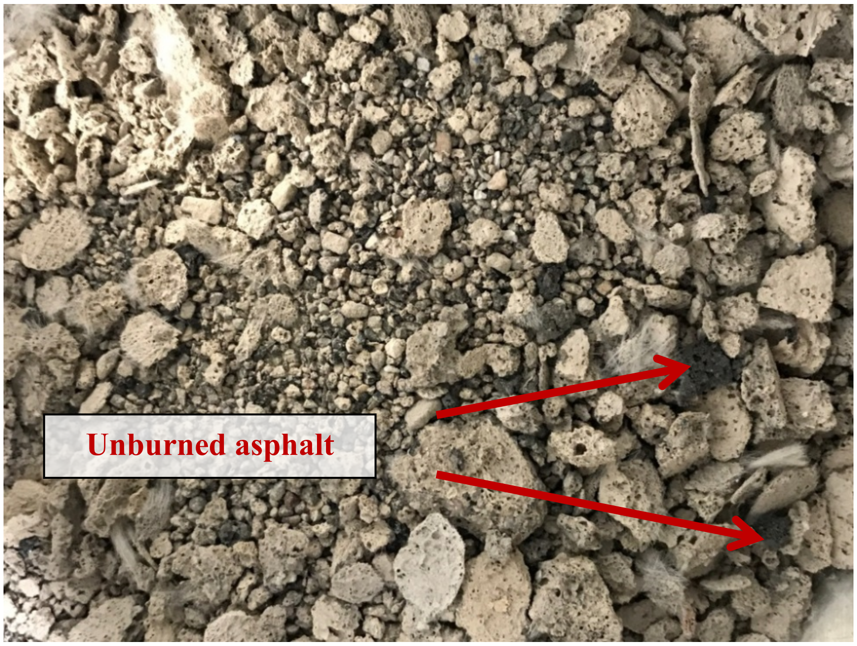

Figure 8 RAS aggregate after ignition at 800°F

A statistical summary of the asphalt content for RAP and RAS materials for different methods is presented in Table 20 with a graphical summary shown in Figure 9 and Figure 10 for RAP and RAS materials, respectively. For RAP materials, ignition results correspond to tests conducted at 800°F and, for RAS materials, the ignition results correspond to tests conducted at 900°F. From these test results it can be observed that, in general, centrifuge tends to yield the lowest asphalt content, with the exception of RAP 2 material, followed by reflux and ignition. It is important to keep in mind that ignition tests still yields the highest asphalt content since correction factors can't be determined, but as mentioned before, ignition test results at lower test temperatures yielded lower asphalt content for recycled materials.

Table 20 RAP and RAS asphalt content test results by test method

Material	**Test Method**	**Sample 1**	**Sample 2**	**Sample 3**	**Avg.**	**Std. Dev.**	**CV (%)**	**Statistical Grouping**
RAP 1	Centrifuge	4.36	4.3	4.33	4.33	0.03	0.69	A
	Reflux	4.76	4.77	4.84	4.79	0.04	0.94	B
	Ignition	5.3	5.2	5.13	5.21	0.09	1.64	C
RAP 2	Centrifuge	4.78	4.58	4.73	4.7	0.1	2.22	A
	Reflux	4.22	4.33	4.54	4.36	0.17	3.81	B
	Ignition	5.28	5.03	5.29	5.2	0.15	2.83	C
RAS	Centrifuge	23.8	24.4	23.5	23.9	0.49	2.03	A
	Reflux	24.9	24.5	24	24.46	0.42	1.71	B
	Ignition	26.42	26.1	25.75	26.09	0.34	1.28	B

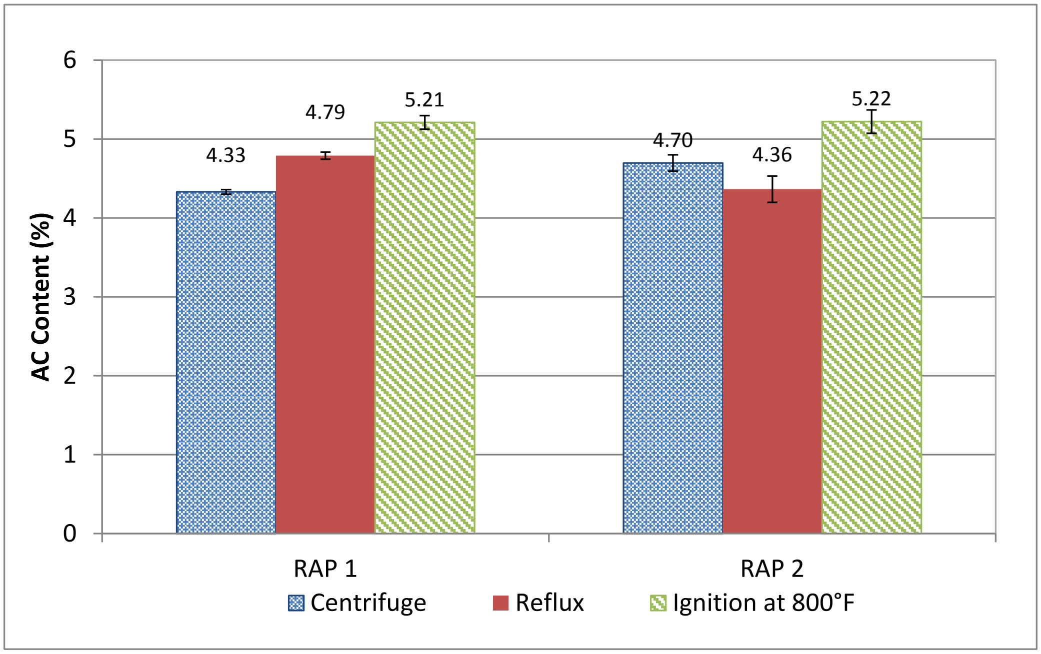

Figure 9 RAP asphalt content by test method

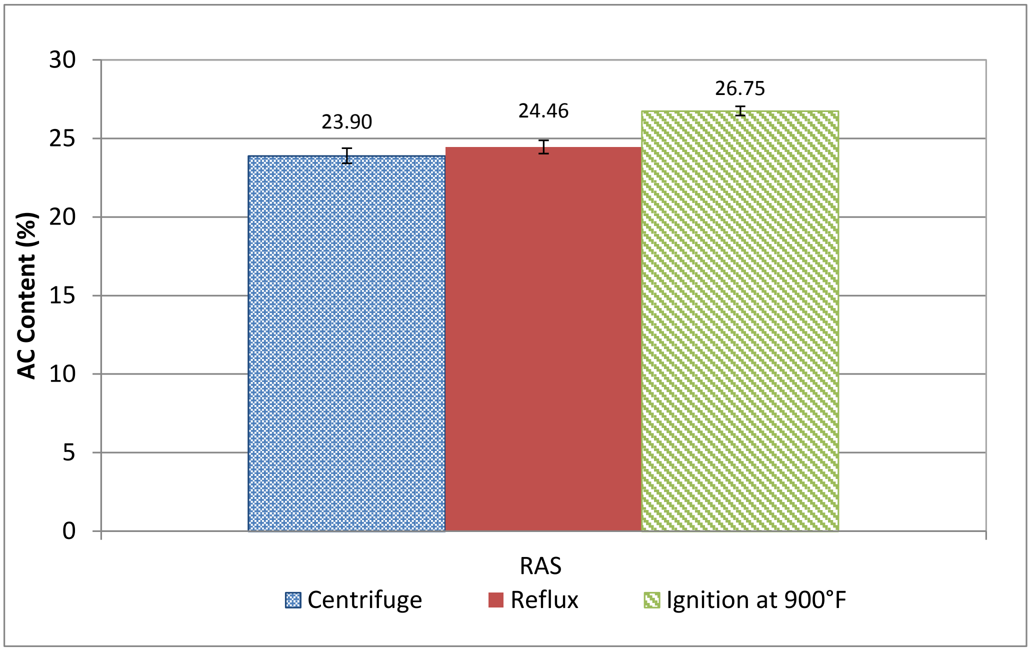

Figure 10 RAS asphalt content by test method

The results were also compared using the Tukey tests for multiple means comparison by each material type. The results are also summarized in Table 20. From this analysis means that do not share a letter are significantly different at a significance level of 0.05. Based on these results, it can be observed that for both RAP materials all the tests methods are considered statistically different. For RAS material, centrifuge and reflux results are considered statistically equivalent, but ignition results are different.

4.2 Asphalt Content Determination of Virgin and Recycled Mixes

For this section, analyses were conducted to evaluate the within-lab and between-lab of the test results for the different test methods. To accomplish this objective, the results of the interlaboratory study were collected and analyzed in accordance with ASTM E 691 and ASTM C 802 *(31, 32)*. These standards are recommended to determine the within-laboratory and between-laboratory precision values for a test method. The within-laboratory precision, or repeatability, provides an expectation of the difference in test results between replicates measured on the same material in the same laboratory by one operator using the same equipment. The between-laboratory precision provides an expectation of the difference in test results between measurements made on the same material in two different laboratories. Analyses for each test method were performed by mix type.

4.2.1 Data Consistency

ASTM E 691 uses k and h statistics to evaluate consistency of the test result for the determination of data that may be suspected outliers. The h statistic is an indicator of how one laboratory's average for a material compares with the average of the other laboratories. The h statistic is based on a two-tailed Student's t test. The k statistic is an indicator of how one laboratory's variability for a given set of replicate samples compares with that of all the other laboratories. A complete description of statistical calculations is included in ASTM E 691.

The h values can be positive or negative with zero representing a laboratory average equal to the average of the laboratory averages. The k values are always positive with a value of 1 representing the average within-lab variability. The k statistic is based on the F-ratio from a one-way analysis of variance. A k value greater than 1 represents higher within-lab variability when compared to the rest of the laboratories combined, and k values less than 1 indicate less within-lab variability compared to the rest of the laboratories combined. ASTM E 691 recommends critical k and h values with a 0.5 percent significance level. This significance level is recommended because experience has shown that 1.0 percent significance values were too sensitive and the 0.1 percent significance values were insensitive to possible outliers.

Following the ASTM E 691 procedure, the within-lab and between-lab standard deviations were calculated for asphalt content for each test procedure using Equations 2 through 7. For each set of lab test results, k and h statistics were calculated using Equations 8 and 9, respectively. The k and h statistics for each mix are compared to the critical values, which are a function of the number of laboratories and number of replicates. For an interlaboratory study consisting of three laboratories and three replicates, the ASTM E 691 recommended critical values of h and k are 1.15 and 1.67 respectively.

$$\bar{\bar{x}} = \sum_1^p \bar{x}/p \tag{2}$$

$$s_{\bar{x}} = \sqrt{\sum_1^p d^2/(p-1)} \tag{3}$$

$$d = \bar{x} - \bar{\bar{x}} \tag{4}$$

$$s_r = \sqrt{\sum_1^p s^2/p} \tag{5}$$

$$s_L^2 = s_{\bar{x}}^2 - s_r^2/n \tag{6}$$

$$s_R = \sqrt{s_L^2 + s_r^2} \tag{7}$$

$$h = \frac{d}{s_{\bar{x}}} \tag{8}$$

$$k = \frac{s}{s_r} \tag{9}$$

Where:

$\bar{\bar{x}}$ = average of the laboratory averages;
$\bar{x}$ = the average of the test results for each laboratory;
p = number of laboratories;
d = deviation for each laboratory;
s = standard deviation of the test results for each laboratory;
n = number of test results for each lab;
$s_{\bar{x}}$ = standard deviation of lab averages;
s_r = repeatability standard deviation;
s_L = between-laboratory standard deviation;
h = between-laboratory consistency statistic;
k = within-laboratory consistency statistic; and
s_R = reproducibility standard deviation.

It is important to point out that although ASTM E 691 recommends having a minimum of six laboratories, it was not possible for this study due to the availability of participating labs from Wisconsin. Therefore, the statistics presented in the next section are based on test results of three laboratories for centrifuge, asphalt analyzer, and ignition, and two laboratories for test results using the reflux method.

4.2.2 Data Analysis for Centrifuge Test Results

Centrifuge test results from the interlaboratory study included six mixes, two virgin mixes (1 and 4) and four recycled mixes (5, 6, 7 and 8). Three replicates were conducted for each mix. Two additional virgin mixes (2 and 3) were evaluated at NCAT only.

Following the procedure recommended in ASTM E 691, the test results were analyzed to determine any inconsistent data. These calculations include the parameters presented in Equations 2 through 9: within-laboratory and between-laboratory standard deviations and the k and h statistics. Each mix was analyzed separately. Tabular summaries of data by mix are presented in Table 22 through Table 26.

The within-laboratory k statistics are presented in Figure 11. Examination of this figure indicates that for lab 1A, h values are consistently higher than 1 for all mixes and closer to the critical k value, that is, a higher within-laboratory variability compared with that for all of the laboratories combined. For mix 4: lab 1A, the k value approaches the critical value but was not exceeded in any case.

The between laboratory h statistics are presented in Figure 12. The critical h value was reached for mix 1- lab 3A, mix 5-lab 2 and, for mix 8-lab 3A the critical was close to being reached. These values are shown in bold in Table 21, Table 23 and Table 26. Although the critical values were reached for mixes 1 and 5, it was decided not to exclude the data for these mixes because the critical value was barely exceeded and because ASTM E 691 suggests that if no clerical, sampling or procedure errors are uncovered, the data should be retained.

Table 21 Interlaboratory test results for mix 1-centrifuge

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 1A	1	5.78	5.83	5.69	5.60	5.71	0.12	0.013	0.10	0.61	1.59
Lab 2			5.73	5.66	5.70	5.70	0.04	0.001	0.09	0.55	0.48
Lab 3A			5.38	5.45	5.41	5.41	0.04	0.001	-0.19	**-1.15**	0.48

$\bar{\bar{x}}$ = average of labs averages 5.61

$s_{\bar{x}}$ = standard deviation of cell averages 0.167

s_r =repeatability standard deviation 0.073

s_L =between-lab standard deviation 0.161

s_R =reproducibility standard deviation 0.177

Table 22 Interlaboratory test results for mix 4-centrifuge

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 1A	4	5.6	5.43	5.58	5.50	5.50	0.08	0.006	0.08	0.45	**1.65**
Lab 2			5.49	5.45	5.46	5.47	0.02	0.000	0.04	0.23	0.46
Lab 3A			5.32	5.32	5.30	5.31	0.01	0.000	-0.11	-0.69	0.25

$\bar{\bar{x}}$ = average of labs averages 5.43

$s_{\bar{x}}$ = standard deviation of cell averages 0.101

s_r =repeatability standard deviation 0.045

s_L =between-lab standard deviation 0.097

s_R =reproducibility standard deviation 0.107

Table 23 Interlaboratory test results for mix 5-centrifuge

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 1A	5	5.7	5.61	5.68	5.42	5.57	0.13	0.018	-0.03	-0.66	1.43
Lab 2			5.63	5.74	5.56	5.64	0.09	0.008	0.05	**1.15**	0.96
Lab 3A			5.56	5.59	5.58	5.58	0.02	0.000	-0.02	-0.49	0.16

$\bar{\bar{x}}$ = average of labs averages 5.6

$s_{\bar{x}}$ = standard deviation of cell averages 0.041

s_r =repeatability standard deviation 0.094

s_L =between-lab standard deviation 0.000

s_R =reproducibility standard deviation 0.094

Table 24 Interlaboratory test results for mix 6-centrifuge

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 1A	6	5.6	5.32	5.12	5.30	5.25	0.11	0.012	0.00	0.09	1.53
Lab 2			5.28	5.20	5.18	5.22	0.05	0.003	-0.02	-1.04	0.74
Lab 3A			5.28	5.28	5.24	5.27	0.02	0.001	0.02	0.95	0.32

$\bar{\bar{x}}$ = average of labs averages 5.24
$s_{\bar{x}}$ = standard deviation of cell averages 0.023
s_r =repeatability standard deviation 0.072
s_L =between-lab standard deviation 0.000
s_R =reproducibility standard deviation 0.072

Table 25 Interlaboratory test results for mix 7-centrifuge

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 1A	7	5.4	5.31	5.03	5.07	5.14	0.15	0.025	0.01	0.12	1.49
Lab 2			5.02	4.95	5.10	5.02	0.08	0.006	-0.10	-1.06	0.71
Lab 3A			5.21	5.17	5.28	5.22	0.06	0.003	0.09	0.93	0.53

$\bar{\bar{x}}$ = average of labs averages 5.13
$s_{\bar{x}}$ = standard deviation of cell averages 0.099
s_r =repeatability standard deviation 0.106
s_L =between-lab standard deviation 0.078
s_R =reproducibility standard deviation 0.131

Table 26 Interlaboratory test results for mix 8-centrifuge

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 1A	8	5.5	5.29	5.55	5.40	5.41	0.13	0.017	0.05	0.70	1.51
Lab 2			5.33	5.47	5.38	5.39	0.07	0.005	0.03	0.44	0.82
Lab 3A			5.25	5.28	5.29	5.27	0.02	0.000	-0.09	**-1.14**	0.24

$\bar{\bar{x}}$ = average of labs averages 5.36
$s_{\bar{x}}$ = standard deviation of cell averages 0.076
s_r =repeatability standard deviation 0.087
s_L =between-lab standard deviation 0.057
s_R =reproducibility standard deviation 0.104

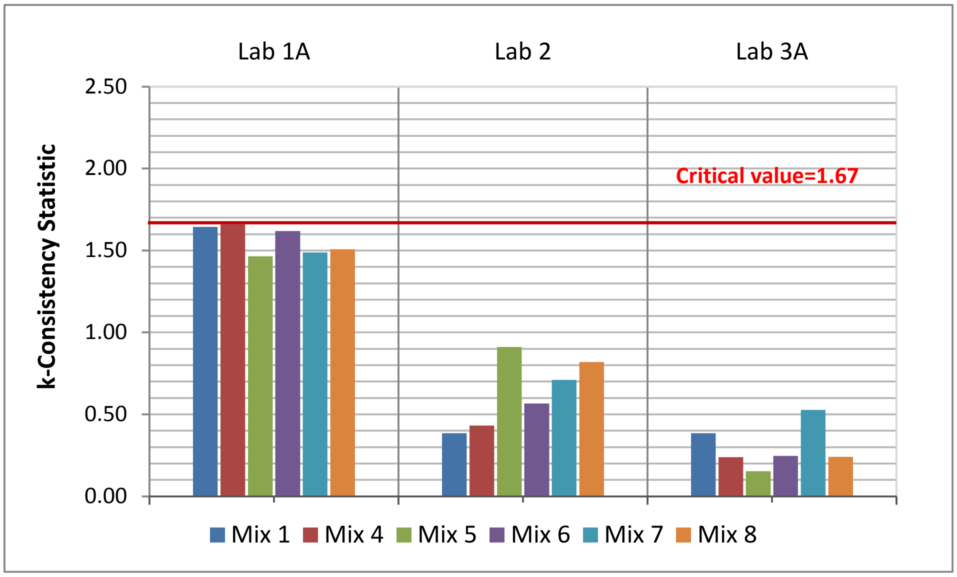

Figure 11 Consistency statistic k for asphalt content -centrifuge-all labs and materials

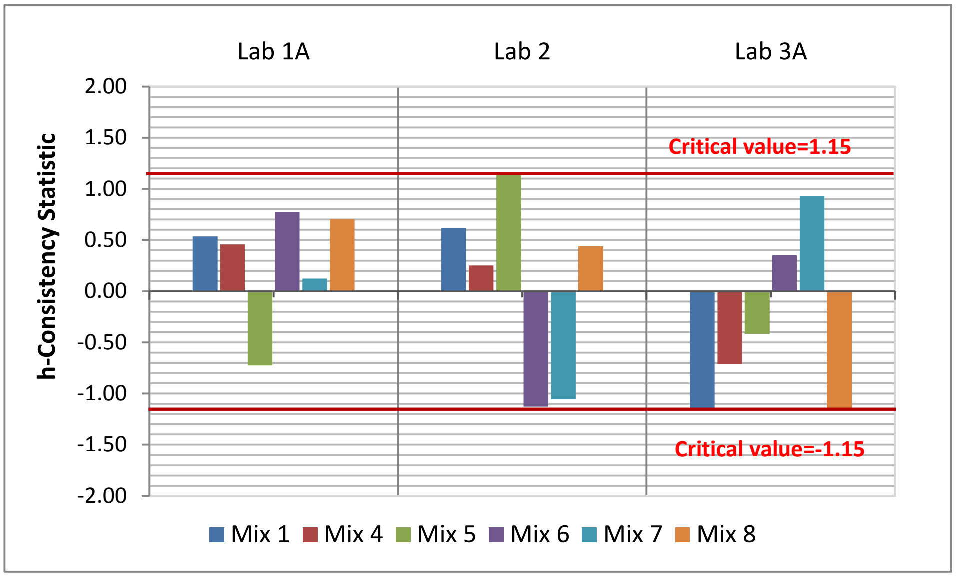

Figure 12 Consistency statistic h for asphalt content -centrifuge-all labs and materials

An additional analysis recommended in ASTM C 802 is to plot the average test results for each lab against the materials/mixes under evaluation to determine if each laboratory is following the same trend for each mix. This information is presented in Figure 13. This figure also includes the actual AC content for each mix. Examination of this figure shows that, in general, similar patterns of change from material to material for all the labs are observed. This supports the decision of keeping all the test results to recommend within-lab and between-lab precision estimates.

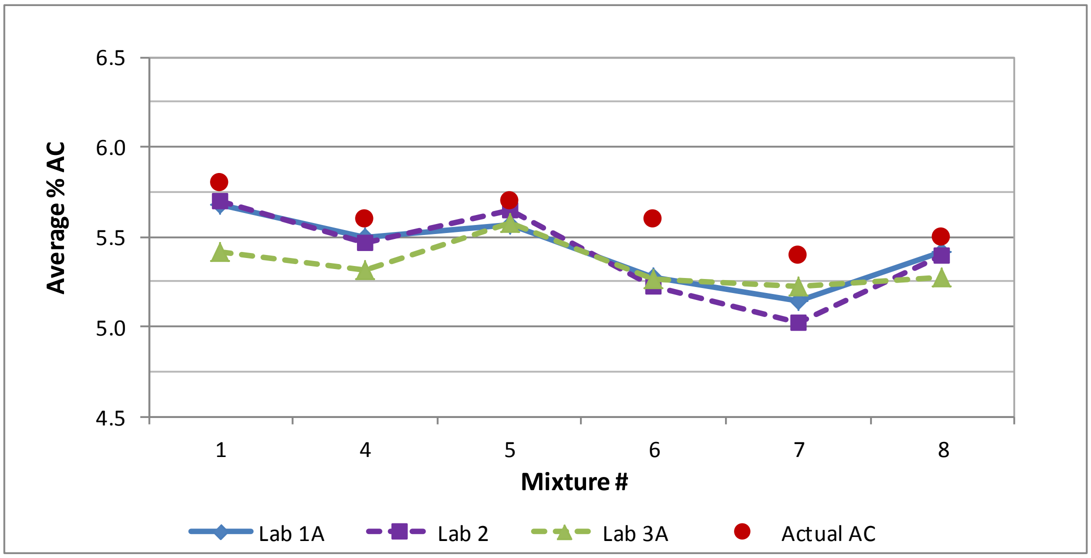

Figure 13 Average percentage AC content per laboratory vs. mixture number for centrifuge test

For every mix tested, Table 27 summarizes the actual and average asphalt contents, the asphalt content difference expressed as the difference of actual minus measured, standard deviations, coefficients of variation and acceptable range of two test results for every mix. The acceptable range of two test results is calculated by multiplying the within-laboratory and between laboratory standard deviations by 2.8 per ASTM E 691. From this table it can be observed that no trend was found for virgin mixes and recycled mixes. Table 27 also summarizes the average within-lab and between-lab precision statistics for all mixes. Standard deviations and acceptable range of two test results calculated are within the current precision statement of AASHTO T 164 presented previously in Table 1 and reproduced in Table 28 for convenience. It can also be noticed that the average difference between actual and measured asphalt content is 0.21%, which may be an indication that the solvent extraction may not be able to remove all of the binder from the mix.

Table 27 Within-lab and between-lab for asphalt content for centrifuge

Mix #	Actual AC, %	Measured AC, %	AC Diff., % (Actual-Measured)	Standard Deviation		Coefficient of Variation		Acceptable Range of Two Test Results	
				Within-Lab	Between-Lab	Within-Lab	Between-Lab	Within-Lab	Between-Lab
1	5.8	5.61	0.19	0.073	0.177	1.3	3.2	0.204	0.495
4	5.6	5.43	0.17	0.045	0.107	0.8	2.0	0.127	0.301
5	5.7	5.60	0.10	0.094	0.094	1.7	1.7	0.263	0.263
6	5.6	5.24	0.36	0.023	0.104	0.4	2.0	0.066	0.292
7	5.4	5.13	0.27	0.106	0.131	2.1	2.6	0.297	0.367
8	5.5	5.36	0.14	0.087	0.104	1.6	1.9	0.242	0.290
Averages			**0.21**	**0.071**	**0.120**	**1.3**	**2.2**	**0.200**	**0.335**

Table 28 AASHTO T 164 -precision estimate *(3)*

Condition	Standard Deviation	Acceptable Range of Two Tests
Single Operator Precision: AC (%)	0.18	0.52
Multilaboratory Precision: AC (%)	0.29	0.81

Table 29 shows the test results for mixes not included in the interlaboratory study and evaluated at NCAT only. These results indicated that the standard deviations for both mixes are within the average within-lab standard deviation of 0.071 estimated for all the other mixes as reported in Table 27.

Table 29 Centrifuge test results for mixes not included in the interlaboratory study

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.
			1	2	3		
Lab 1A	2	5.9	5.87	5.84	5.82	5.84	0.03
	3	6.0	5.80	5.77	5.88	5.82	0.06

4.2.3 Data Analysis for Asphalt Analyzer Test Results

Similar to the analysis conducted for centrifuge test, results were analyzed to determine any inconsistent data. Data was analyzed separately for each mix. Tabular summary of data for mix 7 is presented in Table 30 as an example of the data. Tables for the remaining five mixes are included in Appendix B.

Figure 14 presents the within-laboratory k statistics. These results indicate that for lab 3B- mix 7, k value was 1.7 (shown in bold), and barely exceeded the critical k value. A closer look at the individual test results for lab 3B- mix 7 indicate that replicate 2 shows a significantly lower asphalt content when compared with the other two replicates and to the actual asphalt content. This test result is shown in bold in Table 30. Since the standard deviation calculated for this mix and lab-mix combination is significantly different than any other individual lab results for any of the other mixes, it was decided to deem this number as an outlier. The revised statistics are presented in Table 31 for this particular mix after the outlier was removed.

The between-laboratory h statistics are presented in Figure 15. The critical h value was not reached in any case. Therefore, no additional test results were deemed as outliers.

Table 30 Interlaboratory test results for mix 7-asphalt analyzer

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 4B	7	5.4	4.91	5.00	4.92	4.94	0.05	0.002	-0.11	-0.73	0.32
Lab 3B			5.12	**4.69**	5.16	4.99	0.26	0.068	-0.06	-0.41	**1.70**
Lab 6			5.20	5.21	5.23	5.21	0.02	0.000	0.16	1.14	0.10

$\bar{\bar{x}}$ = average of labs averages 5.05

$s_{\bar{x}}$ = standard deviation of cell averages 0.144

s_r =repeatability standard deviation 0.153

s_L =between-lab standard deviation 0.114

s_R =reproducibility standard deviation 0.191

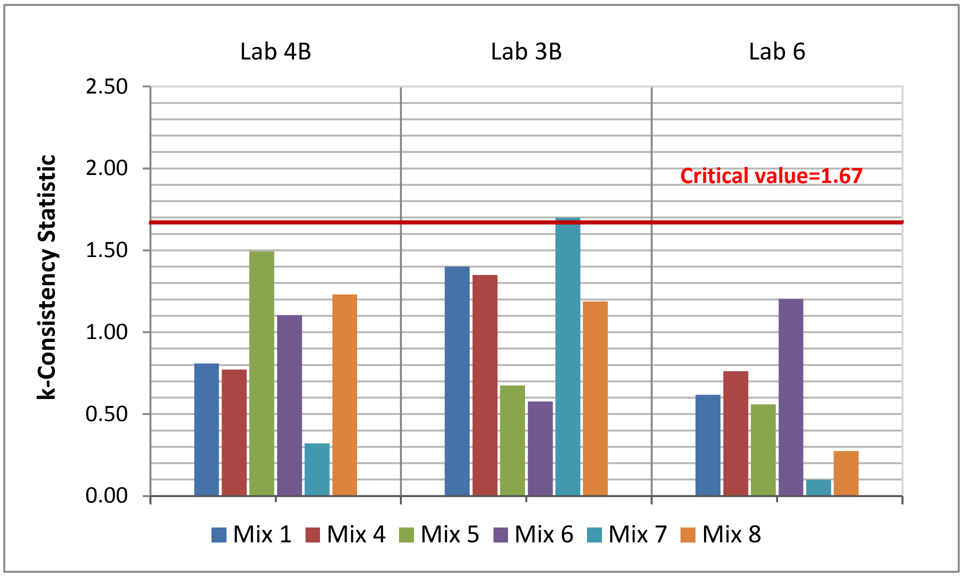

Figure 14 Consistency statistic k for asphalt content –analyzer-all labs and materials

Table 31 Interlaboratory test results for mix 7- analyzer-after outlier removed

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 4B	7	5.4	4.91	5.00	4.92	4.94	0.05	0.002	4.91	-0.69	0.32
Lab 3B			5.12	-	5.16	5.14	0.03	0.001	5.12	0.60	0.18
Lab 6			5.20	5.21	5.23	5.21	0.02	0.000	5.2	1.08	0.10

$\bar{\bar{x}}$ = average of labs averages 5.10

$s_{\bar{x}}$ = standard deviation of cell averages 0.153

s_r =repeatability standard deviation 0.034

s_L =between-lab standard deviation 0.151

s_R =reproducibility standard deviation 0.155

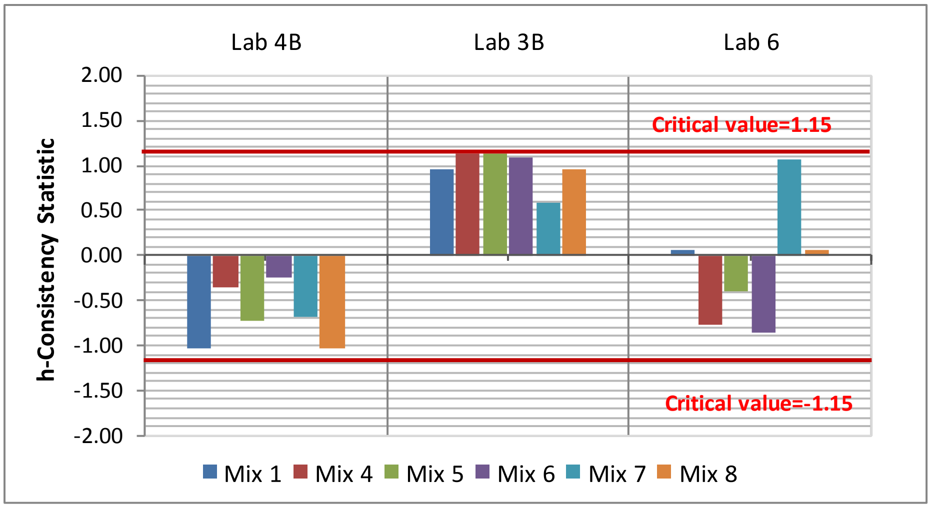

Figure 15 Consistency statistic h for asphalt content –analyzer-all labs and materials

Figure 16 shows the average test results for each lab against the mixes under evaluation to determine if each laboratory is following the same trend for each mixture. Examination of this figure shows similar patterns of change from mix to mix for all the labs.

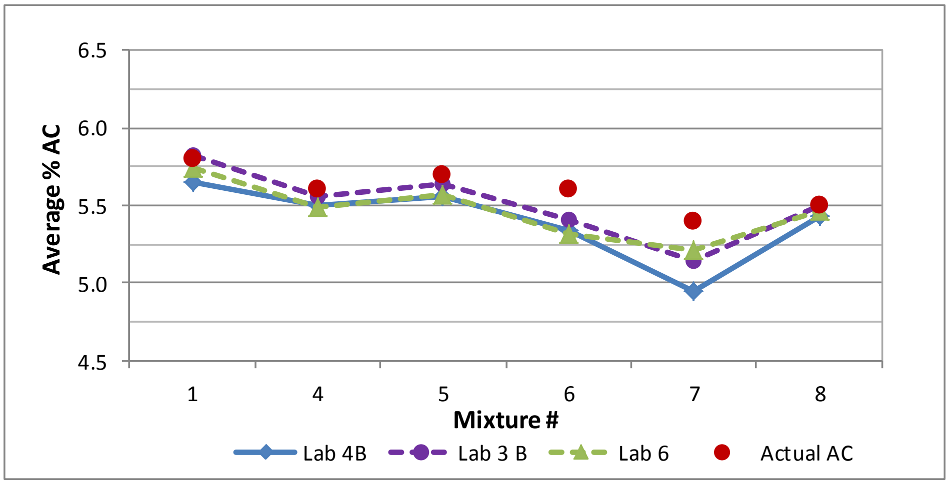

Figure 16 Average percentage asphalt content per laboratory vs. mixture number for asphalt analyzer test

Table 32 summarizes the actual and average asphalt contents, the asphalt content difference expressed as the difference of actual minus measured, standard deviations, coefficients of variation, and acceptable range of two test results for each mix. From this table it can be observed that between lab standard deviation for mixes 7 and 8 (high recycled content mixes) are higher than for the other mixes. This trend was not observed for the centrifuge test results. The average within-lab and between-lab precision statistics for all mixes are also presented. Standard deviations and acceptable range of two test results calculated are within the current precision statement of AASHTO T 164 and similar to the results reported for centrifuge tests. The average difference between actual and measured AC content is 0.14%, which indicates that some asphalt is left after extraction.

Table 32 Within-lab and between-lab for Asphalt Content for Asphalt Analyzer

Mix #	Actual AC, %	Measured AC, %	AC Diff, % (Actual - Measured)	Standard Deviation		Coefficient of Variation		Acceptable Range of Two Test Results	
				Within-Lab	Between-Lab	Within-Lab	Between -Lab	Within-Lab	Between -Lab
1	5.8	5.74	0.06	0.049	0.094	0.9	1.6	0.138	0.264
4	5.6	5.51	0.09	0.065	0.065	1.2	1.2	0.181	0.183
5	5.7	5.59	0.11	0.037	0.051	0.7	0.9	0.104	0.143
6	5.6	5.35	0.25	0.089	0.089	1.7	1.7	0.249	0.249
7	5.4	5.10	0.30	0.034	0.142	0.7	2.8	0.095	0.399
8	5.5	5.47	0.03	0.126	0.126	2.3	2.3	0.353	0.353
Averages			**0.14**	**0.067**	**0.095**	**1.2**	**1.7**	**0.187**	**0.265**

4.2.4 Data Analysis for Reflux Test Results

Reflux testing was only conducted by two laboratories. ASTM E 691 does not include critical k and h values for less than three laboratories. Therefore, results were only analyzed to determine potential tolerances for the test procedure for the mixes under evaluation. Data was analyzed separately for each mix. Tabular summary of data for mix 1 is presented in Table 33 as an example of the data. Tables for the remaining five mixes are included in Appendix B.

Table 33 Interlaboratory test results for mix 1-reflux

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d
			1	2	3				
Lab 1B	1	5.8	5.24	5.22	5.28	5.25	0.03	0.001	-0.17
Lab 4B			5.63	5.49	5.67	5.60	0.09	0.009	0.17

$\bar{\bar{x}}$ = average of labs averages 5.62
$s_{\bar{x}}$ = standard deviation of cell averages 0.167
s_r = repeatability standard deviation 0.073
s_L = between-lab standard deviation 0.161
s_R = reproducibility standard deviation 0.177

Although only two laboratories were available to estimate the tolerances for the within-lab and between-lab for the reflux procedure, it was decided to assess the potential variability of the test method. Table 34 summarizes the actual and average asphalt contents, the asphalt content difference expressed as the difference of actual minus measured, standard deviations, coefficients of variation and acceptable range of two test results for each mix. The average within-lab and between-lab precision statistics for all mixes are also presented. Standard deviations and acceptable range of two test results calculated are within the current precision statement of AASHTO T 164 and they are higher than the results reported for centrifuge and asphalt analyzer tests. The average difference between actual and measured AC content is 0.17%.

Table 35 shows the test results for mixes 2 and 3 not included in the interlaboratory study. These results indicated that standard deviations for both mixes are within the average within-lab standard deviation of 0.099 estimated for all the other mixes as reported in Table 34.

Table 34 Within-lab and between-lab precision estimate for asphalt content for reflux

Mix #	**Actual AC, %**	**Measured AC, %**	**AC Diff, % (Actual - Measured)**	**Standard Deviation**		**Coefficient of Variation**		**Acceptable Range of Two Test Results**	
				Within-Lab	**Between-Lab**	**Within-Lab**	**Between-Lab**	**Within-Lab**	**Between-Lab**
1	5.8	5.62	0.18	0.078	0.078	1.4	1.4	0.218	0.218
4	5.6	5.54	0.06	0.114	0.114	2.1	2.1	0.319	0.319
5	5.7	5.59	0.11	0.109	0.109	2.0	2.0	0.307	0.307
6	5.6	5.42	0.18	0.071	0.266	1.3	4.9	0.198	0.745
7	5.4	5.07	0.33	0.077	0.120	1.5	2.4	0.216	0.337
8	5.5	5.34	0.16	0.143	0.184	2.7	3.5	0.401	0.516
Averages			**0.17**	**0.099**	**0.145**	**1.8**	**2.5**	**0.276**	**0.407**

Table 35 Reflux test results for mixes not included in the interlaboratory study

Lab #	**Mix**	**Asphalt Content**	**Test Results**			**Avg.**	**St. Dev.**
			1	**2**	**3**		
Lab 1A	2	5.9	5.74	5.74	5.81	5.76	0.04
	3	6.0	5.85	5.88	5.88	5.87	0.01

4.2.5 Data Analysis for Ignition Oven Test results without Correction Factors

Similar to the analysis conducted for the other tests, results were analyzed to determine any inconsistent data. The asphalt contents reported correspond to those given from the printouts of the ignition ovens. No correction factors (CFs) were applied to the asphalt content. CFs do not influence the within-laboratory variability, but CFs do affect the between-laboratory precision values. The magnitude of the correction factors is a function of the type of mix tested and the ignition unit used. Findings from NCHRP 9-56 indicate that even when the same oven brand was used to evaluate a particular mix, correction factors were different. This difference is even higher for high mass loss aggregates. Participants' laboratories were only provided with three samples to run ignition tests, but no raw material was provided to prepare calibration samples. Therefore, the analysis presented in this section corresponds to the uncorrected ignition test results and only within-laboratory variability was assessed. An additional analysis is presented in section 4.2.6 to illustrate how the CFs significantly impact test results. The between-lab variability is also estimated in section 4.2.6.

The data was analyzed separately for each mix. Tabular summary of data for mix 1 is presented in Table 36 as an example of the data. Tables for the remaining five mixes are included in Appendix B. Figure 17 presents the within-laboratory k statistics. The critical k value was not reached in any case.

Table 36 Interlaboratory test results for mix 1-ignition oven

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	k
			1	2	3				
Lab 1C	1	5.8	6.26	6.33	6.31	6.30	0.04	0.001	0.70
Lab 4C			5.93	5.93	6.06	5.97	0.08	0.006	1.47
Lab 5			6.5	6.46	6.52	6.49	0.03	0.001	0.60
			$\bar{\bar{x}}$ = average of labs averages					6.26	
			s_r = repeatability standard deviation					0.051	

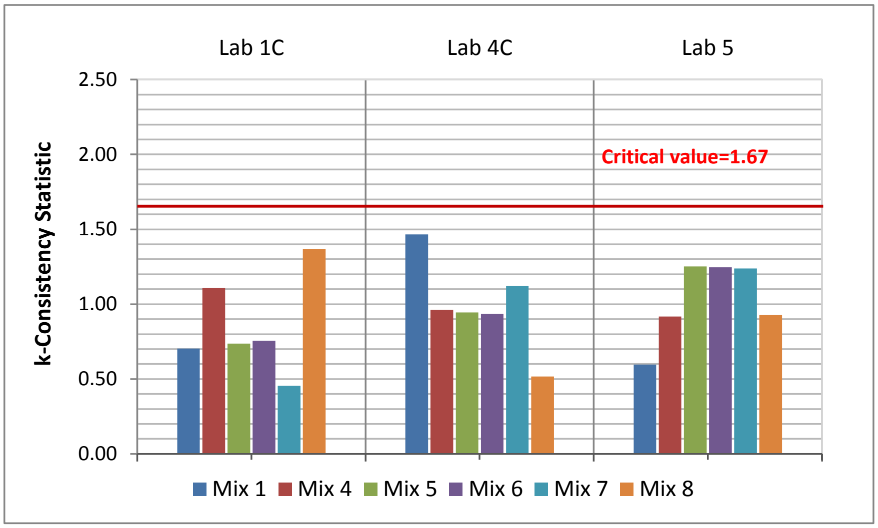

Figure 17 Consistency statistic k for asphalt content -ignition-all labs and materials

Table 32 summarizes the actual and average asphalt contents (without CFs), the asphalt content difference expressed as the difference of actual minus measured, standard deviations, coefficients of variation, and acceptable range of two test results for all the mixes. From this table it can be observed that the asphalt content difference (actual minus measured) is in average -0.46% indicating that aggregates used in this study have a relative high mass loss even at the reduced temperature of 800°F. Significantly higher CFs would be expected if tests were conducted at the higher temperatures currently recommended in the standard. For the Wisconsin aggregates evaluated, not accounting for proper CFs will yield significantly higher asphalt contents.

Table 37 presents the average within-lab precision statistics for all mixes. The within-lab standard deviation and acceptable range of two test results are higher than the precision statistics in AASHTO T 308: 0.069 and 0.196, respectively. As suggested in previous studies, the precision statement in the current standard was likely developed for low mass loss aggregate *(24).*

Table 37 Within-lab and for asphalt content for ignition oven (without CF)

Mix #	Actual AC, %	Measured AC, %	AC Diff, % (Actual-Measured)	Standard Deviation	Coefficient of Variation	Acceptable Range of Two Test Results
				Within-Lab	**Within-Lab**	**Within-Lab**
1	5.8	6.26	-0.46	0.051	0.8	0.143
4	5.6	6.22	-0.62	0.068	1.1	0.191
5	5.7	6.17	-0.47	0.095	1.5	0.267
6	5.6	5.92	-0.32	0.134	2.3	0.374
7	5.4	5.72	-0.32	0.104	1.8	0.291
8	5.5	6.10	-0.60	0.049	0.8	0.136
Averages			**-0.46**	**0.084**	**1.4**	**0.234**

Table 38 shows the test results for mixes 2 and 3 not included in the interlaboratory study. These results indicated that the standard deviations for both mixes are within the average within-lab standard deviation of 0.084 estimated for all the other mixes reported in Table 37.

Table 38 Ignition oven test results for mixes not included in the interlaboratory study

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.
			1	2	3		
Lab 1A	2	5.9	6.05	6.07	5.93	6.02	0.08
	3	6.0	6.42	6.44	6.32	6.39	0.06

4.2.6 Data Analysis for Ignition Oven Test results with Correction Factors

To estimate the between-lab variability of the ignition oven tests, two correction specimens were tested at NCAT for each mix. These calibration samples were prepared following the same procedure that was used to prepare samples for testing. That is, a total of five samples were tested, three replicates, plus two used to estimate CFs. The CF was calculated as the difference between the actual and average measured asphalt binder content (from calibration samples) expressed as a percentage of the asphalt mix mass. Since no calibration samples were available for the other two labs from Wisconsin, the asphalt content for calibration samples were randomly estimated using the mean and standard deviation of the three replicates tested using the excel function NORMINV. The function provides a random set of numbers normally distributed. With these numbers and actual asphalt content, correction factors were calculated. Although this is an arbitrary procedure, it can provide an estimate of the expected between-lab variability of the test procedure. The correction factors for each lab and mix are summarized in Table 39.

Table 39 Correction factors for asphalt content determination

Mix #	Lab 1C	Lab 4C[b]	Lab 5[b]
1	0.37	0.15	0.70
4	0.80	0.5	0.78
5	0.54	0.19	0.58
6	0.31	0.16	0.66
7	0.63	0.07	0.55
8	0.68	0.67	0.82

[b] estimated values

For each mix and laboratory, corrected asphalt contents were calculated by subtracting the CF from the measured asphalt content of these samples. Analysis was then conducted on the corrected asphalt contents per ASTM E 691 for each mix. A summary of data for mix 1 is presented in Table 40 as an example of the data. Results for the remaining five mixes are included in Appendix B.

The between-laboratory h statistics are presented in Figure 18. The critical h value was not reached in any case. The k statistics are not changed when corrected asphalt contents are used; therefore, results presented in Figure 17 remain the same.

Table 40 Interlaboratory test results for mix 1-ignition oven with correction factors

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 1C	1	5.8	5.89	5.96	5.94	5.93	0.04	0.001	0.08	1.13	0.70
Lab 4C			5.78	5.78	5.91	5.82	0.08	0.006	-0.03	-0.36	1.47
Lab 5			5.80	5.76	5.82	5.79	0.03	0.001	-0.06	-0.77	0.60

$\bar{\bar{x}}$ = average of labs averages 5.85

$s_{\bar{x}}$ = standard deviation of cell averages 0.072

s_r =repeatability standard deviation 0.051

s_L =between-lab standard deviation 0.065

s_R =reproducibility standard deviation 0.083

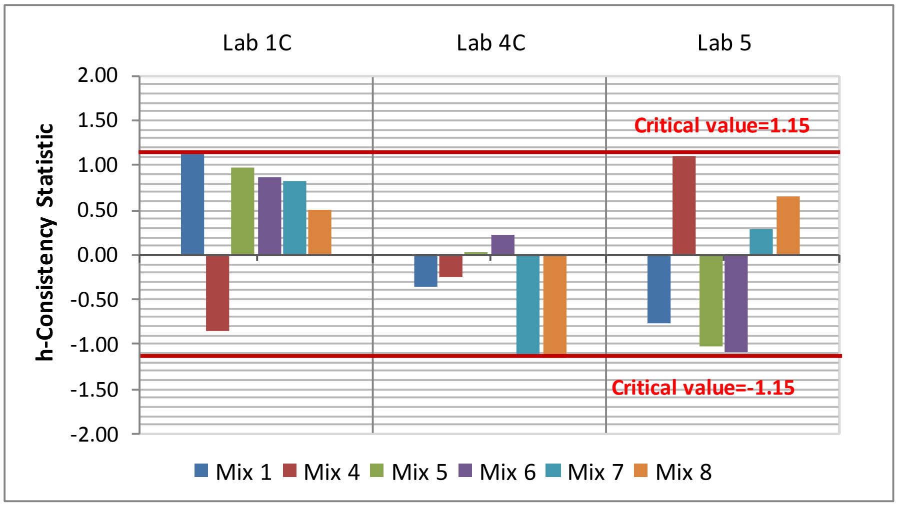

Figure 18 Consistency statistic k for asphalt content-ignition, all labs and materials

Table 41 summarizes the actual and measured asphalt contents (corrected), the asphalt content difference expressed as the difference of actual minus measured, standard deviations, coefficients of variation and acceptable range of two test results for each mix. As mentioned before, the within-lab statistics for each mix were not affected when CFs were applied. The within-lab standard deviation and acceptable range of two test results are slightly higher than the precision statistics in AASHTO T 308, 0.117 and 0.33, respectively.

It is important to emphasize that, in practice, the actual CFs must be determined for every JMF and ignition unit. As it was explained in section 2.3, the procedure to obtain a correction factor involves the preparation of two samples of the asphalt mixture at the design asphalt content. The mix calibration samples are tested and the calibration factor is calculated as the difference between the actual and measured asphalt binder contents expressed as a percentage of the asphalt mix mass. Nevertheless, the approach presented in this section illustrates the importance of applying CFs properly.

Table 41 Within-lab and between-lab precision estimate for asphalt content for ignition oven (with CF)

Mix #	Actual AC, %	Measured AC, %	AC Diff, % (Actual - Measured)	Standard Deviation		Coefficient of Variation		Acceptable Range of Two Test Results	
				Within -Lab	Between -Lab	Within -Lab	Between -Lab	Within -Lab	Between -Lab
1	5.8	5.85	-0.05	0.051	0.074	0.9	1.4	0.143	0.233
4	5.6	5.52	0.08	0.068	0.178	1.2	3.2	0.191	0.499
5	5.7	5.73	-0.03	0.095	0.109	1.7	1.9	0.267	0.306
6	5.6	5.54	0.06	0.134	0.213	2.4	3.8	0.374	0.597
7	5.4	5.25	0.15	0.104	0.108	2.0	2.1	0.291	0.303
8	5.5	5.40	0.10	0.049	0.084	0.9	1.8	0.136	0.236
Averages			**0.05**	**0.084**	**0.129**	**1.5**	**2.3**	**0.234**	**0.362**

4.3 Performance Grade Characteristics of Recovered Asphalt Binders

Asphalt binders that were extracted and recovered by centrifuge, reflux, or asphalt analyzer for asphalt content determination were used to determine PG grade characteristics. Upon completion of the extractions and recoveries, the recovered asphalt binder from each set of three recoveries was combined into a single container for testing. A small variation used for one lab was to combine the extracted materials and recovered them all at the same time. All the labs treated the recovered residue as un-aged binder.

The performance grades and continuous grades for each blend were determined as described in AASHTO M 320, “Standard Specification for Performance-Graded Asphalt Binder,” AASHTO R 29, “Standard Practice for Grading and Verifying the Performance Grade (PG) of an Asphalt Binder,” and ASTM D7643, “Standard Practice for Determining the Continuous Grading Temperature and Continuous Grades for PG Graded Asphalt Binders.” The recovered asphalt binders were short-term and long-term aged in the rolling thin film oven (RTFO) and pressure aging vessel (PAV) prior to testing. Samples were short term aged in the RTFO right after extraction and recovery prior to Dynamic Shear Rheometer Testing (DSR) to remove any solvent left in the samples. AASHTO T 350, “Standard Test Method for Multiple Stress Creep Recovery (MSCR) of Asphalt Binder using a DSR,” was performed to determine the non-recovered creep compliance (J_{nr}) and percent elastic recovery at 58°C. Table 42 shows the tests performed and the criteria used for determining continuous grades.

The base grade of the binders used in this evaluation corresponds to a PG 58-28. The continuous grade of this binder was obtained to give a better assessment of the effect of the recovery process and recycled binder content on the properties of the recovered binder. The continuous binder grade for the virgin binder was found to be PG 61.4-30.0. The intermediate continuous temperature was 18.1°C.

Table 42 Laboratory tests for asphalt binders

Test	Method	Output	Criteria
Dynamic Shear Rheometer	AASHTO T315	G*binder (kPa) and δ	RTFO Aged Binder: $G^*_{binder} / \sin(\delta_{binder}) \geq 2.20$ kPa PAV aged binder: $G_{*binder} \sin(\delta_{binder}) \leq 5{,}000$ kPa
Dynamic Shear Rheometer	AASHTO T350	J_{nr} (1/kPa) and % Recovery	n/a
Bending Beam Rheometer	AASHTO T313	S (MPa) and m-value	S ≤ 300 MPa m-value ≥ 0.300
Rolling Thin Film Oven Aging	AASHTO T240	Mass Change, %	Mass change ≤ 1.00%
Pressure Aging Vessel Aging	AASHTO R28	Aged asphalt binder for further testing	No criteria

Table 43 through Table 48 show the results of the binder tests conducted on the recovered binder from the different mixes. The percent mass loss after RTFO is reported first; in some instances, these values are higher than 1%, most likely due to leftover solvent after recovery, these percentages were particularly higher for binder from Lab 4. These tables also show the results of the high, intermediate and low temperature grades and limited MSCR parameters obtained for each lab. Finally, these tables also include averages, standard deviations and range of values for each property.

Table 43 and Table 44 show the results for the virgin mixes with no recycled materials corresponding to mixes 1 and 4. For these mixes the maximum standard deviation for the high PG continuous grade was 1.6°C and 0.6°C for the low PG continuous grade. The average high temperature continuous grades were 65.7 and 65.3°C for mixes 1 and 4, respectively. The average low temperature continuous grades were -29.6°C and -29.8°C.

Table 45 and Table 46 show the results for mixes with low and moderate amounts of recycled binder. Both of these mixes show similar PG grading results. The maximum standard deviation for the high PG continuous grade was 1.3°C and 0.5°C for the low PG continuous grade. The average high temperature continuous grades were 67.8 and 69.9°C for mixes 5 and 6, respectively. The average low temperature continuous grades were -29.0°C and -28.5°C for mixes 5 and 6.

Table 47 presents the results for mix 7 with high recycled binder content (RAP only). The results show that the maximum standard deviation for the high PG continuous grade was 1.3°C and 0.8°C for the low PG continuous grade. The average test results indicate a high continuous grade of 71.2°C and a low temperature continuous grade of -26.5°C.

Lastly, Table 48 summarizes the results for mix 8, the mix with high recycled binder content containing both RAP and RAS. The standard deviation for the high PG continuous grade was 0.8°C and 0.3°C for the low PG continuous grade. The average high continuous grade was 77.8 °C and the low temperature grade -24.1°C.

The continuous grading properties for the recovered binders are compared in Figure 19 through Figure 21 for the high, intermediate and low temperature, respectively. In each figure, the red line shows the continuous grade at that temperature level for the virgin binder. These figures indicate consistent results for the different labs.

The results presented in Table 43 through Table 48 suggest that the extraction method and type of solvent does not have a significant effect on the PG grade properties of the recovered binders.

Table 43 PG characteristics of recovered binders for virgin mix 1

Lab #	**Mix #**	**Extraction / Solvent**	**Mass Loss, %**	**PG Continuous Grade**					**MSCR Parameters**	
				High	**Inter.**	**Stiffness Low**	**m-value Low**	**Low**	**Jnr at 3.2 kPa @ 58°C**	**%Rec at 3.2 kPa @ 58°C**
Lab 2	Mix #1	Centrifuge/ nPB	1.7	64.3	18.1	-30.0	-30.1	-30.0	1.81	1.9
Lab 3A		Centrifuge/ Toluene	0.9	66.4	19.4	-29.5	-29.9	-29.5	1.40	3.9
Lab 3B		Analyzer/ TCE	1.0	67.6	20.2	-29.9	-29.0	-29.0	1.11	6.0
Lab 4		Analyzer/ TCE	4.8	64.6	19.3	-31.2	-29.8	-29.8	1.59	2.9
		Average=	2.1	65.7	19.3	-30.2	-29.7	-29.6	148	3.7
		Std. Dev=	1.9	1.6	0.9	0.7	0.5	0.4	0.30	7.8
		Max=	4.8	67.6	20.2	-29.5	-29.0	-29.0	1.81	6.0
		Min=	0.9	64.3	18.1	-31.2	-30.1	-30.0	1.11	1.9
		Range=	3.9	3.3	2.1	1.7	1.1	1.0	0.70	4.1

Table 44 PG characteristics of recovered binders for virgin mix 4

Lab #	Mix #	Extraction / Solvent	Mass Loss, %	PG Continuous Grade					MSCR Parameters	
				High	Inter.	Stiffness Low	m-value Low	Low	Jnr at 3.2 kPa @ 58°C	%Rec at 3.2 kPa @ 58°C
Lab 1A	Mix #4	Centrifuge/ TCE	0.7	65.2	18.2	-28.0	-28.0	-28.0	1.56	3.6
Lab 1B		Reflux/ TCE	0.8	65.3	16.8	-32.5	-32.3	-32.3	1.59	3.7
Lab 2		Centrifuge/ nPB	2.0	65.1	18.7	-29.9	-29.7	-29.7	1.62	2.3
Lab 3A		Centrifuge/ Toluene	0.9	65.9	18.5	-29.6	-30.5	-29.6	1.55	3.3
Lab3 B		Analyzer/ TCE	0.9	67.3	18.0	-29.9	-29.6	-29.6	1.17	5.7
Lab 4B		Analyzer/ TCE	4.2	64.8	19.5	-30.9	-29.6	-29.6	1.71	2.9
Average=			1.6	65.6	18.3	-30.1	-30.0	-29.8	1.53	3.6
Std. Dev=			1.4	0.9	0.9	1.5	1.4	1.4	0.19	1.2
Max=			4.2	67.3	19.5	-28.0	-28.0	-28.0	1.71	5.7
Min=			0.7	64.8	16.8	-32.5	-32.3	-32.3	1.17	2.3
Range=			3.5	2.5	2.7	4.5	4.3	4.3	0.54	3.4

Table 45 PG characteristics of recovered binders for recycled mix 5

Lab #	Mix #	Extraction / Solvent	Mass Loss, %	PG Continuous Grade					MSCR Parameters	
				High	Inter.	Stiffness Low	m-value Low	Low	Jnr at 3.2 kPa @ 58°C	%Rec at 3.2 kPa @ 58°C
Lab 1A	Mix #5	Centrifuge/ TCE	0.7	68.6	18.6	-29.0	-28.8	-28.8	1.05	5.6
Lab 1B		Reflux/ TCE	0.6	69.0	18.7	-29.9	-29.2	-29.2	1.09	5.6
Lab 2		Centrifuge/ nPB	1.6	66.5	19.3	-29.1	-28.7	-28.7	1.29	3.5
Lab 3A		Centrifuge/ Toluene	0.8	67.7	21.0	-29.2	-29.3	-29.2	1.10	5.4
Lab3 B		Analyzer/ TCE	0.7	67.9	20.5	-29.4	-29.3	-29.3	1.06	5.8
Lab 4		Analyzer/ TCE	5.8	67.2	21.7	-30.3	-29.0	-29.0	1.20	5.0
Average=			1.7	67.8	20.0	-29.5	-29.1	-29.0	1.13	5.1
Std. Dev=			2.0	0.9	1.3	0.5	0.3	0.2	0.09	0.8
Max=			5.8	69.0	21.7	-29.0	-28.7	-28.7	1.29	5.8
Min=			0.6	66.5	18.6	-30.3	-29.3	-29.3	1.05	3.5
Range=			5.2	2.5	3.1	1.3	0.6	0.6	0.24	2.3

Table 46 PG characteristics of recovered binders for recycled mix 6

Lab #	Mix #	Extraction / Solvent	Mass Loss, %	PG Continuous Grade					MSCR Parameters	
				High	Inter.	Stiffness Low	m-value Low	Low	Jnr at 3.2 kPa @ 58°C	%Rec at 3.2 kPa @ 58°C
Lab 1A	Mix 6	Centrifuge/ TCE	0.6	69.6	19.3	-29.5	-28.8	-28.8	0.88	7.5
Lab 1B		Reflux/ TCE	0.5	69.8	19.6	-29.5	-28.0	-28.0	0.89	7.5
Lab 2		Centrifuge/ nPB	1.6	67.4	19.4	-29.0	-28.1	-28.1	1.16	3.9
Lab 3A		Centrifuge/ Toluene	0.7	69.1	20.6	-28.8	-29.1	-28.8	0.87	7.2
Lab 3B		Analyzer/ TCE	1.0	69.9	20.6	-29.0	-28.5	-28.5	0.80	8.7
Lab 4		Analyzer/ TCE	6.4	66.9	21.8	-30.7	-29.0	-29.0	1.03	4.7
Average=			1.8	68.8	20.2	-29.4	-28.6	-28.5	0.94	6.6
Std. Dev=			2.3	1.3	1.0	0.7	0.5	0.4	0.13	1.9
Max=			6.4	69.9	21.8	-28.8	-28.0	-28.0	1.16	8.7
Min=			0.5	66.9	19.3	-30.7	-29.1	-29.0	0.80	3.9
Range=			5.9	3.0	2.5	1.9	1.1	1.0	0.35	4.8

Table 47 PG characteristics of recovered binders for recycled mix 7

Lab #	Mix #	Extraction / Solvent	Mass Loss, %	PG Continuous Grade					MSCR Parameters	
				High	Inter.	Stiffness Low	m-value Low	Low	Jnr at 3.2 kPa @ 58°C	%Rec at 3.2 kPa @ 58°C
Lab 1A	Mix 7	Centrifuge/ TCE	0.7	68.9	24.3	-29.5	-26.0	-26.0	0.62	10.2
Lab 1B		Reflux/ TCE	0.8	71.1	21.5	-28.1	-27.2	-27.2	0.62	10.0
Lab 2		Centrifuge/ nPB	2.0	72.4	22.6	-27.5	-25.7	-25.7	0.39	16.5
Lab 3A		Centrifuge/ Toluene	0.7	71.5	19.5	-28.6	-26.7	-26.7	0.57	13.5
Lab 3B		Analyzer/ TCE	0.9	72.3	22.4	-28.2	-27.3	-27.3	0.59	11.3
Lab 4		Analyzer/ TCE	5.0	71.1	24.3	-29.5	-26.0	-26.0	0.62	10.2
Average=			1.7	71.2	22.4	-28.6	-26.5	-26.5	0.57	12.0
Std. Dev=			1.7	1.3	1.8	0.8	0.7	0.7	0.09	2.6
Max=			5.1	72.4	24.3	-27.5	-25.7	-25.7	0.62	16.5
Min=			0.7	68.9	19.5	-29.5	-27.3	-27.3	0.39	10.0
Range=			4.4	3.5	4.8	2.0	1.6	1.6	0.24	6.5

Table 48 PG characteristics of recovered binders for recycled mix 8

Lab #	Mix #	Extraction / Solvent	Mass Loss, %	PG Continuous Grade					MSCR Parameters	
				High	Inter.	Stiffness Low	m-value Low	Low	Jnr at 3.2 kPa @ 58°C	%Rec at 3.2 kPa @ 58°C
Lab 1A	Mix 8	Centrifuge/ TCE	0.5	79.2	19.1	-27.9	-24.6	-24.6	0.17	33.9
Lab 1B		Reflux/ TCE	0.5	77.1	23.3	-28.5	-23.6	-23.6	0.23	29.7
Lab 2		Centrifuge/ nPB	1.4	77.5	23.5	-28.0	-24.2	-24.2	0.19	27.8
Lab 3A		Centrifuge/ Toluene	0.8	78.3	23.7	-27.5	-24.7	-24.7	0.17	31.6
Lab 3B		Analyzer/ TCE	1.0	77.9	24.0	-28.0	-24.3	-24.3	0.18	30.0
Lab 4		Analyzer/ TCE	8.4	76.9	26.0	-28.1	-23.3	-23.3	0.17	30.0
Average=			2.1	77.8	23.3	-28.0	-24.1	-24.1	0.18	30.5
Std. Dev=			3.1	0.8	2.3	0.3	0.6	0.6	0.02	2.1
Max=			8.4	79.2	26.0	-27.5	-23.3	-23.3	0.23	33.9
Min=			0.5	76.9	19.1	-28.5	-24.7	-24.7	0.17	27.8
Range=			8.0	2.3	6.9	1.0	1.4	1.4	0.06	6.1

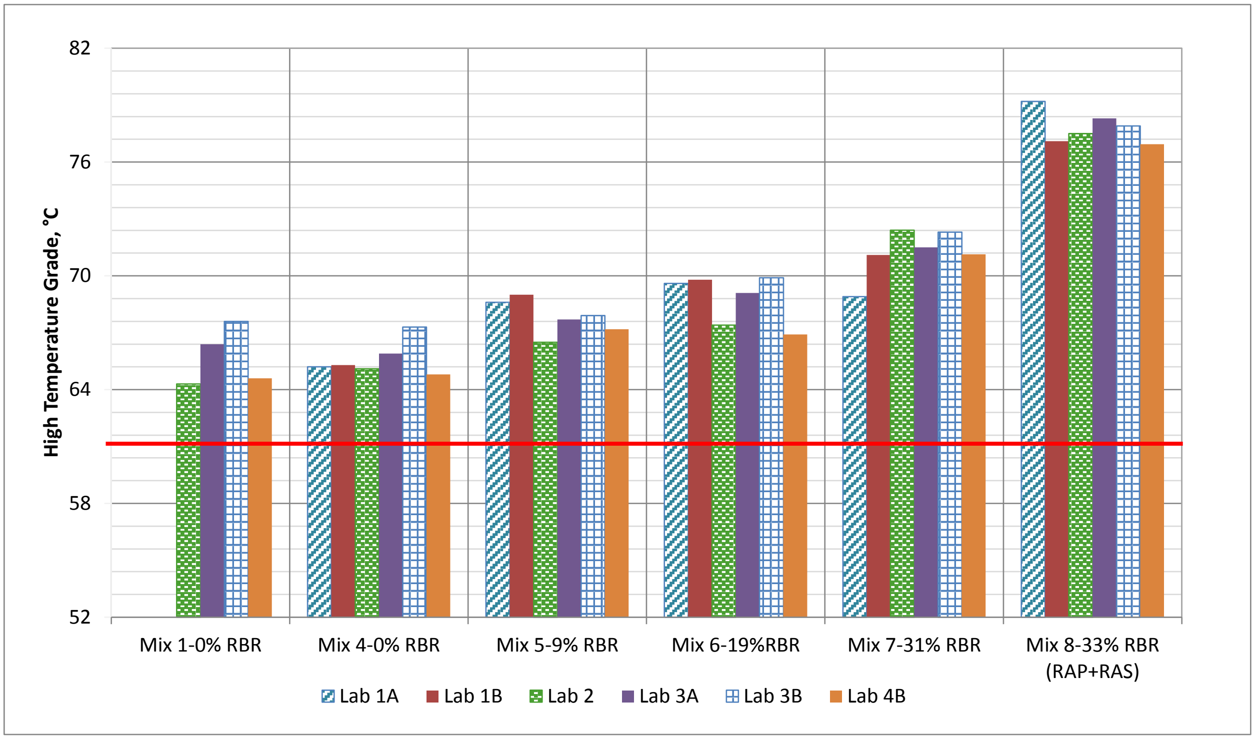

Figure 19 Comparison of recovered binder high temperature continuous grade

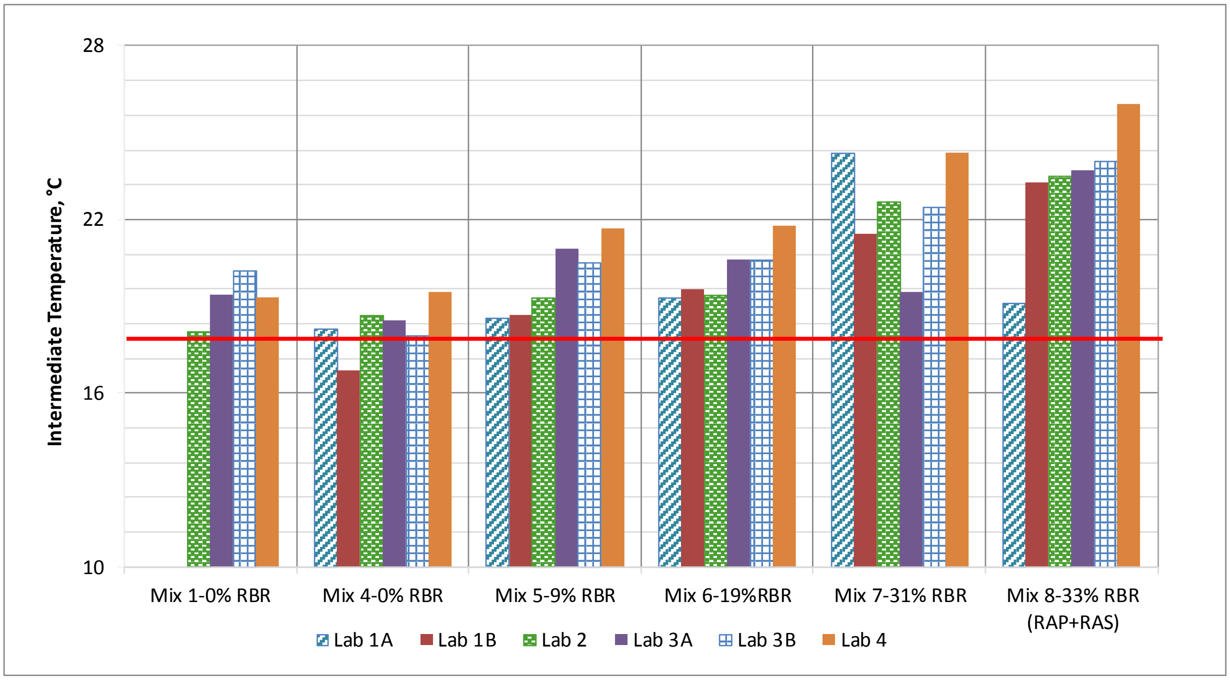

Figure 20 Comparison of recovered binder intermediate temperature continuous grade

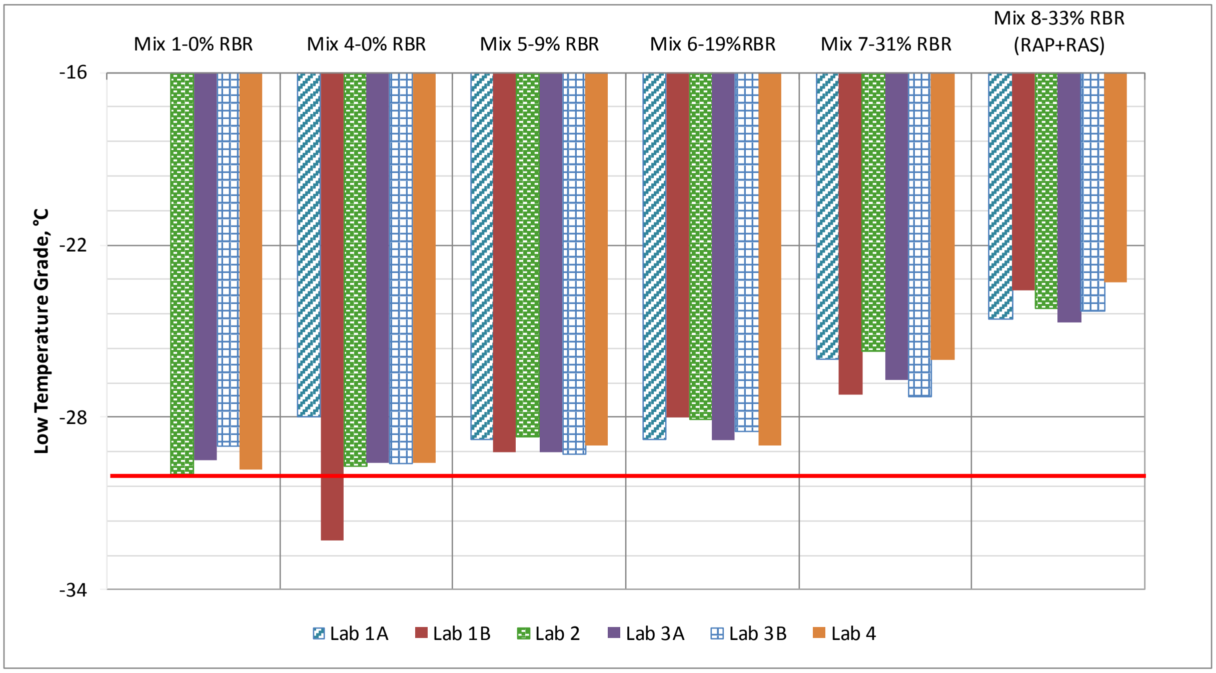

Figure 21 Comparison of recovered binder low temperature continuous grade

Table 49 presents a summary of the PG characteristics of the recovered binders for each mix per AASHTO M 320. The average results for the mixes with no or low percentage of recycled materials (Mixes 1, 4, 5 and 6) indicate that the recovered binder will be classified as a PG 64-28. These results indicate an increase in the high temperature grade of the binder by one grade;

the low temperature was not affected. The results for mix 7 with the high recycled binder (RAP only) shows that the binder classifies as a PG 70-22, indicating that the higher recycled binder content increased the high temperature grade of the binder by two grades. The low temperature grade decreased one grade. For mix 8 with a high recycled binder content from a RAP and RAS combination, the recovered binder is classified as a PG 76-22, which is three grades higher for the high temperature grade and one grade lower for the low temperature grade. These results indicate that although the amount of recycled binders for mixes 7 and 8 are similar, the RAP and RAS combination change the grade of a binder differently yielding a more drastic increment in the high PG grade of the binder. These results are in agreement with previous research conducted by Bonaquist in 2011 as part of project WHRP # 0092-10-06 (*33*).

All of the recovered asphalt binders had intermediate temperature continuous grade values that were slightly above 19°C. If testing for a specification that requires a PG 58-28 binder grade according to AASHTO M 320, these asphalt binders (with the exception of mix 4) will not meet the low temperature requirement due to failing intermediate temperature results.

Table 49 Summary PG characteristics of recovered binders

Mix #	RBR	High PG Temp	Intermediate Temp. Grade	Low PG Temp	Continuous PG Grade	PG Grade
1	0% RBR	65.7	19.3	-29.6	65.7 -29.6	64-28
4	0% RBR	65.6	18.3	-29.8	65.6 -29.8	64-28
5	9% RBR	67.8	20.0	-29.0	67.8 -29.0	64-28
6	19%RBR	68.8	20.2	-28.5	68.8 -28.5	64-28
7	31% RBR	71.2	22.4	-26.5	71.2 -26.5	70-22
8	33% RBR (RAP+RAS)	77.8	23.3	-23.3	77.8 -23.3	76-22

A limited evaluation was conducted for the MSCR test results. Participant labs reported MSCR parameters J_{nr} and percent recovery at 3.2 kPa measured at 58°C. A binder with a high value of recovery and low J_{nr} may yield less permanent deformation. The percent recovery parameter provides an indication of the delayed elastic response of the asphalt binder with a high delayed elastic response indicating that the asphalt binder has a significant elastic component at the test temperature.

Table 43 through Table 48 present the MSCR results for the recovered binders for each mix. Figure 22 and Figure 23 compare the results of J_{nr} and percentage recovery for each mix and laboratory. Based on the results presented in Figure 22, adding RAP decreased the non-recoverable creep compliance (J_{nr}) value, which suggests a better rutting resistance.

According to the Combined State Binder Group's (CSBG) 2017 *Method of Acceptance for Asphalt Binder* based on the J_{nr} at 3.2 kPa all recovered binders for mixes 1, 4, and 5 are "H" grade binders, the binder recovered for mixes 6 and 7 are "V" grade binders, and the binder recovered for mix 8 is classified as “E” grade *(34)*. There is no requirement for percent recovery for standard traffic “S” binders, for heavy traffic “H, V and E” the percent recovery must be a minimum of 30, 55, or 75% respectively. The results presented in Figure 23 showed that the recovered binders for mixes 1, 4, 5, 6 and 7 with virgin binders and low to moderate recycled binder content have average percentage recovery of less than 12% and recovered binder from mix 8 shows an average percentage recovery of 30.5%. These results indicated that the higher percent recovery for mix 8 can be attributed to the RAP and RAS combination. None of the recovered binders met the percent recovery requirement for their respective grade. This is not unexpected, as the virgin PG 58-28 met the requirements for an “S” grade binder and had a low percent recovery of 1.2%.

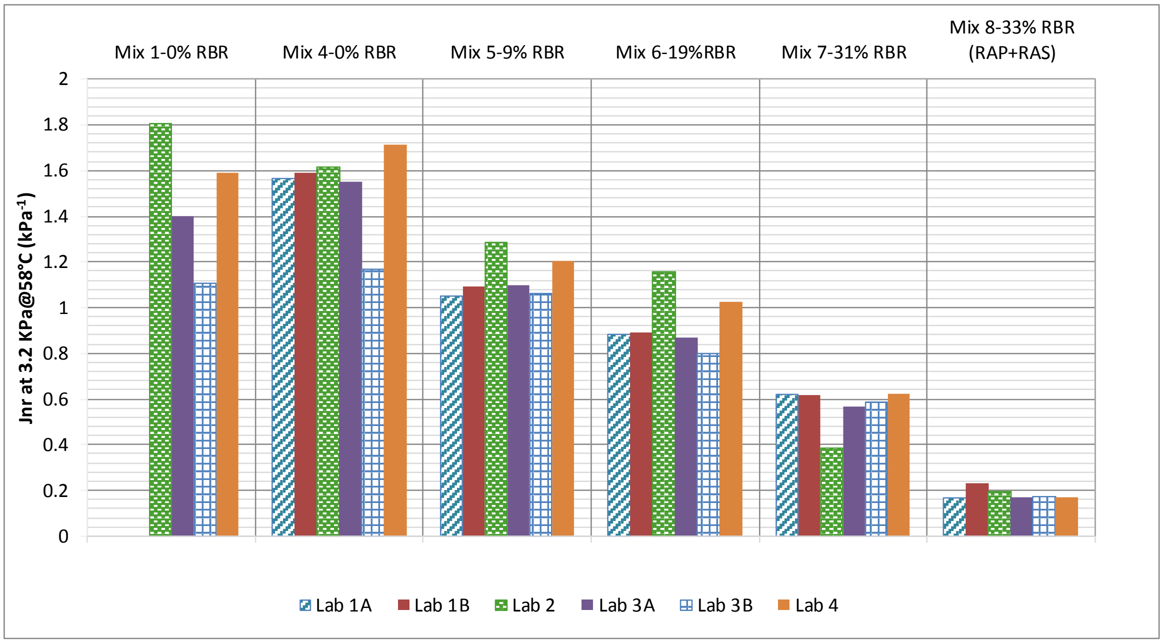

Figure 22 J_{nr} at 3.2 kPa for recovered binders at 58°C

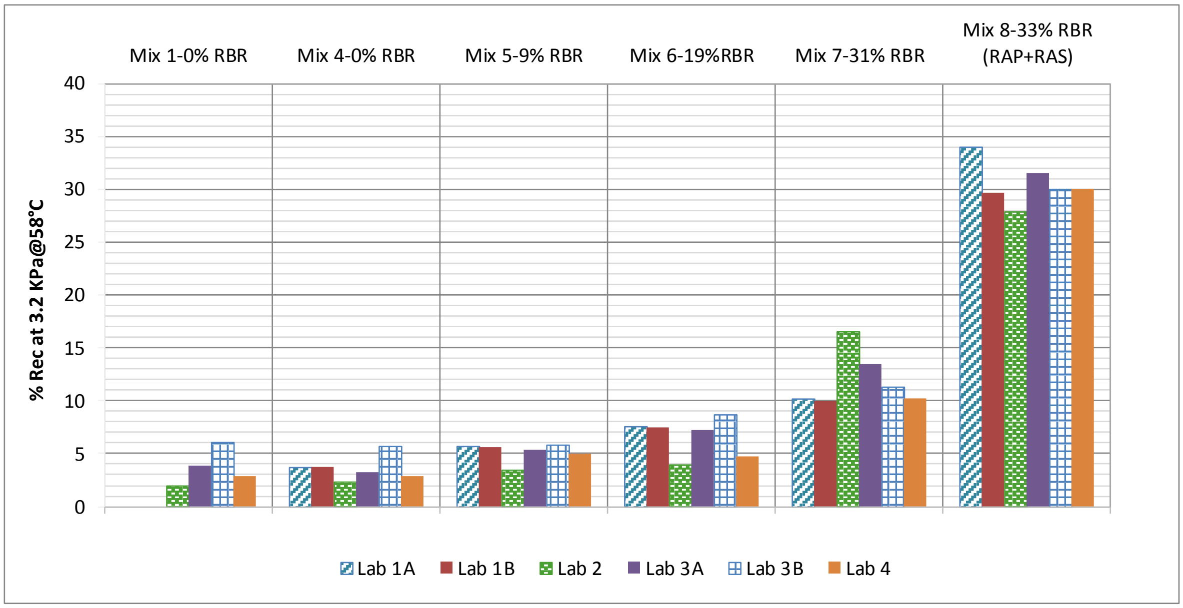

Figure 23 Percentage recovery at 3.2 kPa for recovered binders at 58°C

5. CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Project WHRP 0092-16-02 evaluated several test procedures to quantify the amount of asphalt in asphalt mixtures. Although there are different well-established AASHTO procedures to determine the asphalt content of mixes, the accuracy of these test procedures may be sensitive to local materials. In addition, as the use of high recycled binder content mixes increases, there is a need to evaluate within-lab and between-lab variability. To accomplish this, several laboratories from Wisconsin along with the National Center for Asphalt Technology (NCAT) lab participated in the study. A second goal for this project was to evaluate the variability of the PG properties of the extracted binder after recovery.

The experimental plan included the evaluation of AASHTO T 164 method A (centrifuge extraction) and method B (reflux extraction), asphalt analyzer, and AASHTO T 308. A total of eight mixes that included virgin mixes and mixes with various contents of recycled binder were evaluated to quantify the within-lab and between-lab variability of the measured asphalt content for the various test methods. Two sources of recycled asphalt pavement (RAP) and one source of recycled asphalt shingles (RAS) were also evaluated. All of the materials evaluated represent commonly used materials in Wisconsin. The results of this study are presented below.

Within-lab and between-lab variability for each test procedure was developed for Wisconsin materials and then compared to the current AASHTO standards (when available). Table 50 summarizes the within-lab and between-lab precision estimates for the different test procedures evaluated in this study. Precision statements for AASHTO T 164 and AASHTO T 308 are also included for comparison.

Table 50 Within-lab and between-lab precision estimate for different test procedures

Tet Procedure	**AC Diff., % (Actual - Measured)**	**Standard Deviation**		**Acceptable Range of Two Test Results**	
		Within-Lab	**Between-Lab**	**Within-Lab**	**Between-Lab**
Centrifuge	0.21	0.071	0.120	0.200	0.335
Asphalt Analyzer	0.14	0.067	0.097	0.187	0.271
Reflux	0.17	0.099	0.145	0.276	0.407
Ignition	0.05	0.084	0.129	0.234	0.362
AASHTO T 164		**0.18**	**0.29**	**0.52**	**0.81**
AASHTO T 308		**0.069**	**0.117**	**0.196**	**0.33**

Although the evaluation included a limited number of laboratories it was possible to assess the variability of each test procedure as follows.

Centrifuge Extraction

- Within-lab and between-lab variability for asphalt content does not seem to be affected by the use of recycled mixes. Individual test results by mix type for moderate and high recycled mixes were similar to the results for virgin and low recycled mixes.
- The average difference between the actual and measured asphalt content for all mixes was found to be equal to 0.21%. This indicates that solvent extraction is not able to remove all of the binder from the mix. For moderate and high recycled mixes, the average was higher than that for virgin and low recycled mixes. For example, Mixes 6 and 7 had differences of 0.36% and 0.27%, respectively.
- Standard deviations and acceptable range of two test results found for this test method are within the current precision statement of AASHTO T 164.
- AASHTO T 164 includes one precision statement regardless of the extraction method used (e.g centrifuge, reflux). Within-lab and between-lab variability for this test method was found to be lower than the precision statistic included in AASHTO T 164. This suggests than the current precision estimate in the AASHTO procedure might be adjusted, but it is recommended that no changes be made since the number of labs participating was small.

Asphalt Analyzer

- The asphalt analyzer has the lowest standard deviations of all test procedures evaluated for tests conducted within a lab and between-labs.
- Similar to the results from centrifuge, within-lab and between-lab variability for asphalt content does not seem to be affected by the use of recycled mixes. Test results by mix type for moderate and high recycled mixes were similar to the results for virgin and low recycled mixes with the exception of Mix 8, which showed a relative high variability compared to that for virgin and low recycled mixtures.
- The average difference between the actual and measured asphalt content was 0.17%. Once again, solvent may not be able to remove all of the binder from the mix. For moderate and high recycled mixes (6 and 7), the differences were higher at 0.25% and 0.30%, respectively.
- Although there is currently no precision statement for this test method, the within-lab and between-lab variability developed in this study was found to be within the current precision statement of AASHTO T 164 and similar to the variability for centrifuge testing. Hence, the precision information in AASHTO T 164 should be sufficient for the asphalt analyzer.

Reflux

- Tests conducted with this test procedure take a significant amount of time to conduct, particularly for recycled mixes. Also, this test procedure does not seem to be widely used.
- Within-lab and between-lab variability for this test procedure were developed with results from only two labs. The results show higher variability than the centrifuge and asphalt analyzer but are still within the AASHTO T 164 precision statement. Hence, the same precision as provided in AASHTO T 164 can be used. The average difference between the actual and measured asphalt content was 0.17%.

Ignition

- With the exception of aggregate 2 used in Mix 2 (tested at NCAT only), the aggregates used in this study had a relatively high mass loss even at the reduced temperature of 800°F. Higher mass loss translates into higher correction factors (CFs). Conducting the tests at higher temperatures would yield even higher CFs and more variability in the test results.
- Using the corrected asphalt content test results, standard deviations and acceptable range of two test results found for this test were slightly higher than the ones currently specified in the test procedure. This may be attributed to the aggregates used in this study, which in general showed high mass loss even at the reduced temperature of 800°F.
- Within-lab and between-lab variability for individual test results by mix type for few of the recycled mixes were higher than those for virgin and low recycled mixes.
- The average difference between the actual and measured asphalt content for all mixes was found to be equal to 0.05% (with CFs). Although some of the values used in this analysis were estimated as explained in section 4.2.6, the analysis indicates that if correction factors are properly applied, ignition tests can yield measured asphalt contents closer to the actual asphalt contents.
- For ignition tests conducted on RAS material only, it was observed that at 800°F, there was some unburned asphalt left in the residue. Tests conducted at 900°F didn't show any signs of unburned asphalt. Hence, it is recommended that for testing RAS, the test temperature be set at 900°F.

PG Characteristics of Recovered Asphalt Binders

- Continuous grading properties for the recovered binders for virgin and recycled mixes were found to be similar for the different labs with the exception of lab 4, which indicated lower values for high continuous grades for all mixes.
- For the materials evaluated in this study, extraction method and type of solvent used does not have a significant effect on the PG properties of recovered binders.

- Regarding PG characteristics of the recovered binders, more recycled binder leads to stiffer materials. The results for the mixes with no or low percentages of recycled materials (Mix 1, 4, 5, and 6) showed that the recovered binder was a PG 64-28. Prior to mixing, the base PG grade was a PG 58-28. This indicates an increase in the high temperature grade of the binder one grade; the low temperature was not affected. The results for Mix 7 with the high recycled binder (RAP only) show that the binder classifies as a PG 70-22, indicating that the higher recycled binder content increased the high temperature grade two grades; the low temperature grade decreased one grade. For Mix 8 with high recycled binder content from a RAP and RAS combination, the recovered binder classifies as a PG 76-22, which is three grades higher for the high temperature grade and one grade lower for the low temperature grade. This indicates that although the amount of recycled binders for Mixes 7 and 8 are similar, the RAP and RAS combination change the grade of a binder differently, yielding a higher increment in the high PG grade of the binder.
- The recovered asphalt binders, except binder from Mix 4, did not meet the intermediate temperature requirement according to AASHTO M 320.
- Multiple stress creep recovery (MSCR) test results showed that all recovered binders met J_{nr} requirement as follows: Mixes 1, 4, and 5, heavy traffic “H”, Mixes 6 and 7 very heavy traffic “V”, and the binder recovered for Mix 8, extremely heavy traffic “V”. However, none of the recovered binders met the percent recovery requirement for their respective grade.

5.2 Recommendations

A number of appropriate recommendations are provided below based on the tests and analysis completed in this project.

- It is recommended that, in addition to AASHTO T 164 by centrifuge, the WisDOT specifications should allow the ignition test and the asphalt analyzer to be used. Section 460.2.6 for recovered asphaltic binders in the current specifications only allows for AASHTO T 164. The asphalt analyzer results showed the lowest standard deviations of all test procedures and similar to the results obtained with centrifuge. Results also showed that when correction factors are properly applied, ignition test can yield accurate asphalt content results.
- If the ignition method is incorporated into WisDOT specifications, it is recommended to conduct tests at 800°F for all mixes instead of 1000°F currently recommended in AASHTO T 308. The results showed that most of the aggregates used in this study had a high mass loss, tests conducted at 800°F have proved to be effective in reducing the variability in measured asphalt content since the lower temperature reduced the asphalt correction factors for asphalt mixes study.

- For the ignition test conducted on RAS material only, it is recommended to conduct the test for RAS at 900°F. When tests were conducted at 800°F there was some unburned asphalt left in the residue that was not observed when tests were conducted at 900°F.
- JMF limits currently included in WisDOT specifications section 460.2.8.2.1.5 can potentially be revised and adjusted as needed using the precision estimates for the different test procedures developed in this project. To accomplish this, it is important to consider the following:
  - All of the solvent extraction methods underestimated the actual asphalt content by approximately 0.2%. The job mix formula (JMF) limit for asphalt content is currently set at -0.3% with a warning limit of -0.2%. This indicates that if any of these solvent extraction methods are used, the asphalt content will be in the warning limit for most mixes, allowing very small error due to test procedures variability. When high recycled content mixes were evaluated, the measured asphalt content was found to be more than 0.3% lower than the actual asphalt content. Therefore, the tolerances may need to be increased when high recycled content mixes are used or a CF of 0.2% could be applied to the measured asphalt content based on the results of this study.
  - For the ignition method, the actual asphalt content was underestimated by approximately 0.1%. If the ignition oven method is used and CFs are properly applied, the current JMF limit for asphalt content may be adequate.
- A draft language for WisDOT specification sections 4.2.6 and 460.2.8.2.1.5 was included as Appendix C to reflect potential recommendations for asphalt content determination and control limits related to asphalt content for quality control (QC) based on the results of this study.
- Although this study did not include any polymer modified binders, information presented in the literature review indicates that some solvents such as nPB may have compatibility issues with polymer modified binders due to the type of stabilizer used with those solvents. Therefore, it is recommended that if a different solvent is used, the results should be compared to those typically obtained with their traditional solvents.

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APPENDIX A

Notes of Phone Interviews and/or Email Communication

Illinois DOT – James Trepanier

- Does not use AC as a pay item: air voids and VMA
- Contractor takes random location sample behind the paver and DOT tests for AC and do gravities for PFP program, test one out of four sublots (as long as they compare we use DOT test results for acceptance)
- Districts are first level of testing (mainly use ignition oven) – a few dolomite sources within state (find alternate means by either blue box nuke gauge or solvent extraction)
- Do round-robin testing similar to AMRL for all state labs annually (usually a minimum of 2 private labs per district)
- Only do round robin on ignition oven testing (AMRL specs if they get a rating of 2 or less then they have to do a root cause analysis)
- Use centrifuge extraction for some dispute resolution (use TCE)
- Solvent extraction for RAS, for RAP use ignition oven that is corrected through solvent extraction
- Do not run PG on any extracted material
- If you use ABR over 20% must bump high and low
- Concerns about blending of stiffer binders
- Concerns: They are happy with the system that they have. They are confident in AC because they run G_{sb} and issue those to contractors.

Iowa – Scott Schram

- Iowa pays for oil separately. Paid off tank stick or calibrated flow meter.
- Quality characteristics – lab voids, film thickness, field voids
- For recycled materials – state runs AC tests – chemical extraction (Centrifuge) – n-propyl bromide – TCE was getting expensive and disposal was difficult, recovery unit for recycling
- Nothing contractually PG testing
- Never recover and then grade
- Take virgin sample from line for PG grading – one tin sample per day, run one per week
- 7 DSRs (once a month run a round robin on unaged material), no RTFOs
- Concerns: Pay off one binder content off the entire day, know the average, do not know the average because they are not running the extractions multiple times
- The extraction on shingles – account for 2/3 of binder as effective
- They break the shingles into components (average recipe for RAS)

Michigan-John Barak

- Michigan determines asphalt content in asphalt mixtures by ignition test or vacuum extraction, method 1 and method 2, respectively. Both methods are used at about the same frequency.
    - Method 1. Asphalt binder content based on ignition method (MTM 319). If method 1 is selected, the burning temperature is established at the pre-production meeting. The contractor provides a laboratory mixture sample to the QA acceptance laboratory to establish the correction factor for each mix.
    - Method 2. Asphalt binder content based on vacuum extraction by (MTM 325).
    - The determination of which method to use for each mix is made by the contractor at the pre-production meeting. The method selected cannot be changed during mix production without submitting a new mix design to the MDOT construction field services HMA laboratory for verification.
- For RAP and RAS materials either method can be used.
- During production, random mixture samples are obtained by MDOT approximately once every 1000. These samples, which are taken behind the paver, are tested using either method.
- MDOT uses asphalt content as a pay factor but not as a pay item.

Minnesota – John Garrity and Dave Linell

- Minnesota recently took AC as a pay item
- AFT instead of VMA
- Add oil to total oil ratio
- Determine AC during paving process
- Use ignition oven in QC process
- Use chemical extraction to develop correction factors
- 3 or 4 years ago – had issues matching AC to contractor
- Both state and DOT correct to same AC – sample taken on first day of production (run chemical extraction by state) –alternatives like n-propyl bromide and d-limonene are suitable replacements for
- PG grading is never a control – never done unless its specifically requested
- Require asphalt samples between pump and burner
- Concerns: Don't fully trust IO results, but correction gives them more confidence
- IO – windy days and cold days influence the IO results
- Correction factors are oven dependent
- Oven tolerance is 0.3% from oven to oven (used to be 0.2%)
- Older ovens breaking down some of the aggregate

Wisconsin-Erik Lyngdal

- WisDOT does not require testing for asphalt content. Contractors are required to report asphalt content by calculation, nuclear gauge reading and/or by inventory. The calculation method is specified in WisDOT Construction Materials Manual (CMM) section 8-36 using equation A1. “By inventory” option is based on plant gauge readings of the asphalt mix producer. Readings may involve a calibration procedure and use of correction/correlation factor.

$$P_b = 100 * \left(\frac{G_b}{G_{mm}}\right) * \left(\frac{G_{se} - G_{mm}}{G_{se} - G_b}\right) \qquad \text{Equation A1}$$

Where:

G_{mm}= current sample test result;
G_{se}= previous day; and
G_b= mix design.

- WisDOT requires RAP or RAS asphalt contents to be determined per AASHTO T164. Verification of RAP/RAS asphalt contents is at the discretion of the department. Currently, there is no explicit criterion for acceptance of a mixture design via RAP/RAS asphalt content verification.
- Asphalt content is used as pay item. There is a disincentive pay adjustment for asphalt contents that exceed specification limits during production.

APPENDIX B

Interlaboratory Test Results Analyses Tables per ASTM E 691

ASPHALT ANALYZER ADDITIONAL TEST RESULTS

B1. Interlaboratory test results for Mix 1-asphalt analyzer

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 4B	1	5.8	5.65	5.69	5.61	5.65	0.04	0.002	-0.09	-1.03	0.81
Lab 3B			5.86	5.86	5.74	5.82	0.07	0.005	0.08	0.97	1.40
Lab 6			5.77	5.71	5.75	5.74	0.03	0.001	0.01	0.07	0.62

$\bar{\bar{x}}$ = average of labs averages 5.74

$s_{\bar{x}}$ = standard deviation of cell averages 0.085

s_r = repeatability standard deviation 0.049

s_L = between-lab standard deviation 0.080

s_R = reproducibility standard deviation 0.094

Table B2. Interlaboratory test results for mix 4-asphalt analyzer

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 4B	4	5.6	5.55	5.5	5.45	5.50	0.05	0.002	-0.01	-0.35	0.77
Lab 3B			5.46	5.63	5.58	5.56	0.09	0.008	0.04	1.13	1.35
Lab 6			5.54	5.46	5.45	5.48	0.05	0.002	-0.03	-0.78	0.76

$\bar{\bar{x}}$ = average of labs averages 5.51

$s_{\bar{x}}$ = standard deviation of cell averages 0.038

s_r = repeatability standard deviation 0.065

s_L = between-lab standard deviation 0.009

s_R = reproducibility standard deviation 0.065

Table B3. Interlaboratory test results for mix 5-asphalt analyzer

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 4B	5	5.7	5.51	5.62	5.55	5.56	0.06	0.003	-0.03	-0.73	1.49
Lab 3B			5.66	5.64	5.61	5.64	0.03	0.001	0.05	1.14	0.68
Lab 6			5.55	5.58	5.59	5.57	0.02	0.000	-0.02	-0.41	0.56

$\bar{\bar{x}}$ = average of labs averages 5.59

$s_{\bar{x}}$ = standard deviation of cell averages 0.041

s_r = repeatability standard deviation 0.037

s_L = between-lab standard deviation 0.035

s_R = reproducibility standard deviation 0.051

Table B4. Interlaboratory test results for mix 6- asphalt analyzer

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 4B	6	5.6	5.4	5.4	5.23	5.34	0.10	0.010	-0.01	-0.25	1.10
Lab 3B			5.39	5.36	5.46	5.40	0.05	0.003	0.05	1.10	0.58
Lab 6			5.2	5.34	5.41	5.32	0.11	0.011	-0.04	-0.85	1.20

$\bar{\bar{x}}$ = average of labs averages 5.35

$s_{\bar{x}}$ = standard deviation of cell averages 0.044

s_r = repeatability standard deviation 0.089

s_L = between-lab standard deviation 0.000

s_R = reproducibility standard deviation 0.089

Table B5. Interlaboratory test results for Mix 8-asphalt analyzer

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 4B	8	5.5	5.44	5.58	5.27	5.43	0.16	0.024	-0.04	-1.03	1.23
Lab 3B			5.67	5.46	5.38	5.50	0.15	0.022	0.04	0.97	1.19
Lab 6			5.51	5.45	5.45	5.47	0.03	0.001	0.00	0.06	0.27

$\bar{\bar{x}}$ = average of labs averages 5.47

$s_{\bar{x}}$ = standard deviation of cell averages 0.037

s_r = repeatability standard deviation 0.126

s_L = between-lab standard deviation 0.000

s_R = reproducibility standard deviation 0.126

REFLUX ADDITIONAL TEST RESULTS

Table B6. Interlaboratory test results for mix 4-reflux

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d
			1	2	3				
Lab 1B	4	5.6	5.62	5.42	5.55	5.53	0.10	0.011	-0.01
Lab 4B			5.69	5.45	5.52	5.55	0.12	0.015	0.01

$\bar{\bar{x}}$ = average of labs averages 5.54

$s_{\bar{x}}$ = standard deviation of cell averages 0.017

s_r = repeatability standard deviation 0.114

s_L = between-lab standard deviation 0.000

s_R = reproducibility standard deviation 0.114

Table B7. Interlaboratory test results for mix 5-reflux

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d
			1	2	3				
Lab 1B	5	5.7	5.45	5.72	5.68	5.61	0.15	0.022	0.03
Lab 4B			5.55	5.61	5.52	5.56	0.05	0.002	-0.03
			$\bar{\bar{x}}$	= average of labs averages				5.59	
			$s_{\bar{x}}$	= standard deviation of cell averages				0.038	
			s_r	=repeatability standard deviation				0.109	
			s_L	=between-lab standard deviation				0.000	
			s_R	=reproducibility standard deviation				0.109	

Table B8. Interlaboratory test results for mix 6-reflux

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d
			1	2	3				
Lab 1B	6	5.6	5.24	5.22	5.28	5.25	0.03	0.001	-0.17
Lab 4B			5.63	5.49	5.67	5.60	0.09	0.009	0.17
			$\bar{\bar{x}}$	= average of labs averages				5.42	
			$s_{\bar{x}}$	= standard deviation of cell averages				0.247	
			s_r	= repeatability standard deviation				0.071	
			s_L	= between lab standard deviation				0.256	
			s_R	= reproducibility standard deviation				0.266	

Table B9. Interlaboratory test results for mix 7-reflux

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d
			1	2	3				
Lab 1B	7	5.4	5.12	5.05	4.93	5.03	0.09	0.009	-0.04
Lab 4B			5.13	5.04	5.14	5.10	0.06	0.003	0.04
			$\bar{\bar{x}}$	= average of labs averages				5.07	
			$s_{\bar{x}}$	= standard deviation of cell averages				0.051	
			s_r	= repeatability standard deviation				0.077	
			s_L	= between-lab standard deviation				0.092	
			s_R	= reproducibility standard deviation				0.120	

Table B10 Interlaboratory test results for mix 8-reflux

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d
			1	2	3				
Lab 1B	1	5.5	5.59	5.44	5.31	5.45	0.14	0.019	0.11
Lab 4C			5.32	5.31	5.06	5.23	0.15	0.022	-0.11

$\bar{\bar{x}}$ = average of labs averages 5.34

$s_{\bar{x}}$ = standard deviation of cell averages 0.154

s_r = repeatability standard deviation 0.143

s_L = between-lab standard deviation 0.116

s_R = reproducibility standard deviation 0.184

IGNITION TEST ADDITIONAL RESULTS (UNCORRECTED)

Table B11. Interlaboratory test results for mix 4- ignition oven

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	k
			1	2	3				
Lab 1C	4	5.6	6.19	6.25	6.10	6.18	0.08	0.006	1.11
Lab 4C			5.92	5.97	6.05	5.98	0.07	0.004	0.96
Lab 5			6.42	6.54	6.51	6.49	0.06	0.004	0.92

$\bar{\bar{x}}$ = average of labs averages 6.22

s_r = repeatability standard deviation 0.068

Table B12. Interlaboratory test results for mix 5- ignition oven

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	k
			1	2	3				
Lab 1C	5	5.7	6.25	6.39	6.31	6.32	0.07	0.005	0.74
Lab 4C			6.05	5.87	5.96	5.96	0.09	0.008	0.94
Lab 5			6.30	6.33	6.11	6.25	0.12	0.014	1.25

$\bar{\bar{x}}$ = average of labs averages 6.17

s_r = repeatability standard deviation 0.181

Table B13. Interlaboratory test results for mix 6- ignition oven

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	k
			1	2	3				
Lab 1C	6	5.6	5.95	6.13	5.96	6.01	0.10	0.010	0.76
Lab 4C			5.86	5.73	5.61	5.73	0.13	0.016	0.94
Lab 5			5.87	5.95	6.19	6.00	0.17	0.028	1.25
			$\bar{\bar{x}}$	= average of labs averages				5.92	
			s_r	=repeatability standard deviation				0.134	

Table B14. Interlaboratory test results for mix 7- ignition oven

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	k
			1	2	3				
Lab 1C	7	5.4	5.87	5.96	5.94	5.92	0.05	0.002	0.45
Lab 4C			5.32	5.40	5.55	5.42	0.12	0.014	1.12
Lab 5			5.78	5.71	5.96	5.82	0.13	0.017	1.24
			$\bar{\bar{x}}$	= average of labs averages				5.72	
			s_r	= repeatability standard deviation				0.263	

Table B15. Interlaboratory test results for mix 8-ignition oven

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	k
			1	2	3				
Lab 1C	8	5.4	6.03	6.15	6.14	6.11	0.07	0.004	1.37
Lab 4C			5.93	5.91	5.96	5.93	0.03	0.001	0.52
Lab 5			6.27	6.31	6.22	6.27	0.05	0.002	0.93
			$\bar{\bar{x}}$	= average of labs averages				6.10	
			s_r	= repeatability standard deviation				0.049	

IGNITION TEST ADDITIONAL RESULTS (CORRECTED)

Table B16. Interlaboratory test results for mix 4-ignition oven

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 1C	4	5.6	5.39	5.45	5.30	5.38	0.08	0.006	-0.14	-0.85	1.11
Lab 4C			5.42	5.47	5.55	5.48	0.07	0.004	-0.04	-0.26	0.96
Lab 5			5.64	5.76	5.73	5.71	0.06	0.004	0.19	1.10	0.92

$\bar{\bar{x}}$ = average of labs averages 5.52

$s_{\bar{x}}$ = standard deviation of cell averages 0.169

s_r = repeatability standard deviation 0.068

s_L = between-lab standard deviation 0.165

s_R = reproducibility standard deviation 0.178

Table B17. Interlaboratory test results for mix 5-ignition oven

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 1C	5	5.7	6.25	6.39	6.31	6.32	0.07	0.005	0.07	0.984	0.74
Lab 4C			6.05	5.87	5.96	5.96	0.09	0.008	0.00	0.032	0.94
Lab 5			6.30	6.33	6.11	6.25	0.12	0.014	-0.07	-1.015	1.25

$\bar{\bar{x}}$ = average of labs averages 5.72

$s_{\bar{x}}$ = standard deviation of cell averages 0.070

s_r = repeatability standard deviation 0.095

s_L = between-lab standard deviation 0.043

s_R = reproducibility standard deviation 0.105

Table B18. Interlaboratory test results for mix 6-ignition oven

Lab #	Mix	Asphalt Content	Test Results			Avg.	St. Dev.	s^2	d	h	k
			1	2	3						
Lab 1C	6	5.6	5.95	6.13	5.96	6.01	0.10	0.010	0.16	0.87	0.76
Lab 4C			5.86	5.73	5.61	5.73	0.13	0.016	0.04	0.22	0.94
Lab 5			5.87	5.95	6.19	6.00	0.17	0.028	-0.20	-1.09	1.25

$\bar{\bar{x}}$ = average of labs averages 5.54

$s_{\bar{x}}$ = standard deviation of cell averages 0.183

s_r = repeatability standard deviation 0.134

s_L = between-lab standard deviation 0.166

s_R = reproducibility standard deviation 0.203

Table B19. Interlaboratory test results for mix 7-ignition oven

Lab #	Mix	Asphalt Content	Test Results 1	2	3	Avg.	St. Dev.	s^2	d	h	k
Lab 1C	7	5.4	5.25	5.34	5.32	5.30	0.05	0.002	0.06	0.83	0.45
Lab 4C			5.07	5.15	5.30	5.17	0.12	0.014	-0.07	-1.11	1.12
Lab 5			5.23	5.16	5.41	5.27	0.13	0.017	0.02	0.28	1.24

$\bar{\bar{x}}$ = average of labs averages 5.25

$s_{\bar{x}}$ = standard deviation of cell averages 0.067

s_r =repeatability standard deviation 0.104

s_L =between-lab standard deviation 0.030

s_R =reproducibility standard deviation 0.108

Table B 20. Interlaboratory test results for mix 8-ignition oven

Lab #	Mix	Asphalt Content	Test Results 1	2	3	Avg.	St. Dev.	s^2	d	h	k
Lab 1C	8	5.5	5.36	5.48	5.47	5.44	0.07	0.004	0.04	0.001	0.51
Lab 4C			5.31	5.29	5.34	5.31	0.03	0.001	-0.09	0.007	-1.15
Lab 5			5.45	5.49	5.40	5.45	0.05	0.002	0.05	0.002	0.64

$\bar{\bar{x}}$ = average of labs averages 5.40

$s_{\bar{x}}$ = standard deviation of cell averages 0.074

s_r =repeatability standard deviation 0.049

s_L =between-lab standard deviation 0.069

s_R =reproducibility standard deviation 0.084

Appendix C. Draft for WisDOT Specifications-Sections 460.2.6 and 460.2.8.2.1.5

460.2.6 Recovered Asphaltic Binders

(1) Establish the percent of recovered asphaltic binder from FRAP, RAP, and RAS for the mixture design according to any the following procedures: AASHTO T164 method A (centrifuge extraction) and asphalt analyzer (per manufacturer's recommendation). In addition, AASHTO T 308 can be used at a temperature of 800°F for FRAP and RAP and 900°F for RAS materials. For RAS materials the sample size should not exceed 500 grams.

(2) The procedure selected cannot be changed during mix production without submitting a new mix design to the WisDOT for verification.

(3) If production test results indicate a change in the percent of recovered asphaltic binder, the contractor or the engineer may request a change in the design recovered asphaltic binder. Provide the department with at least 2 recent tests samples supporting that change. Ensure that those samples were prepared according to CMM 8-65 by a WisDOT qualified laboratory.

(4) The contractor may replace virgin binder with recovered binder up to the maximum percentage allowed under 460.2.5 without changing the asphaltic binder grade. If using more than the maximum allowed under 460.2.5, furnish test results indicating that the resultant binder meets the grade the contract originally specified.

460.2.8.2.1.5 Control Limits

(1) Conform to the following control limits for the JMF and warning limits based on a running average of the last 4 data points:

ITEM	JMF LIMITS	WARNING LIMITS
25.0-mm	+/- 6.0	+/- 4.5
19.0-mm	+/- 5.5	+/- 4.0
12.5-mm	+/- 5.5	+/- 4.0
9.5-mm	+/- 5.5	+/- 4.0
2.36-mm	+/- 5.0	+/- 4.0
75-µm	+/- 2.0	+/- 1.5
Asphaltic content in percent	- 0.5(1)/ -0.3(2)	-0.4(1)/ 0.2(2)
Air voids in percent	+/- 1.3	+/- 1.0
VMA in percent	- 0.5	- 0.2

(1) Asphaltic content in percent when AASHTO T164 method A or asphalt analyzer is used to determine asphalt content.

(2) Asphaltic content in percent when AASHTO T 308 is used to determine asphalt content and correction factors are properly applied.