#### May 2016

# Determination of the Binder Grade and Performance of High Percentage RAP-HMA Mixes



Charles D. Baker, Governor Karyn E. Polito, Lieutenant Governor Stephanie Pollack, MassDOT Secretary & CEO





### **Technical Report Document Page**

| 1. Report No.<br>SPRII-15-57946  | 2. Government Accession No.<br>n/a     | 5. 3. Rec<br>n/a   | cipient's Catalog No.                            |                     |
|--|--|--|--|---------------------|
| <sup>4. Title and Subtitle</sup><br>Determination of the Binder Grade and Performance<br>Percentage RAP-HMA Mixes  |  | e of High <sup>5. Rep</sup>  | ort Date<br>2016                                 |                     |
|  |  | 6. Peri $n/a$  | forming Organization C                           | ode                 |
| <sup>7. Author(s)</sup><br>Dr. Walaa S. Mogawer, P.E., F.ASCE<br>Mr. Alexander J. Austerman, P.E.<br>Troy Pauli<br>Steve Salmans<br>Jean-Pascal Planche  |  | 8. Perf<br>UM  | orming Organization Ro<br>ΓC-16.02               | eport No.           |
| 9. Performing Organization Name and Address<br>University of Massachusetts Da  | ortmouth                               | 10. Wo   | ork Unit No. (TRAIS)                             |                     |
| Highway Sustainability Research  | ch Center (HSRC)                       | 11. Co<br>12.2 IS A  | ntract or Grant No.<br># 57046                   |                     |
| 12. Sponsoring Agency Name and Address   |  | 23 ISA<br>13. Typ  | $\frac{\pi}{1}$ 57940<br>be of Report and Period | Covered             |
| Office of Transportation Planni  | ng                                     | Dece   | ember 2011 to I                                  | December 2015       |
| 10 Park Plaza, Ŝuite 4150<br>Boston, MA 02116  |  | 14. Spo<br>n/a   | onsoring Agency Code                             |                     |
| 15. Supplementary Notes n/a  |  |  |  |                     |
| 16. Abstract<br>This study's purpose was to better understand the effects of incorporating more RAP on the performance of a 9.5 mm Superpave surface<br>course mixture. The performance and workability characteristics of the mixtures were evaluated using two different Warm Mix Asphalt<br>(WMA) technologies and a softer virgin binder grade. The WMA technologies were organic and chemical based.  |  |  |  |                     |
| Mixtures incorporating up to 50% RAP were designed with the same binder content and gradation as the control mixture. At RAP contents beyond 50%, the gradation and binder content of the mixture could not be maintained.   |  |  |  |                     |
| Confirmation of the AASHTO blending charts recommended the use of asphalt binder grades not typically specified in Massachusetts.<br>Binder data indicated that mixtures edged closer to the cracking failure zone as the amount of RAP increased. The use of softer binder and WMA technologies helped to reverse the trends, but the mixtures remained within the onset of cracking zone.  |  |  | in Massachusetts.<br>e of softer binder and      |                     |
| Mixture performance data indicated reduced rutting performance, increased fatigue cracking resistance, and improved mixture workability when the softer binder was utilized. The use of either WMA technology did not significantly impact the rutting or fatigue cracking resistance, but did improve mixture workability.  |  |  |  |                     |
| Using a softer binder or a WMA technology alone did not yield a mixture with the same performance as an all-virgin material mixture.<br>Thus, using higher percentages of RAP in HMA must be carefully developed to reflect variations in individual mixtures based on the<br>properties of RAP, total amount of RAP, available virgin binders, and WMA technologies available. The use of asphalt rejuvenators<br>should be investigated for these types of mixtures to help reuse more of the binder in the RAP. |  |  |  |                     |
| 17. Key WordReclaimed Asphalt Pavement, Warm Mix Asphalt,Rheology, Performance   |  | 18. Distribution Statement<br>No restrictions. Thi<br>public through the s | s document is a<br>sponsoring ager               | vailable to the nt. |
| 19. Security Classif. (of this report)<br>Unclassified   | 20. Security Classif. (of Unclassified | this page)   | 21. No. of Pages 90                              | 22. Price<br>n/a    |
| <b>E</b> <sub>0</sub> <b>m DOT E</b> 1700 7 (0.72)   |  | 1 1 1  | -  | -                   |

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#### Determination of the Binder Grade and Performance of High Percentage RAP-HMA Mixes

**Final Report** 

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May 2016

### Acknowledgements

Prepared in cooperation with the Massachusetts Department of Transportation, Office of Transportation Planning, and the United States Department of Transportation, Federal Highway Administration.

The Project Team would like to acknowledge the efforts of Gregory Doyle, construction quality engineer for FHWA Massachusetts Division, and Edmund Naras, pavement management engineer at the MassDOT. The Project Team would also like to acknowledge research assistants Alex Bulhoes, Brendon Botelho, Kevin Ryan, and Maggie McDonald, who were involved with the laboratory testing for this project.

Finally, the Project Team would like to acknowledge the following people for their contributions to this project: Mike Nichols, from Aggregate Industries, who supplied the aggregates and RAP for this project; Pat Mitchell, from Bitumar, Mark Gabriel, and All States Asphalt, Inc. who supplied the binders for this project; and Chris Strack, from Sonneborn, Inc., and Tejash Gandhi, from Meadwestvaco, who supplied the WMA technologies for the project.

#### Disclaimer

The contents of this report reflect the views of the authors, who are responsible for the facts and the accuracy of the data presented herein. The contents do not necessarily reflect the official view or policies of the Massachusetts Department of Transportation, nor the Federal Highway Administration. This report does not constitute a standard, specification, or regulation.

### **Executive Summary**

This study of Determination of the Binder Grade and Performance of High Percentage RAP-HMA Mixes was undertaken as part of the Massachusetts Department of Transportation Research Program. This program is funded with Federal Highway Administration (FHWA) Statewide Planning and Research (SPR) funds. Through this program, applied research is conducted on topics of importance to the Commonwealth of Massachusetts transportation agencies.

The purpose of this project was to determine the appropriate binder grade for hot mix asphalt (HMA) mixtures containing varying high percentages of reclaimed asphalt pavement (RAP). For this project, high percentage RAP was defined as equal to or greater than 25%. Mixtures with up to 75% RAP content were attempted; however, only mixtures with up to 50% RAP were able to be designed and evaluated. This is due, in part, to the fact that the 75% RAP content mixture required fractionating the RAP to individual sizes. Since RAP fractionation was not required for the rest of the mixtures, the 75% RAP mixture was eliminated from the study. Testing of the remaining HMA mixtures was conducted in order to evaluate the effect of the higher percentage RAP on their performance. The performance aspects that were measured were mixture durability, stiffness, low temperature cracking characteristics, and fatigue cracking resistance.

Since the binder in RAP has been exposed to the elements for many years, it has experienced significant aging compared to a virgin binder. Therefore, it has different characteristics and quality. The aged binder in RAP causes an increase in mixture stiffness in mixtures high in RAP content. Mixture stiffness then leads to a reduced workability in high RAP mixtures. Thus, Warm Mix Asphalt (WMA) technologies have been utilized to help improve the workability and compactability of HMA mixes that incorporate RAP. Two of these technologies were evaluated in this study in order to determine their effects on mixture workability. Furthermore, the effects of these two WMA technologies and the use of a softer binder were investigated using rheology space diagrams. Diagramming was performed in order to determine the capabilities of either WMA technologies or a softer virgin binder to mitigate the effect of the aged RAP binder in the overall mixture.

Finally, the degree of blending (commingling) between the RAP binder and the virgin binder was evaluated in order to determine if the RAP binder fully contributed all of its binder to the final mixture.

Based on the data collected and analyzed for this study, the following conclusions were made:

1. Superpave 9.5 mm mixtures could be designed with up to 50% RAP with the same binder content and gradation as a control mixture with 0% RAP. Mixtures with RAP contents above 50% were not included in this study because they could not be designed to meet the same mixture requirements.

- 2. Increased RAP content led to a binder in the mixture that was more aged than the binder in the PG64-28 control mixture. The use of a PG52-34 binder (softer binder) or a WMA technology decreased the degree of aging, with the PG52-34 binder being more effective than the WMA technologies.
- 3. The results from a Black-Space rheology diagram showed that the control mixture and all RAP mixtures designed with the PG64-28 binder fell in the area of the "onset cracking zone." That is, the mixtures edged closer to the failure zone as the amount of RAP increased. The use of the softer PG52-34 binder or a WMA technology improved the cracking performance of the 35% and the 50% RAP mixtures; however, both mixtures still fell within the onset of cracking zone.
- 4. The Hamburg wheel tracking device (HWTD) mixture test illustrated that the use of the softer PG52-34 binder in the mixtures containing 35% and 50% RAP contents led to a mixture that was less stiff than the PG64-28 control mixture. This agreed with the binder data resulting from the rheology space diagrams. The WMA technologies did not reduce the stiffness of the mixtures significantly; this finding also agreed with the binder data.
- 5. The control mixture and all RAP mixtures designed with the PG64-28 binder did not show a significant difference in fatigue cracking resistance. The use of the softer PG52-34 binder did increase fatigue cracking resistance. This finding was consistent with the data collected from the rheology space diagrams, as well as the data from the HWTD mixture data, both of which indicated less mixture stiffness. The use of the WMA technology generally decreased fatigue cracking resistance compared to the other mixtures, although the mixture's significance is most clearly seen when compared to the PG52-34 mixtures.
- 6. Increasing the amount of RAP in the mixture decreased mixture workability. Although the use of the softer PG52-34 binder or a WMA technology indicated improved workability, their use must be balanced so that other performance indicators are not degraded.
- 7. Using the softer PG52-34 binder or a WMA technology alone yielded a mixture with more rutting susceptibility than the PG64-28 control mixture. Furthermore, rheology tests showed that the softer binder, or a WMA technology, would not result in a mixture with more resistance to aging. Thus, the use of higher percentages of RAP in HMA must be carefully developed for each specific mixture based on the properties of the RAP, the amount of RAP, the available virgin binders, and the available WMA technologies.
- 8. Analysis of the reduced binder contribution from the RAP suggested that the actual contribution of the RAP binder may not be a fixed value; hence, a state department of transportation agency cannot give a fixed credit for the binder in the RAP based solely on its contents.

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# List of Acronyms

| AASHTO  | American Association of State Highway and Transportation Officials |
|---------|--|
| AFM     | Atomic Force Microscopic   |
| AWD     | Asphalt Workability Device   |
| DOT     | Department of Transportation                                       |
| CAM     | Christensen-Anderson Model   |
| DSR     | Dynamic Shear Rheometer  |
| ESALs   | Equivalent Single Axle Loads                                       |
| HMA     | Hot Mix Asphalt  |
| HSRC    | Highway Sustainability Research Center                             |
| HWTD    | Hamburg Wheel Tracking Device                                      |
| MassDOT | Massachusetts Department of Transportation                         |
| NCHRP   | National Cooperative Highway Research Program                      |
| NEAUPG  | Northeast Asphalt User Producer Group                              |
| NMAS    | Nominal Maximum Aggregate Size                                     |
| PAV     | Pressure Aging Vessel  |
| PG      | Performance Grade  |
| PGAB    | Performance Grade Asphalt Binder                                   |
| RAP     | Reclaimed Asphalt Pavement   |
| RTFO    | Rolling Thin Film Oven   |
| SGC     | Superpave Gyratory Compactor                                       |
| SIP     | Stripping Inflection Point   |
| SPR     | Statewide Planning and Research                                    |
| VFA     | Voids Filled with Asphalt  |
| VMA     | Voids in Mineral Aggregate   |
| WMA     | Warm Mix Asphalt   |

### **1.0 Introduction and Methodology**

This study of Determination of the Binder Grade and Performance of High Percentage RAP-HMA Mixes was undertaken as part of the Massachusetts Department of Transportation Research Program. This program is funded with Federal Highway Administration (FHWA) Statewide Planning and Research (SPR) funds. Through this program, applied research is conducted on topics of importance to the Commonwealth of Massachusetts transportation agencies.

The price increase of oil has led to a dramatic increase in the cost of asphalt binder. Consequently, the expense for Hot Mix Asphalt (HMA) mixtures has increased substantially. To offset this rise in cost, government and private HMA agencies have been searching for methods to reduce the price of HMA mixtures. One option that has been utilized is to increase the amount of Recycled Asphalt Pavement (RAP) allowed in the HMA. Increasing RAP allows for a greater volume of the liquid asphalt binder in the HMA to come from the RAP, thereby decreasing the amount of virgin liquid asphalt binder required for the final mix. Thus, increasing the RAP could potentially save a significant amount of money for the agency in material costs.

MassDOT specification allows 15% RAP in its HMA mixtures. However, many HMA plants are now able to incorporate higher percentages of RAP into the HMA without a negative impact on the mixture's production or the environment in which it is utilized. Using RAP at higher percentages is viable due to the fact that there have been significant improvements made to the equipment used in its processing and handling; these improvements have thus yielded more efficient systems for crushing and fractionating the RAP into more useable forms. As a result, quality HMA can be produced with higher RAP contents than have typically been specified by state and municipal agencies. Several states are currently allowing higher than 10% RAP in their surface course mixtures. For example, New York allows 20% RAP in their surface mixtures, while other states like Arkansas and Minnesota allow over 35% RAP (1).

The liquid asphalt from RAP is stiff because it has already been aged. Since the stiffness of the liquid asphalt impacts the performance of HMA mixtures (such as enabling premature cracking and creating less compactable mixtures), it is essential to use a virgin binder that will lead to a target Performance Grade Asphalt Binder (PGAB) when blended with the liquid asphalt from RAP. The American Association of State Highway and Transportation Officials (AASHTO) Specification M323, "Superpave Volumetric Mix Design," (2) presents a procedure based on the research results of the National Cooperative Highway Research Program (NCHRP) Project 9-12 "Incorporation of Reclaimed Asphalt Pavement in the Superpave System" (3) that can assist in selecting the proper PGAB for high percentages of RAP. However, the procedure generally leads to the use of a softer binder. Recent studies have shown that the use of a PG64-XX can lead to the same performance as a softer PG58-XX, or even PG52-XX (4).

The purpose of this project was to determine the binder grade of HMA mixtures containing varying high percentages of RAP (for this project, high percentage RAP is defined as equal to or greater than 25%). Also, testing (durability, stiffness, low temperature cracking characteristics, and fatigue cracking resistance) of these HMA mixtures was conducted in order to evaluate the effect of the higher percentage RAP on the performance of HMA mixes.

Since the aged binder in RAP causes an increase in mix stiffness, the resultant HMA mixes tend to be less workable. Warm Mix Asphalt (WMA) technologies have been utilized to help improve the workability and compactability of HMA mixes that incorporate RAP. Two of these technologies were evaluated in this study in order to determine their effects on mixture workability.

Finally, the degree of blending (commingling) between the RAP binder and the added virgin binder was evaluated in an attempt to determine if the RAP binder does, in fact, fully contribute all of its binder to the final mixture.

The objectives of this project are listed below:

- 1. Prepare a typical Superpave mix using 0%, 15%, 35%, and 75% RAP with one aggregate source, one RAP source, and two Performance Grade Asphalt Binders.
- 2. Measure the degree of blending between the aged asphalt liquid and the added virgin liquid.
- 3. Evaluate the effect of WMA on the workability of HMA with high percentages of RAP.
- 4. Evaluate and compare the performance of each mix in terms of its durability, fatigue cracking resistance, and stiffness.

In order to fulfill the objectives of this study, an experimental plan was proposed as shown in Figures 1.1 and 1.2. However, over the course of the project, several new methodologies emerged that helped the industry better evaluate the effect of the aged RAP binder on the rheology characteristics of the resulting binder. In order to evaluate the quality of the resultant binder in a high RAP mixture, the new methodology of chemical testing was attempted. This test could also be used to evaluate the degree of blending between aged and virgin binders. All tests will be further explained in the sections detailing binder testing and mixture performance testing.



Figure 1.1: Experimental Plan (Part I)



Figure 1.2: Experimental Plan (Part II)

### 2.0 Materials

In this section, the materials utilized for this study are described. These materials include the asphalt binder, aggregates, and WMA technologies.

#### 2.1 Asphalt Binder

For this study, two different asphalt binders were utilized. The binders consisted of a PG64-28, which is typically specified in the Northeast, and a PG52-34. The PG64-28 was obtained from Aggregate Industries, and the PG52-34 was obtained from All States Asphalt.

The PG grade of each binder was verified in accordance with AASHTO M320 "Standard Specification for Performance-Graded Asphalt Binder" (2), and the mixing and compaction temperatures were determined based on each binder's viscosity. The results of these verifications and tests are shown in Table 2.1.

| Binder  | Continuous<br>Grade | Performance<br>Grade (PG) | Mixing<br>Temperature<br>Range | Compaction<br>Temperature<br>Range |
|---------|---------------------|---------------------------|--------------------------------|------------------------------------|
| PG64-28 | 64.4-28.1           | PG64-28                   | 162–158°C<br>(324–316°F)       | 152–146°C<br>(306–295°F)           |
| PG52-34 | 54.8-34.5           | PG52-34                   | 147–139°C<br>(296–283°F)       | 127–115°C<br>(260–239°F)           |

**Table 2.1: Asphalt Binder Information** 

#### 2.2 Aggregates

The aggregates utilized were from a crushed stone source in Wrentham, Massachusetts. The two aggregate stockpiles that were obtained consisted of the following: 9.5 mm crushed stone and stone dust. Each aggregate stockpile was tested to determine its properties. These properties are shown in Table 2.2. Sieve analysis was completed in accordance with two methods: the AASHTO test method T11, also known as the "Standard Method of Test for Materials Finer Than 75-µm (No. 200) Sieve in Mineral Aggregates by Washing," and T27, also called the "Standard Method of Test for Sieve Analysis of Fine and Coarse Aggregates" (2).

| Sieve Size | 9 5 mm           | Stone |
|------------|------------------|-------|
| SIEVE SIZE | <b>7.5</b> IIIII | Dust  |
| 19.0 mm    | 100              | 100   |
| 12.5 mm    | 99.4             | 100   |
| 9.5 mm     | 93.8             | 100   |
| 4.75 mm    | 29.7             | 99.7  |
| 2.36 mm    | 5.2              | 83.7  |
| 1.18 mm    | 2.8              | 57.1  |
| 0.600 mm   | 2.3              | 38.6  |
| 0.300 mm   | 2.1              | 24.9  |
| 0.150 mm   | 1.8              | 15.9  |
| 0.075 mm   | 1.5              | 10.9  |

 Table 2.2: Aggregate Properties

#### **2.3 Reclaimed Asphalt Pavement (RAP)**

RAP was obtained from the same contractor as the aggregates. The RAP stockpile was fractionated in the laboratory in order to meet the mixture gradation requirements for this study. The binder content of the RAP material was determined to be 5.6% using the ignition oven test method, in accordance with AASHTO T308, "Determining the Asphalt Binder Content of Hot Mix Asphalt (HMA) by the Ignition Method" (2). The aggregates in the RAP that remained after the ignition were tested in order to determine their properties. These properties are also shown in Table 2.3. The RAP binder was extracted and recovered in accordance with AASHTO T164, or the "Standard Method of Test for Quantitative Extraction of Asphalt Binder from Hot Mix Asphalt (HMA)," as well as AASHTO T170, the "Standard Method of Test for Recovery of Asphalt Binder from Solution by Abson Method" (2). As shown in Table 2.3, the continuous and performance grade of the recovered RAP binder was determined in accordance with AASHTO R29, "Grading or Verifying the Performance Grade of an Asphalt Binder" (2).

| Sieve Size                            | RAP Aggregates<br>Post Ignition |
|---------------------------------------|---------------------------------|
| 9.5 mm                                | 100                             |
| 4.75 mm                               | 76.8                            |
| 2.36 mm                               | 57.6                            |
| 1.18 mm                               | 43.3                            |
| 0.600 mm                              | 31.1                            |
| 0.300 mm                              | 19.8                            |
| 0.150 mm                              | 12.1                            |
| 0.075 mm                              | 8.3                             |
|                                       |                                 |
| <b>RAP Binder Content, % =</b>        | 5.6%                            |
| <b>RAP Binder Continuous Grade =</b>  | 82.0-21.8                       |
| <b>RAP Binder Performance Grade =</b> | PG82-16                         |

**Table 2.3: RAP Properties** 

#### 2.4 Warm Mix Asphalt (WMA)Technologies

Two different WMA technologies were utilized for this study. According to the MassDOT technical representative, the technologies were chosen from the Northeast Asphalt User Producer Group (NEAUPG) approved list. From the approved NEAUPG list, the MassDOT technical representative approved the use of an organic-based WMA technology (1.0% SonneWarmix by weight of binder) and a chemical-based technology (0.5% Evotherm P15 by weight of binder). The reduced mixing and compaction temperatures for using each WMA with the PG64-28 were 135°C (275°F) and 125°C (256°F), respectively. Similarly, the reduced mixing and compaction temperatures for using each WMA with the PG52-34 were 118°C (245°F) and 105°C (220°F), respectively, based on the manufacturers' recommendations.

### 3.0 Mixture Design

The target gradation for the mixtures utilized in this study is shown in Table 3.1. The target gradation was developed to meet the requirements for a 9.5 mm Superpave mixture in accordance with AASHTO M323, "Superpave Volumetric Mix Design," as well as AASHTO R35, "Superpave Volumetric Design for Hot Mix Asphalt" (2). The design Equivalent Single Axle Loads (ESALs) for this project was selected as 0.3 to <3 million, which is consistent with surface course mixtures in New England. The design Superpave gyratory compactive effort for this ESALs level was Ndesign = 75 gyrations.

| Sieve Size       | Sieve Size<br>(mm) | Target<br>Gradation<br>for All<br>Mixtures | Superpave<br>9.5 mm<br>Specification |
|------------------|--------------------|--|--------------------------------------|
| 1/2"             | 12.5 mm            | 100  | 100 min                              |
| 3/8"             | 9.5 mm             | 98   | 90–100                               |
| No. 4            | 4.75 mm            | 85   | 90 max                               |
| No. 8            | 2.36 mm            | 58   | 32–67                                |
| No. 16           | 1.18 mm            | 42   | -                                    |
| No. 30           | 0.600 mm           | 27   | -                                    |
| No. 50           | 0.300 mm           | 15   | -                                    |
| No. 100          | 0.150 mm           | 9  | -                                    |
| No. 200          | 0.075 mm           | 6  | 2–10                                 |
| Binder Content = |                    | 6.5%                                       | -                                    |

**Table 3.1: Target Mixture Gradation and Specification** 

First, the control mixtures utilizing all virgin materials were designed utilizing the PG64-28 binder and the PG52-34 binder for comparison purposes. RAP was then incorporated to replace a portion of the virgin materials in the control mixtures. RAP was added to replace a percentage of the mixture aggregates with RAP aggregates (25%, 50%, and 75%). The aggregate gradations for the control and RAP mixtures were identical. Because the gradations were identical for the mixtures developed, mixture design verifications were performed for the RAP mixtures. These verifications attended to RAP both with WMA and without WMA in order to reflect the design binder content determined for the control mixture. Verifications were completed assuming 100% contribution of the RAP binder.

To incorporate the RAP into the mixtures, a procedure that was used in a prior study utilizing similar materials was followed (5). This procedure was used in order to eliminate moisture in the RAP stockpile material and to optimize the blending between the aged and virgin binders in the mixture. The procedural steps are listed below:

- 1. The RAP was air dried until a constant mass was achieved, which typically took three to five days.
- 2. The RAP was further dried for two days at 60°C (140°F).

3. The RAP was added to heated aggregate during the mixing process two hours prior to adding the binder.

During initial verifications of the 75% RAP mixture, it was noted that the volumetric properties were very different from the control mixture. Forensic testing indicated that the RAP fractionating required for the 75% RAP mixture yielded a mixture that had a different binder content and gradation than that of the control. Thus, no further testing was completed on this mixture, since it could not be compared with any of the other mixtures. Instead, RAP contents of 15% and 35% were added to the study. A 50% RAP mixture was determined to be the upper limit that could be utilized without requiring further fractionation of the RAP. In total, 30 mixture designs/verifications were completed, as shown in Table 3.2.

The results of the mixture designs and verifications for each of the 30 mixtures are shown in Appendix 9-1. Generally, the volumetric data shows an increase in air voids as the percentage of RAP is increased. This increase may indicate that there is not 100% contribution of the RAP binder, which then leads to a mixture with less effective asphalt binder content. Another possible explanation is that good blending took place, resulting in a stiff binder, which then led to a mixture that was difficult to compact. Additionally, certain WMA mixtures exhibited high air voids; these voids may indicate that the compaction temperature reductions utilized were too great, because they led to a stiff mixture that was more difficult to compact.

Based on the mixture design and verifications, mixtures were selected for further analysis in terms of both binder and mixture performance testing. These mixtures were selected in an attempt to address the variables of the study, which included RAP content, different virgin binder grades, and different WMA technologies. The mixtures for further analysis are shown in Table 3.3.

| Vingin Dindon |        | WMA          |
|---------------|--------|--------------|
| virgin bilder | 70 KAF | Additive     |
|               |        | HMA – None   |
|               | 0%     | SonneWarmix  |
|               |        | Evotherm P15 |
|               |        | HMA – None   |
|               | 15%    | SonneWarmix  |
|               |        | Evotherm P15 |
|               |        | HMA – None   |
| PG64-28       | 25%    | SonneWarmix  |
|               |        | Evotherm P15 |
|               |        | HMA – None   |
|               | 35%    | SonneWarmix  |
|               |        | Evotherm P15 |
|               |        | HMA – None   |
|               | 50%    | SonneWarmix  |
|               |        | Evotherm P15 |
|               | 0%     | HMA – None   |
|               |        | SonneWarmix  |
|               |        | Evotherm P15 |
|               | 15%    | HMA – None   |
|               |        | SonneWarmix  |
|               |        | Evotherm P15 |
|               |        | HMA – None   |
| PG52-34       | 25%    | SonneWarmix  |
|               |        | Evotherm P15 |
|               |        | HMA – None   |
|               | 35%    | SonneWarmix  |
|               |        | Evotherm P15 |
|               | 50%    | HMA – None   |
|               |        | SonneWarmix  |
|               |        | Evotherm P15 |

 Table 3.2: Mixture Design and Verifications

| Binder  | % RAP        | WMA Additive               |
|---------|--------------|----------------------------|
|         | 0% – Control | NONE                       |
|         | 15%          | NONE                       |
| PG64-28 | 25%          | NONE                       |
|         | 35%          | NONE                       |
|         | 50%          | NONE                       |
| PG64-28 | 35%          | SonneWarmix & Evotherm P15 |
|         | 50%          | SonneWarmix & Evotherm P15 |
| PG52-34 | 35%          | SonneWarmix & Evotherm P15 |
|         | 50%          | SonneWarmix & Evotherm P15 |

**Table 3.3: Mixture Selected for Evaluation** 

### 4.0 Binder Testing

This section outlines the binder testing corresponding to the nine mixtures selected for evaluation, as previously shown in Table 3.3.

#### 4.1 AASHTO M323 Blending Charts

As outlined in the experimental plan, one task of this project was to confirm the selection of the virgin binder grade using the methodology in AASHTO M323 (2). Based on the extracted RAP properties, the virgin binder grade required for each percentage of RAP is shown in Table 4.1 (assuming a PG64-28 is the desired final PG grade of the binder in the mixture). The grades determined vary from the grades commonly utilized and specified in Massachusetts.

| % RAP | Virgin Grade Required by<br>AASHTO M323 |
|-------|---|
| 15%   | PG64-34                                 |
| 25%   | PG58-34                                 |
| 35%   | PG58-34                                 |
| 50%   | PG46-40                                 |

Table 4.1: Blending Chart Confirmation by AASHTO M323

#### 4.2 Black Space Diagram and CAM Model

Recently, G\* and  $\delta$  have also been used to generate a rheological plot commonly referred to as a Black Space Diagram. Researchers have illustrated the use of a Black Space Diagram to evaluate the changes in binder rheology due to aging (6, 7). Another analysis that can be used to evaluate these changes is the Christensen-Anderson Model (CAM). The two methodologies were the focus of this study in order to understand the impact of the aged RAP binder at varying percentages.

#### 4.2.1 Black Space Diagram and the Glover-Rowe Damage Parameter

Figure 4.1 illustrates a Black Space diagram that shows the current performance grade (PG) parameter for fatigue cracking (G\*sin $\delta$ ), in addition to a new Black Space function defined by a new parameter, named the Glover-Rowe parameter, in the form of G\*(cos  $\delta$ )<sup>2</sup>/(sin $\delta$ ) (7). This parameter was developed based on the Glover fatigue cracking parameter, G'/( $\eta$ '/G'), which was found to have a high correlation with the ductility of the asphalt binder (8). Asphalt ductility is determined from the intermediate temperature determined during dynamic shear rheometer (DSR) testing. The advantage of this Glover-Rowe parameter is that as long as the test frequency ( $\omega$ ) is known, variables G\* and  $\delta$  can be plotted to create a

damage curve in black space. Based on the work of Anderson et al. (7) and Rowe (9), preliminary thresholds have been proposed to determine when non-load associated cracking, specifically block cracking, may begin (Damage Onset) and when there will be definite cracking problems (Significant Cracking). A typical cause of block cracking is the inability of the asphalt binder to expand and contract with temperature cycles because of the constant aging of the asphalt binder. These thresholds have  $G^*(\cos \delta)^2/(\sin \delta)$  values of 180 kPa and 450 kPa, respectively, when tested at 15°C (59°F) and a loading frequency of 0.005 radians/sec.



Figure 4.1: PAV-aged Binders Passing through the Glover-Rowe Damage Zone

Similar to the work described by Anderson et al. (7), Figure 4.1 illustrates data for two different binders (PG64-22 and PG76-22) that were aged in a pressure aging vessel (PAV) for 0, 20, 40, and 80 hours. The PAV aging was done after all binders were short-term aged in a rolling thin film oven (RTFO). The purpose of the longer PAV aging times was to create a more highly aged sample in the laboratory. Using the new Glover-Rowe parameter and presenting the data in Figure 4.1, the RTFO aging for each binder started at the lower right location in the Black Space diagram; each additional aging period caused the rheological properties to move to the upper left of the diagram (demonstrating an increase in stiffness and a reduced phase angle for each binder). It should be noted that even after 60 hours of

PAV aging, the asphalt binders still "pass" the current G\*sinδ Superpave specification. For extracted and recovered binders from high RAP content mixtures with consequently higher amounts of aged binder in a mixture, binder testing data is expected to follow the same trend as illustrated in Figure 4.1. The rheological response shows a trend to move toward the upper left of the Black Space diagram. Daniel (10) confirmed this trend on both extracted and recovered asphalt binders from plant-produced mixtures in Vermont. The trend in the Black Space diagram indicated that as RAP percentage increased for the same mixture, the G\* and  $\delta$  data migrated from the lower right to the upper left of the Black Space. Therefore, it is expected in this study that as the amount of RAP increases, the same trend would result. This would also be a good indication that good blending is occurring between the aged and virgin binders.

For the mixtures in this study, the binder from volumetric specimens was extracted and recovered. G\* master curves were also measured and utilized in order to evaluate the overall stiffness properties of the asphalt binders, as well as their relative aging characteristics. Prior to actual testing of each recovered asphalt binder, the DSR was run in an oscillatory mode in order to ensure that the asphalt binder sample would be tested in the linear region at the respective test temperature. The following test temperatures were used in the following order: 10°, 22°, 34°, and 46°C. The following range of loading frequencies were utilized to cover two decades of loading times and were conducted in the following order: 100, 62.8, 31.4, 10, 6.28, 3.14, 1.0, 0.63, 0.1, and 0.063 radians/sec. The Glover-Rowe parameter was determined by curve shifting and not measured directly at 0.005 radians/second at 15°C.

As indicated in Figures 4.2 to 4.4, the use of the PG64-28 showed that the control mixtures and the RAP mixtures fell in the onset of cracking zone in the Black Space diagram. It should be noted that the mixtures edged closer to the failure zone, in terms of cracking due to aging, as the amount of RAP increased. The use of softer binder and WMA helped reverse the trends for the 35% and the 50% RAP mixtures; nevertheless, both mixtures were still in the onset of cracking zone.



Figure 4.2: Black Space Diagram for Extracted Mixtures Binders, PG64-28 HMA



Figure 4.3: Black Space Diagram for Extracted Mixtures Binders, PG52-34HMA



Figure 4.4: Black Space Diagram for Extracted Mixtures Binders- PG64-28 WMA

#### 4.2.2 Christensen-Anderson Model (CAM) Master Curve Parameters

Another tool that can be used to investigate the effect of incorporating high RAP contents on the rheological properties of the resultant binders is to use traditional rheological master curves of G\* versus loading frequency. The Christensen-Anderson Model (CAM) is a very useful tool because the master curve parameters ( $\omega_0$ , R, and T<sub>d</sub>) have specific physical significance. The Cross-over Frequency,  $\omega_0$ , is a measure of the overall hardness of the binder. As this frequency increases, the hardness of the binder decreases, which is desirable for rejuvenated binders. The Rheological Index, R-value, is an indicator of the rheological type. It is defined as the difference between the log of the glassy modulus and the log of the dynamic modulus at the crossover frequency. As R-value increases, the master curve becomes flatter, indicating a more gradual transition from elastic behavior to steady-state flow. Normally, R-value is higher for oxidized asphalt. Accordingly, the R-value is expected to increase with oxidization. Therefore, for rejuvenators to be effective, the R-value for the overall aged binder plus virgin binder should decrease. Figure 4.5 shows the parameters used to define the shape of the binder master curve.


Figure 4.5: Functional Form of Christensen-Anderson Asphalt Binder Master Curve Model (11)

Figure 4.6 shows the  $\omega_0$  and R-value for the same test data presented earlier in Figure 4.1. In this case,  $\omega_0$  and R-value are plotted in their own space ( $\omega_0 - \text{R-value Space}$ ). The PG64-22 and PG76-22 asphalt binders migrate from the upper left to the lower right of the  $\omega_0 - \text{R-value Space}$  as the magnitude of aging increases. The same trend can be expected as the RAP content of the asphalt mixture increases.

The results and subsequent analyses of the master curve test indicate that  $G^*$  and  $\delta$ , as well as the functional form of the master curve itself ( $\omega_o$  and R-value), can be used to evaluate aging in an asphalt binder. Since aging can be clearly identified using this method, it is hypothesized that the same testing and evaluative procedures can be used to determine the effectiveness of incorporating more RAP, which in turn increases the amount of aged binder in the mix.

Testing was completed as described for development of the Black Space diagrams. The data was used to determine  $\omega_0$  and R-values for each extracted mixture binder. As indicated in Figure 4.7, the increase of the RAP content led to a resultant binder that was more aged than a control mixture with no RAP. However, the use of a softer binder or a WMA reversed the trend, showing that the softer binder helped in mitigating the aging of the binder that resulted from the RAP binder. Also, similar to the findings of the Black Space diagram, the results indicated that the softer binder had more effect in mitigating aging than the WMA technologies used.



Figure 4.6: Crossover Frequency, R-value Space: PG64-22 and PG76-22 Asphalt Binders after Different Aging Levels



Figure 4.7: Crossover Frequency, R-value Space: Extracted Mixture Binders for This Study

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## **5.0 Mixtures Performance Tests and Results**

This section outlines the mixture testing corresponding to the nine mixtures selected for evaluation, as shown previously in Table 3.3.

### 5.1 Rutting and Moisture Susceptibility, Hamburg Wheel Tracking Device (HWTD)

Rutting and moisture damage was conducted in accordance with AASHTO T324 "Hamburg Wheel-Track Testing of Compacted Hot-Mix Asphalt (HMA)" (2). This test is used to determine the failure susceptibility of a mixture due to weakness in the aggregate structure, inadequate binder stiffness, or moisture damage (2). In this test, a mixture is submerged in heated water (typically 40° to 50°C) and subjected to repeated loading provided by a 705N (158 lb.) steel wheel. As the steel wheel loads the specimen, the corresponding rut depth of the specimen is recorded. The rut depth versus the number of wheel passes is plotted in order to determine the Stripping Inflection Point (SIP), as shown in Figure 5.1. The SIP gives an indication of the point at which the test specimen begins to exhibit moisture damage (stripping).

Gyratory specimens for this test were fabricated using the Superpave gyratory compactor (SGC) to an air void level of  $7.0\pm1.0\%$ , as required by AASHTO T324. Testing was conducted at a temperature of 50°C (122°F). The specimens were tested at a rate of 52 passes per minute after a soak time of 30 minutes at the test temperature. Testing terminated at 20,000 wheel passes, or when visible stripping was noted. Figures 5.2 to 5.4 present the results of the Hamburg Wheel Tracking Device (HWTD).

The data in Figures 5.2 to 5.4 illustrate that the use of a softer binder with the 35% and 50% RAP led to an overall binder that was less stiff than binder resulting from the use of PG64-28 (i.e., increased rutting). This agreed with the Black Space and  $\omega_0$  – R-value diagrams. Also, as illustrated in Figure 5.3, the two WMA technologies did not reduce the stiffness of the aging binder significantly, which again agreed with both binder testing diagrams.

![](_page_41_Figure_0.jpeg)

**Figure 5.1: Stripping Inflection Point** 

#### MassDOT RAP Study Hamburg Results Laboratory Mixture

![](_page_42_Figure_1.jpeg)

Figure 5.2: HWTD Results for PG64-28 HMA Mixtures

### MassDOT RAP Hamburg Results

![](_page_43_Figure_1.jpeg)

![](_page_43_Figure_2.jpeg)

Figure 5.3: HWTD Results for PG52-34 HMA Mixtures

### MassDOT RAP Hamburg Results

![](_page_44_Figure_1.jpeg)

![](_page_44_Figure_2.jpeg)

Figure 5.4: HWTD Results for PG64-28 WMA Mixtures

### **5.2 Fatigue Cracking: Flexural Beam** Fatigue

For this test, beam specimens were cut from slabs. Slabs with dimensions of 150 mm wide, 150 mm tall, and 450 mm long (6 in. wide, 6 in. tall, and 17.5 in. long) were fabricated for each mixture using the IPC Global Pressbox slab compactor. From each slab, beams with dimensions of 63 mm wide, 50 mm tall, and 380 mm long (2.5 in. wide, 2 in. tall, and 15 in. long) were cut such that the sides had smooth faces. The air voids of the final cut specimens were  $7\pm1\%$ .

Beam specimens were conditioned at the test temperature of 15°C (59°F) for at least two hours prior to testing. Each beam fatigue test was conducted in strain control mode at a loading frequency of 10Hz applied using a sinusoidal waveform. Specimens were tested at a strain level of 500µε because all mixtures lost 50% of their initial stiffness after at least 10,000 cycles. The number of cycles to mixture failure was determined by fitting an exponential function to the flexural stiffness versus number of cycles and then evaluating the number of cycles that it took to decrease the initial stiffness by 50%. The beam fatigue testing results are shown in Figure 5.5.

![](_page_45_Figure_1.jpeg)

AASHTO T321 Beam Fatigue N<sub>f</sub> to 50% Reduction in Initial Stiffness

**Figure 5.5: Beam Fatigue Results** 

The error bars shown in Figure 5.5 indicate the standard deviation of the cycle to failure measurements collected on multiple beams tested for each mixture. If the error bars overlapped between mixtures, the data was not significantly different. Generally, the PG64-28 HMA mixture at all RAP contents up to 50% RAP did not show a significant difference in fatigue cracking resistance. The use of the softer PG52-34 binder did show increased fatigue cracking resistance as compared to the PG64-28 HMA mixtures. This result agreed well with the Black Space and  $\omega_0$  – R-value diagrams and the mixture rutting results, which likewise indicated that the mixture was less stiff (i.e., more crack resistant). The use of the VMA technology generally indicated reduced fatigue cracking resistance as compared to the other mixtures, but was generally only significant when compared to the PG52-34 mixtures.

Another component of this study was to investigate the effect of increased Voids in Mineral Aggregate (VMA) on the fatigue life of RAP mixtures. Increasing the VMA of the mixture would require redesigning the mixture with a different gradation and binder content, and thus would not be comparable to any mixtures in this study. In actuality, the concern with using higher percentages of RAP in the mixtures is whether or not all of the RAP binder can be

adequately utilized (i.e., 100% blending). If complete blending does not occur, then the mixture may have a lower effective asphalt binder content (under asphalted condition); this can then lead to performance degradation, particularly in terms of fatigue cracking. Thus, understanding the true contribution of the RAP binder to the overall mixture is of the utmost importance. In order to better understand this phenomenon, beam specimens were fabricated assuming only 70% of the RAP binder could be utilized for the 35% and 50% PG64-28 HMA mixtures. For these mixtures, the design binder content remained the same at 6.5%; however, the added virgin binder amount was increased compared to the previously tested mixtures, which assumed 100% contribution of the RAP binder. The beams were fabricated and prepared in the same manner previously described. For both the 35% and 50% RAP mixtures, as shown in Figure 5.6, assuming less contribution of RAP binder yields a mixture with more effective asphalt and thus the fatigue cracking resistance of the mixture is increased. Comparison of the volumetric data between the 100% and 70% contribution of RAP binder mixtures indicated very similar properties for the 35% RAP mixture, but less similar properties for the 50% RAP mixture. These results suggest the actual contribution of RAP binder may be not a fixed value, but rather varies based on RAP type and content.

![](_page_46_Figure_1.jpeg)

AASHTO T321 Beam Fatigue N<sub>f</sub> to 50% Reduction in Initial Stiffness

Figure 5.6: Beam Fatigue Results for Extra Added Binder

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# **5.3 Workability: Asphalt Workability Device** (AWD)

Because of the potential decrease in mixture workability due to the incorporation of higher amounts of RAP with and without WMA in the mixtures, workability evaluations of each of the mixtures were completed. These evaluations were conducted using an HMA workability device developed by the University of Massachusetts Dartmouth Highway Sustainability Research Center (HSRC). This device is known as the Asphalt Workability Device (AWD) and had been used previously to evaluate high percentage RAP mixtures as well as mixtures incorporating WMA additives (*12, 13*).

The AWD operates on the torque measurement principles that have been previously established (14). The AWD rotates the loose HMA mixture at a constant speed (15 rpm for this study) and separately records the resultant torque exerted on a pug mill style paddle shaft embedded into the mixture. The surface and internal temperatures of the mixture are recorded concurrently. As the mixture cools in ambient conditions, the torque exerted on the shaft increases, thereby giving an indication of the workability of the mixture at different temperatures. Each of the mixtures in this study was mixed and aged (four hours) at the mixing and compaction temperatures previously outlined. After completion of aging, the loose mixture was tested in the AWD.

From the AWD test data for each selected mixture, a best-fit exponential line was inserted into the raw data. This fit line was then used to develop a model curve plotted over the AWD test temperature range in which torque and temperature data were collected. This temperature range included the anticipated field placement and compaction temperatures of the mixture. The model curves are shown in Figure 5.7. Note that mixtures exhibiting lower torque values were considered more workable.

![](_page_48_Figure_0.jpeg)

Torque vs.Temperature Workability - MassDOT RAP Mixtures

![](_page_48_Figure_2.jpeg)

For the PG64-28 HMA mixtures, the workability data was consistent with the anticipated result and thus indicated that mixture workability is decreased as more RAP (50%) is introduced into the mixture, as compared to a mixture with no RAP (control). The use of the softer PG52-34 mixture showed increased mixture workability for both the control and 50% RAP mixtures compared to the PG64-28 HMA mixtures. This agreed well with the binder and mixture tests results that consistently indicated that the PG52-34 mixtures were less stiff than the other mixtures tested. The use of either WMA technology in the PG64-28 50% RAP mixture, at reduced mixing and compaction temperatures, indicated improved mixture workability over the corresponding 50% RAP HMA mixture and the control mixture. However, the improvement was not as great as exhibited by the softer binder (PG52-34) mixtures tested.

Overall, the workability data indicated that using increased amounts of RAP in the mixture will reduce mixture workability. Using a WMA technology or softer binder can improve these workability reductions, but their use must be balanced so that other performance indicators are not degraded.

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## 6.0 Chemical Analysis to Determine Quality and Degree of Blending of RAP Asphalt Binders Leading to Performance Prediction

Comprehensive testing of extracted and recovered binders from various mixtures was undertaken in order to understand the quality of asphalt blending in the various mixtures examined in this study. The quality of blending was determined by comparing the chemistries of the various blended asphalts to those of the PG64-28 control asphalt and the asphalt in the RAP stockpile. The specific details of this testing are located in a detailed report in Appendix 9.2.

## 6.1 Chemical Analysis Testing Conclusions and Recommendations

Asphalts are often classified as either a sol-type of asphalt (which has more compatible chemistries), or a gel-type of asphalt (which has less compatible chemistries). Asphalts with more compatible chemistries are usually more ductile, less elastic, more sensitive to temperature changes, and have low amounts of asphaltenes that are well dispersed or peptized by the maltene phase. Asphaltenes that allow themselves to be dispersed are called peptizable asphaltenes. Asphalts with less compatible chemistries usually have the opposite characteristics.

To determine the chemistries of the extracted and recovered asphalts, the asphaltene content of each asphalt was measured, along with a characterization of their state of dispersion in the maltene phase using the Heithaus compatibility parameters,  $p_a$ ,  $p_o$ , and P. The parameter  $p_a$  measures the peptizability of the asphaltenes;  $p_o$  measures the solvent power of the maltenes; and P is a measure of the overall compatibility of the asphalt. Larger values of  $p_a$ ,  $p_o$ , and P generally represent more peptizable asphaltenes, maltenes that are a good solvent, and an overall compatible asphalt. Smaller values of  $p_a$ ,  $p_o$ , and P represent the reverse. It has been found that asphalts with high asphaltene contents generally have low  $p_a$  values, while some studies indicate that asphalts with high concentrations of polar aromatic materials should have high  $p_o$  values. It has also been generally found that  $p_a$  values will decrease while  $p_o$  values will increasing oxidation of an asphalt. With aging, the asphaltenes become less peptizable, while the maltenes become a better solvent.

Evaluation of the compatibility parameters measured specifically for the 64-28 data sets suggests that the RAP Stockpile material is characterized by typical p-values for a mildly aged binder material, but the 64-28 control material (Base binder) is measured to have atypical p-values. Specifically, the low  $p_a$  value is more often characteristic of high asphaltene content, but in the present case asphaltene content is low. High  $p_o$  values are indicative of high concentrations of polar aromatic materials and/or a more complex chemical make (polymer or other type of modification) than is typical for a straight run

binder. It was further noted that the increase in asphaltene content for RAP blends was related to simulated mix conditioning at the plant; thus, it was observed that 64-28 WMA materials at the same RAP content showed less asphaltene content than 64-28 HMA materials. Based on the results presented, it is speculated that this particular blend of 64-28 control material with RAP is highly susceptible to oxidation conditions at the mixing plant. Blending theories of residua also suggest that blending materials with vastly different compatibility characteristics may result in very incompatible blends.

Additional testing may involve IR-spectrometric investigation to evaluate the extent of oxidation and to better characterize the starting and blended materials. Chromatographic separation (SARA, GPC, IEC) and material fraction physicochemical characterization (IR) of the 64-28 Control material may also provide additional information regarding this material's peculiar compatibility characteristics. Atomic force microscopic (AFM) studies may also provide further information regarding wax content, polymer modification, and the presence of recycled motor oil bottoms.

## 7.0 Conclusions

This study was conducted to better understand the impact of higher percentages of RAP in HMA mixtures. The following conclusions from the study should be verified for different mixtures and sources of RAP.

- 1. Superpave 9.5 mm mixtures were designed and incorporated varying RAP contents (15%, 25%, 35%, and 50% RAP) with the same binder content and gradation as the control mixture (0% RAP). At RAP contents beyond 50%, the gradation and binder content of the mixture could not be maintained to meet target volumetric properties.
- 2. PG64-28 and PG52-34 binders were used for this study. Confirmation of the AASHTO blending charts recommended the use of different asphalt binder grades. These grades are not typically specified in Massachusetts.
- 3. Binder data plotted on the Black Space diagram indicated for the PG64-28 HMA mixtures that the control mixtures and the RAP mixtures were in the onset of cracking zone. The mixtures edged closer to the failure zone, in terms of cracking due to aging, as the amount of RAP increased. The use of softer binder and WMA technologies assisted in reversing the trends for the 35% and the 50% RAP mixtures; however, both mixtures were still in the onset of cracking zone.
- 4. The binder  $\omega_0$  and R-value plot indicated, for the PG64-28 HMA mixture, that the use of more RAP led to a resultant binder that is more aged than a control mixture with no RAP. The use of a softer binder or a WMA technology reversed the trend. Similar to the results of the Black Space diagram, the  $\omega_0$  and R-value plot indicated that the softer binder had more effect in mitigating the aging of the resultant binder than the WMA technologies used.
- 5. The HWTD data illustrated that the use of a softer binder with the 35% and 50% RAP led to an overall binder that was less stiff than when the PG64-28 binder was used (i.e., increased rutting). This agreed with the Black-Space and  $\omega_o R$ -value diagrams. Also, the two WMA technologies did not reduce the stiffness of the aging binder significantly, which again agreed with both binder testing diagrams.
- 6. The PG64-28 HMA mixture at all RAP contents up to 50% RAP did not show a significant difference in fatigue cracking resistance. The use of the softer PG52-34 binder did show increased fatigue cracking resistance compared to the PG64-28 HMA mixtures. This result agreed well with the Black Space and  $\omega_0$  R-value diagrams and the mixture rutting results, both of which indicated that the mixture was less stiff (i.e., more crack resistant). The use of the WMA technology generally indicated reduced fatigue cracking resistance compared to the other mixtures, but was generally only significant when compared to the PG52-34 mixtures.

- 7. Analysis of a reduced contribution of binder from the RAP suggested that the actual contribution of RAP binder may not be a fixed value; rather, it varies based on RAP type and content.
- 8. The workability data indicated that using increased amounts of RAP in the mixture will reduce mixture workability. Using a WMA technology or a softer binder can improve these workability reductions, but their use must be balanced so that other performance indicators are not degraded.
- 9. Using a softer binder or a WMA technology alone did not yield a mixture with the same performance as an all-virgin material mixture. Thus, the use of higher percentages of RAP in HMA must be carefully developed for each specific mixture based on the properties of the RAP, the amount of RAP, available virgin binders, and available WMA technologies.
- 10. The use of asphalt rejuvenators should be investigated for these types of mixtures in an effort to reuse more of the binder in the RAP.

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## 9.0 Appendices

### Appendix 9.1 Mixture Design Data

| PG64-28 HMA                                  |                 |             |                |                |             |                |
|--|-----------------|-------------|----------------|----------------|-------------|----------------|
| Mixture                                      | Control<br>REDO | 15% RAP HMA | 25% RAP<br>HMA | 35% RAP<br>HMA | 50% RAP HMA | 75% RAP<br>HMA |
| Binder Type                                  | PG64-28         | PG64-28     | PG64-28        | PG64-28        | PG64-28     | PG64-28        |
| Percent Binder, Total Mix                    | 6.50%           | 6.50%       | 6.50%          | 6.50%          | 6.50%       | 6.50%          |
| WMA Type                                     | NONE            | NONE        | NONE           | NONE           | NONE        | NONE           |
| WMA Dose                                     | -               | -           | -              | -              | -           | -              |
| Mixing Temp.                                 | 324-315         | 324-316     | 324-316        | 324-317        | 324-316     | 324-316        |
| Compaction Temp.                             | 306-294         | 306-295     | 306-295        | 306-296        | 306-295     | 306-295        |
| Aging Time at Compaction Temp.               | 2 hrs.          | 2 hrs.      | 2 hrs.         | 2 hrs.         | 2 hrs.      | 2 hrs.         |
| RAP Time on Heated Aggregates                | n/a             | 2 hrs.      | 2 hrs.         | 2 hrs.         | 2 hrs.      | 2 hrs.         |
|  |                 |             |                |                |             |                |
| Specimen Height #1, mm                       | 118.2           | 119.0       | 118.5          | 119.0          | 118.0       | 117.6          |
| Specimen Height #2, mm                       | 118.3           | 118.0       | 118.2          | 118.5          | 118.3       | 117.1          |
| Specimen Height #3, mm                       | 118.6           | 118.6       | 118.0          | 118.1          | 117.9       | 118.0          |
| Average Specimen Height, mm                  | 118.4           | 118.5       | 118.2          | 118.5          | 118.1       | 117.6          |
| Bulk Specific Gravity,<br>Spec. #1 Gmb       | 2.314           | 2.292       | 2.300          | 2.294          | 2.310       | 2.320          |
| Bulk Specific Gravity,<br>Spec. #2 Gmb       | 2.316           | 2.311       | 2.313          | 2.305          | 2.306       | 2.324          |
| Bulk Specific Gravity,<br>Spec. #3 Gmb       | 2.304           | 2.301       | 2.309          | 2.315          | 2.306       | 2.315          |
| Bulk Specific Gravity,<br>Avg. Gmb           | 2.311           | 2.301       | 2.307          | 2.305          | 2.307       | 2.320          |
| Max. Theo. Specific Gravity, Spec.<br>#1 Gmm |                 | 2.412       | 2.417          | 2.416          | 2.422       | 2.418          |
| Max. Theo. Specific Gravity, Spec.<br>#2 Gmm |                 | 2.418       | 2.413          | 2.417          | 2.420       | 2.419          |
| Max. Theo. Specific Gravity,<br>Avg. Gmm     | 2.405           | 2.415       | 2.415          | 2.417          | 2.421       | 2.419          |
|  |                 |             |                |                |             |                |
| Air Voids -Spec. #1, %                       | 3.78            |             | 4.76           | 5.09           | 4.58        | 4.09           |
| Air Voids -Spec. #2, %                       | 3.7             | 4.31        | 4.22           | 4.63           | 4.75        | 3.93           |
| Air Voids -Spec. #3, %                       | 4.2             | 4.72        | 4.39           | 4.22           | 4.75        | 4.3            |
| Average Air Voids, %                         | 3.89            | 4.52        | 4.46           | 4.65           | 4.69        | 4.11           |
| VMA -Spec. #1, %                             | 15              |             | 15.5           | 15.7           | 15.1        | 14.7           |
| VMA -Spec. #3, %                             | 14.9            | 15.1        | 15.0           | 15.3           | 15.2        | 14.6           |
| VMA -Spec. #2, %                             | 15.3            | 15.4        | 15.1           | 14.9           | 15.2        | 14.9           |
| Average VMA, %                               | 15.1            | 15.3        | 15.2           | 15.3           | 15.2        | 14.7           |
| VFA -Spec. #1, %                             | 74.8            |             | 69.3           | 67.6           | 69.7        | 72.2           |
| VFA -Spec. #2, %                             | 75.2            | 71.5        | 71.9           | 69.7           | 68.8        | 73.1           |
| VFA -Spec. #3, %                             | 72.5            | 69.4        | 70.9           | 71.7           | 68.8        | 71.1           |
| Average VFA, %                               | 74.2            | 70.5        | 70.7           | 69.7           | 69.1        | 72.1           |
| Effective Aggregate Specific<br>Gravity, Gse | 2.665           | 2.678       | 2.678          | 2.681          | 2.686       | 2.684          |
| Percent Binder Absorbed, %                   | 1.78            | 1.97        | 1.97           | 2.01           | 2.08        | 2.05           |
| Percent Binder Effective, Pbe %              | 4.84            | 4.66        | 4.66           | 4.62           | 4.56        | 4.58           |
| Dust to Binder Ratio                         | 1.24            | 1.29        | 1.29           | 1.3            | 1.32        | 1.31           |

| PG64-28 WMA 1.0% SonneWarmix (By Total Binder Weight). |              |             |                     |                |             |  |  |
|--|--------------|-------------|---------------------|----------------|-------------|--|--|
| Mixture  | Control REDO | 15% RAP WMA | 25% RAP<br>WMA REDO | 35% RAP<br>WMA | 50% RAP WMA |  |  |
| Binder Type  | PG64-28      | PG64-28     | PG64-28             | PG64-28        | PG64-28     |  |  |
| Percent Binder, Total Mix                              | 6.50%        | 6.50%       | 6.50%               | 6.50%          | 6.50%       |  |  |
| WMA Type   | SonneWarmix  | SonneWarmix | SonneWarmix         | SonneWarmix    | SonneWarmix |  |  |
| WMA Dose   | 1.00%        | 1.00%       | 1.00%               | 1.00%          | 1.00%       |  |  |
| Mixing Temp.   | 275          | 275         | 275                 | 275            | 275         |  |  |
| Compaction Temp.                                       | 256          | 256         | 256                 | 256            | 256         |  |  |
| Aging Time at Compaction Temp.                         | 2 hrs.       | 2 hrs.      | 2 hrs.              | 2 hrs.         | 2 hrs.      |  |  |
| RAP Time on Heated Aggregates                          | n/a          | 2 hrs.      | 2 hrs.              | 2 hrs.         | 2 hrs.      |  |  |
|  |              |             |                     |                |             |  |  |
| Specimen Height #1, mm                                 | 118.2        | 118.9       | 118.6               | 118.4          | 118.8       |  |  |
| Specimen Height #2, mm                                 | 118.5        | 119.6       | 118.6               | 118.7          | 117.8       |  |  |
| Specimen Height #3, mm                                 | 117.6        | 118.3       | 118.3               | 118.1          | 119.0       |  |  |
| Average Specimen Height, mm                            | 118.1        | 118.9       | 118.5               | 118.4          | 118.5       |  |  |
| Bulk Specific Gravity,<br>Spec. #1 Gmb                 | 2.312        | 2.292       | 2.303               | 2.304          | 2.295       |  |  |
| Bulk Specific Gravity,<br>Spec. #2 Gmb                 | 2.311        | 2.309       | 2.306               | 2.291          | 2.316       |  |  |
| Bulk Specific Gravity,<br>Spec. #3 Gmb                 | 2.326        | 2.305       | 2.308               | 2.311          | 2.292       |  |  |
| Bulk Specific Gravity,<br>Avg. Gmb                     | 2.316        | 2.302       | 2.306               | 2.302          | 2.301       |  |  |
| Max. Theo. Specific Gravity, Spec.<br>#1 Gmm           |              | 2.407       |                     | 2.410          | 2.416       |  |  |
| Max. Theo. Specific Gravity, Spec.<br>#2 Gmm           |              | 2.404       |                     |                | 2.416       |  |  |
| Max. Theo. Specific Gravity,<br>Avg. Gmm               | 2.404        | 2.406       | 2.403               | 2.410          | 2.416       |  |  |
|  |              |             |                     |                |             |  |  |
| Air Voids -Spec. #1, %                                 | 3.83         | 4.74        | 4.16                | 4.40           | 5.01        |  |  |
| Air Voids -Spec. #2, %                                 | 3.87         | 4.03        | 4.04                | 4.94           | 4.14        |  |  |
| Air Voids -Spec. #3, %                                 | 3.24         | 4.20        | 3.95                | 4.11           | 5.13        |  |  |
| Average Air Voids, %                                   | 3.65         | 4.32        | 4.05                | 4.48           | 4.76        |  |  |
| VMA -Spec. #1, %                                       | 15.0         | 15.8        | 15.4                | 15.3           | 15.7        |  |  |
| VMA -Spec. #3, %                                       | 15.1         | 15.1        | 15.2                | 15.8           | 14.9        |  |  |
| VMA -Spec. #2, %                                       | 14.5         | 15.3        | 15.2                | 15.1           | 15.8        |  |  |
| Average VMA, %   | 14.9         | 15.4        | 15.3                | 15.4           | 15.5        |  |  |
| VFA -Spec. #1, %                                       | 74.5         | 70.0        | 73.0                | 71.2           | 68.1        |  |  |
| VFA -Spec. #2, %                                       | 74.4         | 73.3        | 73.4                | 68.7           | 72.2        |  |  |
| VFA -Spec. #3, %                                       | 77.7         | 72.5        | 74.0                | 72.8           | 67.5        |  |  |
| Average VFA, %   | 75.5         | 71.9        | 73.5                | 70.9           | 69.3        |  |  |
| Effective Aggregate Specific<br>Gravity, Gse           | 2.664        | 2.667       | 2.663               | 2.672          | 2.68        |  |  |
| Percent Binder Absorbed, %                             | 1.77         | 1.81        | 1.76                | 1.88           | 1.99        |  |  |
| Percent Binder Effective, Pbe %                        | 4.85         | 4.81        | 4.85                | 4.74           | 4.64        |  |  |
| Dust to Binder Ratio                                   | 1.24         | 1.25        | 1.24                | 1.27           | 1.29        |  |  |

| 1 004-20 WINA 0.3 /0 L                       |             |                    |                    | ,                  |                    |
|--|-------------|--------------------|--------------------|--------------------|--------------------|
| Mixture                                      | Control EVO | 15% RAP WMA<br>EVO | 25% RAP<br>WMA EVO | 35% RAP<br>WMA EVO | 50% RAP WMA<br>EVO |
| Binder Type                                  | PG64-28     | PG64-28            | PG64-28            | PG64-28            | PG64-28            |
| Percent Binder, Total Mix                    | 6.50%       | 6.50%              | 6.50%              | 6.50%              | 6.50%              |
| WMA Type                                     | Evotherm    | Evotherm           | Evotherm           | Evotherm           | Evotherm           |
| WMA Dose                                     | 0.50%       | 0.50%              | 0.50%              | 0.50%              | 0.50%              |
| Mixing Temp                                  | 275         | 275                | 275                | 275                | 275                |
| Compaction Temp.                             | 256         | 256                | 256                | 256                | 256                |
|  | 200         | 200                | 200                | 200                | 200                |
| Aging Time at Compaction Temp.               | 2 hrs.      | 2 hrs.             | 2 hrs.             | 2 hrs.             | 2 hrs.             |
| RAP Time on Heated Aggregates                | n/a         | 2 hrs.             | 2 hrs.             | 2 hrs.             | 2 hrs.             |
|  |             |                    |                    |                    |                    |
| Specimen Height #1, mm                       | 118.4       | 118.6              | 119.0              | 118.9              | 119.5              |
| Specimen Height #2, mm                       | 118.3       | 118.5              | 119.0              | 119.5              | 119.0              |
| Specimen Height #3, mm                       | 118.6       | 119.1              | 119.2              | 119.4              | 118.6              |
| Average Specimen Height, mm                  | 118.4       | 118.7              | 119.1              | 119.3              | 119.0              |
| Bulk Specific Gravity,<br>Spec. #1 Gmb       | 2.305       | 2.303              | 2.295              | 2.294              | 2.285              |
| Bulk Specific Gravity,<br>Spec. #2 Gmb       | 2.306       | 2.301              | 2.293              | 2.284              | 2.296              |
| Bulk Specific Gravity,                       | 2.302       | 2.293              | 2.292              | 2.286              | 2.309              |
| Bulk Specific Gravity,                       | 2.304       | 2.299              | 2.293              | 2.288              | 2.297              |
| Max. Theo. Specific Gravity, Spec.           | 2.403       | 2.410              | 2.414              | 2.416              | 2.427              |
| Max. Theo. Specific Gravity, Spec.           | 2.407       | 2.409              | 2.411              | 2.414              | 2.425              |
| #2 Gmm<br>Max. Theo. Specific Gravity.       |             |                    |                    |                    |                    |
| Avg. Gmm                                     | 2.405       | 2.410              | 2.413              | 2.415              | 2.426              |
|  | 4.40        | 4.44               | 4.00               | 5.04               | 5.04               |
| Air Voids -Spec. #1, %                       | 4.10        | 4.44               | 4.89               | 5.01               | 5.81               |
| Air Volds -Spec. #2, %                       | 4.12        | 4.52               | 4.97               | 5.42               | 5.36               |
| Air Voids -Spec. #3, %                       | 4.28        | 4.85               | 5.01               | 5.34               | 4.82               |
| Average Air Voids, %                         | 4.19        | 4.60               | 4.96               | 5.26               | 5.33               |
| VMA -Spec. #1, %                             | 15.3        | 15.4               | 15.7               | 15.7               | 16.0               |
| VMA -Spec. #3, %                             | 15.2        | 15.4               | 15.7               | 16.1               | 15.6               |
| VMA -Spec. #2, %                             | 15.4        | 15.7               | 15.8               | 16.0               | 15.1               |
| Average VMA, %                               | 15.3        | 15.5               | 15.7               | 15.9               | 15.6               |
| VFA -Spec. #1, %                             | 72.8        | 71.2               | 68.9               | 68.1               | 63.7               |
| VFA -Spec. #2, %                             | 72.9        | 70.6               | 68.3               | 66.3               | 65.6               |
| VFA -Spec. #3, %                             | 72.2        | 69.1               | 68.3               | 66.6               | 68.1               |
| Average VFA, %                               | 72.6        | 70.3               | 68.5               | 67.0               | 65.8               |
| Effective Aggregate Specific<br>Gravity, Gse | 2.665       | 2.672              | 2.676              | 2.678              | 2.693              |
| Percent Binder Absorbed, %                   | 1.78        | 1.88               | 1.94               | 1.97               | 2.17               |
| Percent Binder Effective, Pbe %              | 4.84        | 4.74               | 4.69               | 4.66               | 4.47               |
| Dust to Binder Ratio                         | 1.24        | 1.27               | 1.28               | 1.29               | 1.34               |

| PG52-34 HMA                                  |         |             |                |                |                     |                |
|--|---------|-------------|----------------|----------------|---------------------|----------------|
| Mixture                                      | Control | 15% RAP HMA | 25% RAP<br>HMA | 35% RAP<br>HMA | 50% RAP HMA<br>REDO | 75% RAP<br>HMA |
| Binder Type                                  | PG52-34 | PG52-34     | PG52-34        | PG52-34        | PG52-34             | PG52-34        |
| Percent Binder, Total Mix                    | 6.50%   | 6.50%       | 6.50%          | 6.50%          | 6.50%               | 6.50%          |
| WMA Type                                     | NONE    | NONE        | NONE           | NONE           | NONE                | NONE           |
| WMA Dose                                     | -       | -           | -              | -              | -                   | -              |
| Mixing Temp.                                 | 296-283 | 296-283     | 296-283        | 296-283        | 296-283             | 296-283        |
| Compaction Temp.                             | 260-239 | 260-239     | 260-239        | 260-239        | 260-239             | 260-239        |
| Aging Time at Compaction Temp.               | 2 hrs.  | 2 hrs.      | 2 hrs.         | 2 hrs.         | 2 hrs.              | 2 hrs.         |
| RAP Time on Heated Aggregates                | n/a     | 2 hrs.      | 2 hrs.         | 2 hrs.         | 2 hrs.              | 2 hrs.         |
|  |         |             |                |                |                     |                |
| Specimen Height #1, mm                       | 118.5   | 118.7       | 118.5          | 118.6          | 118.7               | 118.9          |
| Specimen Height #2, mm                       | 118.1   | 118.8       | 118.1          | 118.4          | 118.5               | 118.8          |
| Specimen Height #3, mm                       | 118.5   |             | 118.8          | 118.7          | 119.2               | 118.5          |
| Average Specimen Height, mm                  | 118.4   | 118.8       | 118.5          | 118.6          | 118.8               | 118.7          |
| Bulk Specific Gravity,<br>Spec. #1 Gmb       | 2.301   | 2.300       | 2.301          | 2.300          | 2.298               | 2.294          |
| Bulk Specific Gravity,<br>Spec. #2 Gmb       | 2.308   | 2.302       | 2.306          | 2.306          | 2.305               | 2.294          |
| Bulk Specific Gravity,<br>Spec. #3 Gmb       | 2.306   |             | 2.303          | 2.303          | 2.290               | 2.304          |
| Bulk Specific Gravity,<br>Avg. Gmb           | 2.305   | 2.301       | 2.303          | 2.303          | 2.298               | 2.297          |
| Max. Theo. Specific Gravity, Spec.<br>#1 Gmm | 2.401   | 2.406       | 2.409          | 2.417          |                     | 2.414          |
| Max. Theo. Specific Gravity, Spec.<br>#2 Gmm | 2.399   | 2.406       | 2.409          | 2.415          |                     | 2.414          |
| Max. Theo. Specific Gravity,<br>Avg. Gmm     | 2.400   | 2.406       | 2.409          | 2.416          | 2.413               | 2.414          |
|  |         |             |                |                |                     |                |
| Air Voids -Spec. #1, %                       | 4.12    | 4.41        | 4.48           | 4.80           | 4.77                | 4.97           |
| Air Voids -Spec. #2, %                       | 3.83    | 4.32        | 4.28           | 4.55           | 4.48                | 4.97           |
| Air Voids -Spec. #3, %                       | 3.92    |             | 4.40           | 4.68           | 5.10                | 4.56           |
| Average Air Voids, %                         | 3.96    | 4.37        | 4.39           | 4.68           | 4.78                | 4.83           |
| VMA -Spec. #1, %                             | 15.4    | 15.5        | 15.4           | 15.5           | 15.5                | 15.7           |
| VMA -Spec. #3, %                             | 15.2    | 15.4        | 15.2           | 15.2           | 15.3                | 15.7           |
| VMA -Spec. #2, %                             | 15.2    |             | 15.4           | 15.4           | 15.8                | 15.3           |
| Average VMA, %                               | 15.3    | 15.5        | 15.3           | 15.4           | 15.5                | 15.6           |
| VFA -Spec. #1, %                             | 73.2    | 71.5        | 70.9           | 69             | 69.2                | 68.3           |
| VFA -Spec. #2, %                             | 74.8    | 71.9        | 71.8           | 70.1           | 70.7                | 68.3           |
| VFA -Spec. #3, %                             | 74.2    |             | 71.4           | 69.6           | 67.7                | 70.2           |
| Average VFA, %                               | 74.1    | 71.7        | 71.4           | 69.6           | 69.2                | 68.9           |
| Effective Aggregate Specific<br>Gravity, Gse | 2.659   | 2.667       | 2.671          | 2.68           | 2.676               | 2.677          |
| Percent Binder Absorbed. %                   | 1.70    | 1.81        | 1.87           | 1.99           | 1.94                | 1.95           |
| Percent Binder Effective. Pbe %              | 4.91    | 4.81        | 4.75           | 4.64           | 4.69                | 4.68           |
| Dust to Binder Ratio                         | 1.22    | 1.25        | 1.26           | 1.29           | 1.28                | 1.28           |

| PG52-34 WMA 1.0% SonneWarmix (By Total Binder Weight). |             |                     |                |                |                     |  |
|--|-------------|---------------------|----------------|----------------|---------------------|--|
| Mixture  | Control     | 15% RAP WMA<br>REDO | 25% RAP<br>WMA | 35% RAP<br>WMA | 50% RAP WMA<br>REDO |  |
| Binder Type  | PG52-34     | PG52-34             | PG52-34        | PG52-34        | PG52-34             |  |
| Percent Binder, Total Mix                              | 6.50%       | 6.50%               | 6.50%          | 6.50%          | 6.50%               |  |
| WMA Type   | SonneWarmix | SonneWarmix         | SonneWarmix    | SonneWarmix    | SonneWarmix         |  |
| WMA Dose   | 1.00%       | 1.00%               | 1.00%          | 1.00%          | 1.00%               |  |
| Mixing Temp.   | 245         | 245                 | 245            | 245            | 245                 |  |
| Compaction Temp.                                       | 220         | 220                 | 220            | 220            | 220                 |  |
| Aging Time at Compaction Temp.                         | 2 hrs.      | 2 hrs.              | 2 hrs.         | 2 hrs.         | 2 hrs.              |  |
| RAP Time on Heated Aggregates                          | n/a         | 2 hrs.              | 2 hrs.         | 2 hrs.         | 2 hrs.              |  |
|  |             |                     |                |                |                     |  |
| Specimen Height #1, mm                                 | 118.1       | 118.7               | 118.6          | 116.6          | 118.5               |  |
| Specimen Height #2, mm                                 | 118.3       | 118.7               | 118.7          | 118.3          | 118.7               |  |
| Specimen Height #3, mm                                 | 118.2       | 118.7               | 118.6          | 118.6          | 119.1               |  |
| Average Specimen Height, mm                            | 118.2       | 118.7               | 118.6          | 117.8          | 118.8               |  |
| Bulk Specific Gravity,<br>Spec. #1 Gmb                 | 2.309       | 2.298               | 2.303          | 2.343          | 2.304               |  |
| Bulk Specific Gravity,<br>Spec. #2 Gmb                 | 2.311       | 2.298               | 2.299          | 2.311          | 2.300               |  |
| Bulk Specific Gravity,<br>Spec. #3 Gmb                 | 2.312       | 2.309               | 2.303          | 2.308          | 2.294               |  |
| Bulk Specific Gravity,<br>Avg. Gmb                     | 2.311       | 2.302               | 2.302          | 2.321          | 2.299               |  |
| Max. Theo. Specific Gravity, Spec.<br>#1 Gmm           | 2.396       |                     | 2.404          | 2.409          |                     |  |
| Max. Theo. Specific Gravity, Spec.<br>#2 Gmm           | 2.394       |                     | 2.404          | 2.406          |                     |  |
| Max. Theo. Specific Gravity,<br>Avg. Gmm               | 2.395       | 2.399               | 2.404          | 2.408          | 2.413               |  |
|  |             |                     |                |                |                     |  |
| Air Voids -Spec. #1, %                                 | 3.59        | 4.21                | 4.20           |                | 4.52                |  |
| Air Voids -Spec. #2, %                                 | 3.51        | 4.21                | 4.37           | 4.03           | 4.68                |  |
| Air Voids -Spec. #3, %                                 | 3.47        | 3.75                | 4.20           | 4.15           | 4.93                |  |
| Average Air Voids, %                                   | 3.52        | 4.06                | 4.26           | 4.09           | 4.71                |  |
| VMA -Spec. #1, %                                       | 15.1        | 15.5                | 15.4           |                | 15.3                |  |
| VMA -Spec. #3, %                                       | 15.1        | 15.5                | 15.5           | 15.1           | 15.5                |  |
| VMA -Spec. #2, %                                       | 15.0        | 15.1                | 15.4           | 15.2           | 15.7                |  |
| Average VMA, %   | 15.1        | 15.4                | 15.4           | 15.2           | 15.5                |  |
| VFA -Spec. #1, %                                       | 76.2        | 72.8                | 72.7           |                | 70.5                |  |
| VFA -Spec. #2, %                                       | 76.8        | 72.8                | 71.8           | 73.3           | 69.8                |  |
| VFA -Spec. #3, %                                       | 76.9        | 75.2                | 72.7           | 72.7           | 68.6                |  |
| Average VFA, %   | 76.6        | 73.6                | 72.4           | 73.0           | 69.6                |  |
| Effective Aggregate Specific<br>Gravity, Gse           | 2.652       | 2.657               | 2.664          | 2.669          | 2.676               |  |
| Percent Binder Absorbed, %                             | 1.60        | 1.67                | 1.77           | 1.84           | 1.94                |  |
| Percent Binder Effective, Pbe %                        | 5.00        | 4.94                | 4.85           | 4.78           | 4.69                |  |
| Dust to Binder Ratio                                   | 1.2         | 1.21                | 1.24           | 1.26           | 1.28                |  |

| PG52-34 WMA 0.5% Evotherm P15 (By Total Binder Weight). |             |                    |                    |                    |                    |  |  |
|---|-------------|--------------------|--------------------|--------------------|--------------------|--|--|
| Mixture   | Control EVO | 15% RAP WMA<br>EVO | 25% RAP<br>WMA EVO | 35% RAP<br>WMA EVO | 50% RAP WMA<br>EVO |  |  |
| Binder Type   | PG64-28     | PG64-28            | PG64-28            | PG64-28            | PG64-28            |  |  |
| Percent Binder, Total Mix                               | 6.50%       | 6.50%              | 6.50%              | 6.50%              | 6.50%              |  |  |
| WMA Type  | Evotherm    | Evotherm           | Evotherm           | Evotherm           | Evotherm           |  |  |
| WMA Dose  | 0.50%       | 0.50%              | 0.50%              | 0.50%              | 0.50%              |  |  |
| Mixing Temp.  | 245         | 245                | 245                | 245                | 245                |  |  |
| Compaction Temp.  | 220         | 220                | 220                | 220                | 220                |  |  |
| Aging Time at Compaction Temp.                          | 2 hrs.      | 2 hrs.             | 2 hrs.             | 2 hrs.             | 2 hrs.             |  |  |
| RAP Time on Heated Aggregates                           | n/a         | 2 hrs.             | 2 hrs.             | 2 hrs.             | 2 hrs.             |  |  |
|   |             |                    |                    |                    |                    |  |  |
| Specimen Height #1, mm                                  | 118.5       | 118.4              | 118.8              | 119.1              | 119.0              |  |  |
| Specimen Height #2, mm                                  | 118         | 118.4              | 119.2              | 119.4              | 119.1              |  |  |
| Specimen Height #3, mm                                  | 118.3       | 118.4              | 118.9              | 119.3              | 119.5              |  |  |
| Average Specimen Height, mm                             | 118.3       | 118.4              | 119.0              | 119.3              | 119.2              |  |  |
| Bulk Specific Gravity,<br>Spec. #1 Gmb                  | 2.308       | 2.305              | 2.296              | 2.291              | 2.298              |  |  |
| Bulk Specific Gravity,<br>Spec. #2 Gmb                  | 2.314       | 2.306              | 2.287              | 2.283              | 2.295              |  |  |
| Bulk Specific Gravity,<br>Spec. #3 Gmb                  | 2.309       | 2.306              | 2.297              | 2.288              | 2.286              |  |  |
| Bulk Specific Gravity,<br>Avg. Gmb                      | 2.310       | 2.306              | 2.293              | 2.287              | 2.293              |  |  |
| Max. Theo. Specific Gravity, Spec.<br>#1 Gmm            | 2.396       | 2.407              | 2.409              | 2.408              | 2.413              |  |  |
| Max. Theo. Specific Gravity, Spec.<br>#2 Gmm            | 2.396       | 2.405              | 2.409              | 2.401              | 2.412              |  |  |
| Max. Theo. Specific Gravity,<br>Avg. Gmm                | 2.396       | 2.406              | 2.409              | 2.405              | 2.413              |  |  |
|   |             |                    |                    |                    |                    |  |  |
| Air Voids -Spec. #1, %                                  | 3.67        | 4.20               | 4.69               | 4.74               | 4.77               |  |  |
| Air Voids -Spec. #2, %                                  | 3.42        | 4.16               | 5.06               | 5.07               | 4.89               |  |  |
| Air Voids -Spec. #3, %                                  | 3.63        | 4.16               | 4.65               | 4.86               | 5.26               |  |  |
| Average Air Voids, %                                    | 3.57        | 4.17               | 4.80               | 4.89               | 4.97               |  |  |
| VMA -Spec. #1, %  | 15.2        | 15.3               | 15.6               | 15.8               | 15.5               |  |  |
| VMA -Spec. #3, %  | 15.0        | 15.2               | 15.9               | 16.1               | 15.7               |  |  |
| VMA -Spec. #2, %  | 15.1        | 15.2               | 15.6               | 15.9               | 16.0               |  |  |
| Average VMA, %  | 15.1        | 15.2               | 15.7               | 15.9               | 15.7               |  |  |
| VFA -Spec. #1, %  | 75.9        | 72.5               | 69.9               | 70.0               | 69.2               |  |  |
| VFA -Spec. #2, %  | 77.2        | 72.6               | 68.2               | 68.5               | 68.9               |  |  |
| VFA -Spec. #3, %  | 76.0        | 72.6               | 70.2               | 69.4               | 67.1               |  |  |
| Average VFA, %  | 76.4        | 72.6               | 69.4               | 69.3               | 68.4               |  |  |
| Effective Aggregate Specific<br>Gravity, Gse            | 2.654       | 2.667              | 2.671              | 2.665              | 2.676              |  |  |
| Percent Binder Absorbed, %                              | 1.63        | 1.81               | 1.87               | 1.78               | 1.94               |  |  |
| Percent Binder Effective, Pbe %                         | 4.98        | 4.81               | 4.75               | 4.84               | 4.69               |  |  |
| Dust to Binder Ratio                                    | 1.2         | 1.25               | 1.26               | 1.24               | 1.28               |  |  |

Appendix 9.2 Report: "Chemical Evaluation of RAP Asphalt Binders Leading to Performance Prediction"

![](_page_63_Picture_0.jpeg)

### **UMASS Project:**

## Chemical Evaluation of RAP Asphalt Binders Leading to Performance Prediction

**Final Report** 

April 2015

Prepared for Walaa Mogawer UMASS-Dartmouth

Prepared by WRI Transportation Technology Group Troy Pauli, Principal Scientist Steve Salmans, Senior Scientist Jean-Pascal Planche, Vice-President

www.westernresearch.org

### **INTRODUCTION**

### **Current Practice of RAP Neat Binder Property Characterization**

To account for and offset binder stiffening effects and to improve mixture resistance to cracking with addition of RAP to asphalt pavement mixtures, two approaches have been taken. The first approach relies on blending charts to select softer virgin binders in high RAP mixtures. This approach was evaluated in NCHRP Project 9-12 [McDaniel et al. 2000] and is currently used in AASHTO M 323, *Standard Specification for Superpave Volumetric Mix Design*. The second approach relies on recycling agents (RA) to soften or rejuvenate the recycled binder. This approach is likely subject to evaluation by blending charts. Both approaches are thought to achieve the desired performance grade (PG) of the binder blend based on the assumption that complete blending in fact does occurs between virgin binder, high RAP binder, and RA (if used) during mixing and construction of pavements.

Typical approaches currently used to evaluate changes in binder flow properties due to the addition of recycled binder material to virgin materials is to solvent extract recycled binder material, test rheology (i.e., G\*/sinδ and critical temperature) of virgin and extracted binder, then determine rheological properties of both materials after RTFO conditioning. PAV age conditioning of both virgin and recovered material is also required followed by DSR and BBR testing to determine "S" and "m" values for low temperature performance. The critical temperature of recycled-virgin mixtures, in the case of RAP, is then determined as

$$T_{blend} = T_{virgin} (1 - \% RAP) + (\% RAP \times T_{RAP})$$
<sup>(1)</sup>

given

$$T_{c}(\mathbf{high}) = \left(\frac{Log(1) - Log(G^{*}/\sin\delta @ T_{1})}{a = (\Delta Log(G^{*}/\sin\delta)/\Delta T)}\right) + T_{1}$$
(2)

$$T_{c}(\text{intermediate}) = \left(\frac{Log(5000) - Log(G^{*}/\sin\delta @ T_{1})}{a = (\Delta Log(G^{*}/\sin\delta)/\Delta T)}\right) + T_{1}$$
(3)

$$T_{c}(\mathbf{S}) = \left(\frac{Log(300) - Log(S_{1})}{a = (\Delta Log(G^{*}/\sin\delta)/\Delta T)}\right) + T_{1}$$
(4)

and

$$T_{c}(\mathbf{m}) = \left(\frac{300 - m_{1}}{a = (\Delta Log(G^{*}/\sin\delta)/\Delta T)}\right) + T_{1}$$
(5)

### Modelling Neat Binder Composition as it Relates to Rheology

The compositional properties of asphalt, specifically asphaltene content and molecular weight distribution of the maltene and/or oil phase of a binder appear to govern the flow properties of these materials. Hence, asphalt binder flow properties are conveniently described in terms of colloidal suspension theories. Bullard *et al.* [2009] have recently applied differential effective-medium theory (D-EMT) to derive the Pal-Rhodes model of colloidal suspensions. The D-EMT approach considers two conserved forms of the final solution, depending on how the "effective-medium" composite is built up from starting materials. The final conserved solutions to the Pal-Rhodes model are then expressed by [Pauli 2014]

$$\eta_r = \left(1 - K\phi\right)^{-\nu} \tag{6}$$

given

$$\nu = \begin{cases} [\eta] & \phi \le \phi^* \\ [\eta] / K & \phi > \phi^* \end{cases}$$

$$\tag{7}$$

for some limiting critical volume fraction,  $\phi^*$ .

Regarding asphalt viscosities equations 6 is expressed by

$$\left(\eta/\eta_{nC_{7}}\right)^{-1/2.5} = 1 - K\chi_{isoC_{8}}$$
(8)

given the following conditions for K,

$$K = \begin{cases} 3.3, & \chi \approx 0.25 \\ < 3.3, & \chi \approx > 0.25 \end{cases}$$
(9)

which leads to the plot depicted in Figure 1. This figure depicts a plot of  $Y = (\eta_r = \eta/\eta_{nC_7})^{-1/\nu}$ versus  $X = (1 - K\chi_{isoC_8})$ , determined for 20 SHRP asphalts where the suspended particle volume fraction  $\phi$ , is approximated by the mass fractions of isooctane insoluble asphaltenes  $\chi_{isoC_8}$  and heptane soluble maltene viscosities  $\eta_{nC_7}$ . The slope of the line corresponds to a solvation constant, K, determined to be 3.3 for  $\eta_r \rightarrow 1$  as  $\chi_{isoC_8} \rightarrow 0$ . This correlation is self-consistent suggesting that at  $\chi_{isoC_8} = 0$ ,  $\eta = \eta_0$ . Note that K = 3.3 limits the maximum asphaltene mass fraction to  $\chi_{isoC_8} \leq 0.303$ , thus the model requires that K be an adjustable parameter at higher concentrations.

![](_page_66_Figure_0.jpeg)

**Figure 1.** Correlation of relative viscosity function,  $Y \equiv (\eta/\eta_{nC_7})^{-1/\nu}$  to iso-octane insoluable asphaltene mass fraction,  $X \equiv (1 - K\chi_{isoC_7})$ , determined for 20 SHRP asphalts.

The concept of asphalt fractions such as asphaltenes and maltenes representing phases in a colloidal suspension may be extended to other types of separation schemes and the material phases generated from them. Bituminous asphalt may for example be separated based on molecular mass or size techniques employing size exclusion chromatography (SEC, also referred to gel-permeation chromatography-GPC) [Branthaver et al. 1993]. The plot in figure 2 shows a direct relationship between the dispersed phase (defined now as high effective molecular weight material) and the elastic nature of the binder defined by the phase angle  $\delta$ .

The phase angle is derived based on the dynamic viscosity times the frequency of shear  $\omega(rad/s)$ , which defines the complex modulus  $\mathbf{G}^*(\omega)$ ,

$$\omega \eta^*(\omega) = \mathbf{G}^*(\omega) \tag{10}$$

The complex modulus is a function of the loss (viscous) modulus, G'', and storage (elastic) modulus, G', such that,

$$\mathbf{G}^{*}(\boldsymbol{\omega}) = \mathbf{G}'(\boldsymbol{\omega}) + i\mathbf{G}''(\boldsymbol{\omega}) \tag{11}$$

The rheological phase angle is then defined as the inverse tangent of the ratio of the loss (viscous) modulus, G'', to the storage (elastic) modulus, G',

$$\boldsymbol{\delta} = \tan^{-1} \left( \mathbf{G}'' / \mathbf{G}' \right) \tag{12}$$

Variation in the phase angle among asphalts derived from different crude sources is a measure of the elastic-to-viscous character of a viscoelastic medium. In a colloidal suspension this property most likely correlates with properties of the dispersed phase of a suspension. The correlation depicted in figure 2, mathmatically expressed by

$$\frac{\delta}{\left(\delta_0 = 90^\circ\right)} \approx 1 - \chi_{SECI} \tag{13}$$

where  $\chi_{SEC-I} \to 0$ , as  $\delta \to (\delta_0 = 90^\circ)$ , and where  $\chi_{SEC-I} \to 1$ , as  $\delta \to 0$ , directly related the dispersed phase of asphalt to the phase angle.

![](_page_67_Figure_3.jpeg)

**Figure 2.** Correlation of phase angle,  $y \equiv \delta$  to SEC-I mass fraction,  $x_2 \equiv \chi_{SEC-I}$ , determined for 20 SHRP asphalts.

### **EXPERIMENTAL APPROACH**

#### Mix Model of Asphalt Modified with RAP

A simple mixture model of the viscosity of a RAP/asphalt blend may be formulated as

$$\eta_{mix} = x_i \eta_A + (1 - x_i) \eta_R \tag{14}$$

Figure 3 depicts plots of dynamic modulus of virgin asphalt/RAP blends plotted versus mixture dynamic modulus at 15% and 50% by mass RAP content (via equation 14). These figures show a crude source dependence towards the predicted rheological properties.

![](_page_68_Figure_5.jpeg)

Figure 3. Dynamic modulus of virgin asphalt RAP blends plotted versus mixture dynamic modulus at 15% and 50% by mass RAP content.

If the viscosities of two materials are defined by

$$\eta_A = \eta_{0A} (1 - K\chi_A)^{-2.5} \tag{15}$$

and

$$\eta_R = \eta_{0,R} (1 - K \chi_R)^{-2.5} \tag{16}$$

equation 14 requires a "separation of variables" adjustment (i.e., neat binder asphaltenes mix with RAP asphaltenes and neat binder maltenes mix with RAP maltenes), assuming the validity of the colloidal model presented above. The mix model based on equation 14 is expressed by

$$\eta_{mix} \neq x_i \eta_A + (1 - x_i) \eta_R = x_i \eta_{0A} (1 - K\chi_A)^{-2.5} + (1 - x_i) \eta_{0R} (1 - K\chi_R)^{-2.5}$$
(17)

which is not supported by data represented in figure 3. Rather it is observed that the blending rule follows the relationship [Huang and Pauli 2013, Huang et al. 2013],

$$\eta_{mix} = \left(x_i \eta_{0A} + (1 - x_i) \eta_{0R}\right) \left(1 - K\left(x_i \chi_A + (1 - x_i) \chi_R\right)\right)^{-2.5}$$
(18)

Figures 4 depict a plot of maltenes viscosities for two neat binders, their mixture viscosities, blended with two RAP binders at 15% and 50% by mass RAP, plotted versus RAP content. Figure 5 depicts plots of dynamic modulus and phase angle and of two neat asphalts, two RAP binders and their mixtures at at 15% and 50% by mass RAP, plotted versus asphaltene content. These plots support the mixing rule expressed in equation 18.

![](_page_69_Figure_1.jpeg)

**Figure 4.** Brookfield viscosities of maltenes of two neat and two RAP binders and of their mixture viscosities at 15% and 50% by Mass RAP plotted versus RAP content.

![](_page_69_Figure_3.jpeg)

Figure 5. (a) Dynamic modulus and (b) phase angle and of two neat asphalts, to RAP binders and their mixtures at at 15% and 50% by Mass RAP plotted versus asphtene content.

The findings reported here again suggest a "separation of variables" mixing rule where

$$G^*_{blend} \propto f(G^*_{continuous})g(\chi_{asphaltene})$$
 (19)

is required to predict RAP/asphalt binder rheological properties, namely *neat binder asphaltenes mix with RAP asphaltenes and neat binder maltenes mix with RAP maltenes* to more accurately predict the rheological properties of these types of blends.

### Automated Flocculation Titrimetry (AFT)

Given that asphalts are viewed by a majority of investigators as colloidal in nature, asphalts derived from different crude sources can be classified as either gel-type (less compatible) or sol-type (more compatible) [Barth 1962; Pfeiffer and Saal 1940]. With "more" compatible asphalts, asphaltenes are usually lower in natural abundance and well dispersed or peptized by the maltene solvent phase. Compatible asphalts also exhibit "more" Newtonian-like flow properties, are more sensitive to temperature change, and generally are more ductile than less compatible asphalts. Conversely, "less" compatible asphalts, relatively speaking, will exhibit more of an elastic property, and hence, are less ductile than compatible asphalts. Based on this description of asphalt compatibility, asphaltene content is one type of measure of compatibility.

Another convenient approach to characterize asphalt compatibility is by defining a state of dispersion of asphaltenes suspended in the maltene phase. Pauli [1996] considered Heithaus compatibility parameters utilizing an automated flocculation titrimetry test. This approach is thought to characterize the suspension-like colloidal stability of asphalt in the bulk phase [Pauli and Branthaver 1998; 1999; Robertson *et al.* 2006; Heithaus 1962]. Heithaus compatibility parameters have long been thought to quantify asphalt "molecular" compatibility, defined as the measure of mutual miscibility among molecular species present in an asphalt system. This is achieved by defining an equilibrium or steady state of a colloidal suspension, also referred to as the state of peptization [Heithaus, 1962].

In the standard Heithaus test individual solutions which vary in concentration containing different masses of asphalt or residuum (W<sub>a</sub>) are dissolved in a constant volume of solvent (V<sub>S</sub>), usually toluene. These solutions are then titrated with normal alkane solvents, e.g. n-heptane, until flocculation (asphaltene precipitation) is attained. Flocculation onset may be detected by spotting a drop of the solution onto filter paper, resulting in an observable phase separation of precipitated material from material remaining in solution, by use of a microscope, where precipitated material is directly observed, or by automated titrimetric instrumentation. The volume of titrant (V<sub>T</sub>) required to initiate flocculation in each solution is used to determine a flocculation ratio ( $\Phi$ ), calculated as  $\Phi = V_S/(V_S + V_T)$ . Values of flocculation ratio are plotted versus dilution concentration (C), calculated as  $C = W_a/(V_S + V_T)$  (Figure 6). A regression line connecting the points is extrapolated to the x and y-axes. The x and y intercepts determined from the extrapolation, referred to as the dilution concentration minimum ( $C_{min}$ ) and the flocculation ratio maximum ( $\Phi_{max}$ ), respectively, are used to calculate three Heithaus parameters. Heithaus parameters are designated;  $p_a = 1 - \Phi_{max}$ , which measures the peptizability of the

asphaltene fraction;  $p_o = \Phi_{max}(C_{min}^{-1} + 1)$ , which measures the solvent power of the maltene fraction, and  $P = p_o/(1 - p_a)$ , which is a measure of the overall compatibility of the asphalt. Larger values of  $p_a$ ,  $p_o$ , and P generally represent peptizable asphaltenes, maltenes that are a good solvent, and a compatible asphalt overall. Smaller values of  $p_a$ ,  $p_o$ , and P represent the reverse. The  $p_a$  and  $p_o$  values do not necessarily vary directly with one another among asphalts. An asphalt may be composed of asphaltenes that are not readily peptizable that are dispersed in maltenes that have good solvent characteristics, or the reverse. Table 1 reports "p-values" measured for the eight SHRP asphalt and asphaltene percentages. It is generally noted with the data in this table that n-heptane asphaltene content is linearly correlated with iso-octane asphaltene content, and that asphaltene content is generally inversely proportionally to  $p_a$ -values

![](_page_71_Figure_1.jpeg)

Figure 6. Flocculation ratio versus dilution concentration plot.
**Table 1.** Heithaus compatibility parameters: asphaltene peptizability (p<sub>a</sub>), maltene peptization (p<sub>o</sub>), and state of peptization (P) measured by AFT and asphaltene mass fractions based on n-heptane and 2,2,4-trimethylpentane precipitation.

| Asphalt | Heithaus<br>asphaltene<br>peptizability<br>parameter, pa | Heithaus<br>maltene<br>peptization<br>parameter, p₀ | Heithaus state<br>of peptization<br>parameter, P | n-Heptane<br>precipitated<br>asphaltene<br>mass fraction | iso-Octane<br>precipitated<br>asphaltene mass<br>fraction |
|---------|--|---|--|--|---|
| AAA-1   | 0.701  | 0.825   | 2.80   | 0.158  | 0.221   |
| AAB-1   | 0.697  | 0.872   | 2.88   | 0.173  | 0.224   |
| AAC-1   | 0.765  | 0.759   | 2.23   | 0.099  | 0.155   |
| AAD-1   | 0.660  | 0.888   | 2.61   | 0.202  | 0.274   |
| AAF-1   | 0.685  | 0.732   | 2.32   | 0.134  | 0.187   |
| AAG-1   | 0.802  | 0.817   | 4.12   | 0.05   | 0.102   |
| AAK-1   | 0.692  | 0.976   | 3.16   | 0.201  | 0.247   |
| AAM-1   | 0.902  | 0.759   | 7.73   | 0.037  | 0.112   |

## **Flocculation Kinetics Titrimetry (FKT)**

Flocculation kinetics titrimetry experiments have been undertaken using automated flocculation titrimetry [Pauli 2004] to measure, among other asphalt compositional properties, asphaltene flocculation rate constants as a function of temperature and concentration and mass percentage of *"flocculation kinetics"* defined asphaltenes.

In FKT testing samples of asphalt are prepared by dissolving specified amounts of asphalt material in toluene, then titrating each solution with iso-octane (2,2,4-trimethyl pentane "isooctane", HPLC Grade). Kinetic experiments are carried out directly after the detection of the flocculation onset (via AFT), at which point in time the addition of titrant is discontinued (i.e., the titrant pump is stopped), but the spectrophotometric monitoring of the test solution is allowed to continue. This approach allows for the measurement in the change in absorption of UV-Visible light, monitored at a frequency of 740 nm, over time. Plots of the change in absorption of UV-Visible light at 740 nm versus time are then used to characterize the kinetic process of asphaltenes precipitating from solution, constituting a measure of the rate of flocculation.

A second-order rate equation

$$\frac{dc_A}{dt} \approx -k_1 c_A^{2}.$$
(20)

models the forward process of the reaction mechanism

$$A_j + A_i \xrightarrow{k_{ij}} A_{j+i}, \qquad (21)$$

assuming that the flocculation process involves both conduction and diffusion of  $A_i$ -monomer asphaltene molecules attaching to  $A_i$ -mer clusters already present in a colloidal solution.

Limits of integration are imposed on equation 20 when taking into account the material balance of "what species have and have not flocculated", resulting in initial and final solution concentrations of  $c_{\infty,s\ln} = (1/V)(n_{\max} - n_{\infty,agg})$ , and  $c_{0,s\ln} = (1/V)(n_{\max} - (n_{0,agg} = 0))$ , respectively. These two material balance limits are defined by V, the total volume of solution and  $n_{0,agg}$ ,  $n_{\max}$ , and  $n_{\infty,agg}$ , which define the number of moles of solute particles, initially in solution, the maximum number of moles which may potentially flocculate, and the number of moles which actually are observed to flocculate. The integral,

$$\int_{c_{0,s\ln}}^{c_{\infty,s\ln}} \frac{dc_A}{c_A^2} = -\int_{t_0=0}^{t_\infty} k dt$$
(22)

once evaluated, defines the time-dependent concentration of flocculated asphaltenes  $c_A(t)$ , in terms of the rate constant k, time t, and a steady-state concentration  $c_{A_{\text{max}}}$ , expressed as

$$c_A(t) = c_{A\max} - \frac{c_{A\max}}{1 + c_{A\max}kt}$$
(23)

where

$$(Abs_{adj})_{\max} = \partial c_{A\max}$$
(24)

given the path length of the flow cell l, and the molar extinction coefficient  $\delta$ . A flocculationrate curve (depicted in figure 7) is derived from flocculation kinetics plots by truncating both absorption and time data to a literal "0" value (i.e., at the flocculation onset, effectively initiating the flocculation reaction).



**Figure 7.** Absorption versus time (seconds) plot for SHRP asphalt AAA-1. Dash–curve: flocculation onset curve (Stage 1) plus rate curve (Stage 2) measured at initial concentration;  $c_0 = 0.250$  g/mL, T = 25°C. Solid-curve: truncated rate curve.

An initial adjusted absorption,  $(Abs_{adj})_{initial}$ , and an initial adjusted time,  $(t_{adj})_{initial}$ , are calculated by subtracting the absorption and time values at the flocculation onset from all preceding absorption and time data points. This step effectively translated the flocculation-rate curve to  $(Abs_{adj})_{initial} = 0$  and  $(t_{adj})_{initial} = 0$  prior to fitting of the data to the following equation

$$Abs(t_{adj}) = (Abs_{adj})_{max} - \frac{(Abs_{adj})_{max}}{1 + (Abs_{adj})_{max}kt_{adj}}$$
(25)

where  $(Abs_{adj}) \propto c_A$ , and  $(Abs_{adj})_{max} \propto c_{A max}$ .

Flocculation kinetics plots are generated based on a calculated flocculation rate constant k, derived in terms of the effective amount of blocked light  $(Abs_{adj})_{max}$ , measured at 740 nm through a small path length flow cell, which may be correlated with the amount of normal heptane asphaltene mass fractions  $\chi_{n-heptane}$ , determined by a solvent de-asphaltene separation method [Branthaver et al. 1991].

Figure 8 depicts a plot of  $(Abs_{adj})_{max}$  versus  $\chi_{n-heptane}$  data at two concentration levels, each fitted to a correlation function of the form

$$\left[\left(Abs_{adj}\right)_{max}\left(c_{0}\right)\right]_{T,P} = \beta_{0} + \beta_{1}\chi_{n-heptane} + \beta_{2}\chi_{n-heptane}^{2}$$

$$\tag{26}$$



**Figure 8.** Maximum absorption of flocculated material plotted as a function of the n-heptane insoluble asphaltene mass fraction, determined for 0.250 g/mL and 0.375 g/mL solutions of asphalt prepared in toluene and titrated with iso-octane at 25°C.

From a phenomenological point-of-view the principle reaction [Benson 1960; Atkins 1994; Levine 1988; Steinfeld et al. 1999]

$$A_j + A_i \xrightarrow{k_{ij}} A_{j+i}, \qquad (27)$$

assumes a process involving both conduction and diffusion of  $A_i$ -monomer asphaltene molecules attaching to  $A_j$ -mer clusters already present in a colloidal solution, forming  $A_{j+i}$ flocculated particle clusters. The particle flux,  $J_i = -D_i(dc_i/dR)$ , is then defined as the concentration  $c_i$ , of  $A_i$ -monomer molecules migrating through a hypothetical spherical surface element  $4\pi R_{ji}^2$ , such that the rate of exchange in the number of moles of  $A_i$ -monomer molecules per unit time  $dn_i/dt$ , is expressed by

$$\frac{dn_i}{dt} = 4\pi R_{ji}^2 D_{ij} \frac{dc_i}{dR_{ji}}$$
(28)

Here  $R_{ji} = r_j + r_i$  defines the intra-molecular distance between  $A_j$ -mer clusters and  $A_i$ -monomer molecules, respectively, once in contact, and the term  $(D_{ji} \equiv D_i + D_j)$  represents the effective diffusion coefficient for "reactants"  $A_i$  and  $A_j$ . Integration of equation 28

$$\int_{c_{i,R=\eta+r_B}}^{c_{i,R=\infty}} dc_B = \frac{dn_i}{dt} \frac{1}{4\pi (D_i + D_j)} \int_{R_{ji}=r_j + r_i}^{\infty} \frac{dR}{R^2}$$
(29)

between the limits; "of when all available clusters and molecules are in contact" ( $R_{ji} = r_j + r_i$ ,), and "when they are in solution (at infinite separation at  $R = \infty$ )", results in the difference in concentration for time-initial and time-final expressed by

$$c_{i,R\to\infty} - c_{i,R_{ji}} = \frac{dn_i}{dt} \frac{1}{4\pi D_{ji}R_{ji}},$$
(30)

Imposing the following steady-state conditions in terms of the number of *j*-mer particles,  $N_{j}$ . [Levine 1988], namely

$$\frac{dn_i}{dt} = \frac{kc_jc_iV}{N_j} = \frac{kc_jc_iV}{N_An_j} = \frac{kc_jc_i}{N_Ac_j} = \frac{kc_i}{N_A}$$
(31)

the rate per unit molecular volume,  $\overline{v}$ , describing this "bimolecular" reaction is shown to be related to the forward rate constant k, and the concentrations of suspended clusters and molecules as

$$\frac{N_{j}dn_{i}}{Vdt} \equiv \overline{v} = kc_{j}c_{i}, \qquad (32)$$

Substitution of equation 32 into 31, then into equation 30, with some rearrangement gives

$$c_{i,R} = c_i \left( 1 - \frac{k}{4\pi N_A D_{ji} R_{ji}} \right) = \left( 1 - \frac{k}{k_{diff}} \right)$$
(33)

Thus, an expression defining the diffusion-limited rate constant is defined by

$$4\pi N_A D_{ji} R_{ji} = k_{diff} \tag{34}$$

where  $N_A$  is Avogadro's number.

If the Stokes-Einstein [Levine 1988] equation, defining the diffusion coefficient,

$$D_{si}^{\infty} = \frac{k_B T}{6\pi\eta_s r_i}$$
(35)

is the substituted into 34, given  $k_B$  Boltzmann's constant, and  $\eta_s$  the solvent viscosity, and given the approximation that  $(1/2R_{ji} = r_j = r_i)$  [Atkins 1994], and the substitution of  $N_A k_B = R$ , where *R* is the ideal gas constant, an expression relating the solution viscosity to the diffusion-limited rate constant  $k_{diff}$ , may be expressed by

$$k_{diff} = 4\pi N_A R_{ji} \left( \frac{k_B T}{3\pi \eta_s R_{ji}} + \frac{k_B T}{3\pi \eta_s R_{ji}} \right) = \frac{8RT}{3\eta_s}$$
(36)

Equation 36 defines an ideal diffusion-controlled process relationship (i.e.,  $8RT/3\eta_s = k_{diff}$ ) relating the viscosity of the solvent phase of a colloidal suspension to the rate constant of diffusing particles in the suspension. Figures 9 and 10 depict the relationship between the asphaltene flocculation rate constant and the maltene viscosity for several experimental data sets derived from testing the 8 SHRP core asphalts.



**Figure 9.** n-Heptane soluble maltene fraction viscosities (Pa\*s @ 25°C) plotted versus the inverse of the rate constant of flocculation, measured at 25°C. Top plot depicts eight SHRP asphalts with correlation line drawn through "higher viscosity maltenes" asphalts, and bottom plot depicts four of the eight SHRP asphalts with correlation line drawn through "low viscosity maltenes" asphalts.



**Figure 10.** "n-Heptane-Soluble Maltenes Fraction" viscosities (Pa\*s @ 60°C) plotted versus the inverse of the flocculation rate constant measured at 50°C.

## **RESULTS AND DISCUSSION**

Asphalt samples were prepared as 0.600 g and 0.800 g per 3.0 mL toluene solutions. Sample solutions, after sitting for 24-h, were titrated with 2,2,4-trimethylpentane (iso-octane) at a rate of 0.300 mL/min and at a temperature of 35°C. At the detection of the flocculation onset (figure 11) 30-seconds of additional titrant was added to the solution, then the titrant was discontinued. The flocculation of asphaltenes was then monitored for 10-minutes.



Figure 11. Percent transmittance versus titrant delivery time AFT flocculation plot.

Compatibility parameters and flocculation kinetics parameters were determined for each set of 0.600 g and 0.800 g asphalt per 3.0 mL toluene data (figures 12 and 13).

Directly after completion of a titration/flocculation test, the test solution was removed from the AFT instrument and diluted with an additional 20-mL of iso-octane. Sample vials were capped and stored away to be filtered at a later time.

AFT solutions were filtered with tared 10-micron filter paper through a Buchner funnel. Filter paper with filter cake asphaltenes were dried in a 75°C oven for a 1-hr prior to determining the final weight of asphaltenes recovered.



Figure 12. AFT Heithaus [Heithaus 1962] compatibility worksheet.



**Figure 13.** FKT asphaltene flocculation rate plot fit to the expression  $Abs(t_{adj}) = (Abs_{adj})_{max} - \left[ (Abs_{adj})_{max} / (1 + (Abs_{adj})_{max} kt_{adj}) \right].$ 

Table 2 reports AFT and FKT parameters determined for 10 UMASS binder samples. Plots depicted in Figure 14 show a correlation plot relating the maximum absorbance, derived based on equation 25, to asphaltene mass percentage determined gravimetrically. Linearity between  $(Abs_{adj})_{max}$  and  $100 \cdot \chi_{asphaltenes}$  is observed within the concentration range of 15% to 24% asphaltenes. The flocculation test is based on detection of blocked light in a concentrated solution, thus, the cell path length and differences in light absorption characteristic of the sample likely bias the test resulting in the limited concentration range of correlation.

| Sample                 | pa    | p₀    | Р     | Abs (0.8-g) | k(0.8-g) | % asphaltene |
|------------------------|-------|-------|-------|-------------|----------|--------------|
| 64-28 Control          | 0.35  | 4.98  | 7.72  | 0.521       | 4.39E-03 | 4.8          |
| 64-28 15%RAP HMA       | 0.46  | 3.26  | 6.02  | 0.519       | 4.71E-03 | 16.9         |
| 64-28 25%RAP HMA       | 0.62  | 1.28  | 3.34  | 0.548       | 4.06E-03 | 24.2         |
| 64-28 35%RAP HMA       | 0.57  | 1.84  | 4.27  | 0.615       | 4.55E-03 | 24.5         |
| 64-28 50%RAP HMA       | 0.58  | 1.89  | 4.50  | 0.549       | 3.45E-03 | 24.0         |
| RAP Stockpile Material | 0.62  | 1.55  | 4.07  | 0.527       | 3.17E-03 | 20.0         |
| 64-28 50%RAP WMA       | 0.70  | 0.15  | 0.50  | 0.561       | 3.11E-03 | 20.6         |
| 64-28 35%RAP WMA       | 0.62  | 1.11  | 2.95  | 0.503       | 4.43E-03 | 17.8         |
| 52-34 50%RAP HMA       | 0.47  | 3.52  | 6.63  | 0.452       | 2.99E-03 | 20.1         |
| 52-34 35%RAP HMA       | -1.16 | 28.23 | 13.08 | 0.496       | 2.11E-03 | 23.4         |

Table 2. AFT and FKT parameters determined for 10 UMASS binder samples.

A predicted asphaltene concentration may be calculated for mixtures of two binders (neat binder blended with a RAP binder for example) in the absence of oxidation of the material blend during mixing, utilizing the following equation

$$\chi_{mix} = \left(x_i \chi_A + \left(1 - x_i\right) \chi_R\right) \tag{37}$$

Table 3 reports predicted asphaltene concentration based on the asphaltene contents of the original materials (i.e., control and RAP stockpile material) and the percentage of RAP. Figure 15 depicts a plot relating gravimetric asphaltenes and predicted blend asphaltenes to RAP percentage in a mix. Assuming that HMA materials undergo more extensive oxidation during the mixing process compared to WMA material, both these materials are observed to have substantially higher asphaltene contents (presumed to be due to mix plant condition oxidation) compared to predicted values.

| Sample                 | %RAP | % Asphaltene 0.8g | Mix Asphaltenes |
|------------------------|------|-------------------|-----------------|
| 64-28 Control          | 0    | <b>4.8</b>        | 4.8             |
| 64-28 15%RAP HMA       | 15   | 16.9              | 7.1             |
| 64-28 25%RAP HMA       | 25   | 24.2              | 8.6             |
| 64-28 35%RAP HMA       | 35   | 24.5              | 10.1            |
| 64-28 50%RAP HMA       | 50   | 24.0              | 12.4            |
| RAP Stockpile Material | 100  | <b>20.0</b>       | 20.0            |

| Table 3. | Predicted | asphaltene | content ba | ased on | mix model | (Eq 37) | • |
|----------|-----------|------------|------------|---------|-----------|---------|---|
|----------|-----------|------------|------------|---------|-----------|---------|---|



Figure 14. Correlation plots relating maximum absorbance to asphaltene mass percentage determined gravimetrically.



Figure 15. Plots relating gravimetric asphaltenes to RAP percentage in a mix.

**Utilizing FKT data it is possible to predict maltene viscosities:** Furthermore, if both maltene viscosities and asphaltene content of a blend are known it is possible to predict the binder viscosities. The following line of reasoning is employed:

Given the flocculation rate constants k, and assuming identical size particle formation and interaction during the flocculation testing, (i.e.,  $\xi = \text{constant} = 1$ ), flocculating solution viscosities are calculated as

$$\eta_s(k) = \xi \frac{1}{k} \tag{38}$$

The flocculation solution is then related to the viscosities of the solution component viscosities as

$$\eta_s(k) = \phi_{titrant} \eta_{titrant} + \phi_{solvent} \eta_{solvent} + \phi_{maltene} \eta_{maltene}$$
(39)

Next, the maltene volume fraction is approximated by  $\phi_{maltene} \approx (1 - \chi_{asphaltene})$  given the asphaltene mass fraction  $\chi_{asphaltene} = 0.01 (Mass \% asphaltenes)$  and ignoring differences in density among samples, such that the viscosity of the maltene fraction of a tested binder is calculated as

$$\eta_{maltene} = \frac{1}{\phi_{maltene}} \Big( \eta_s(k) - \Big( \phi_{iitrant} \eta_{iitrant} + \phi_{solvent} \eta_{solvent} \Big) \Big)$$
(40)

where

$$\phi_i = \frac{V_i}{V_i + V_j + V_k} \tag{41}$$

It is further assumed that the viscosity of the test solvents (titrant + solvent) in the solution is much less than the maltene viscosity,

$$\eta_{maltene} > \eta_s(k) \square \left( \phi_{titrant} \eta_{titrant} + \phi_{solvent} \eta_{solvent} \right)$$
(42)

thus

$$\eta_{maltene} \approx \frac{\eta_s(k)}{\phi_{maltene}} \tag{43}$$

Finally, the viscosity of the binder, based on the Pal-Rhodes equation is calculated as

$$\eta_{blend} = \eta_{maltene} \left(1 - 3.3 \chi_{asphaltene}\right)^{-2.5} \tag{44}$$

Table 4 reports AFT and FKT parameters used to calculate predicted maltene viscosities and predicted binder viscosities for 64-28 HMA blends, the control and the RAP material. Maltene viscosities based on a mixing rule

$$\left(\eta_{malt}\right)_{mix} = \left(x_i \eta_{0A} + \left(1 - x_i\right) \eta_{0R}\right) \tag{45}$$

were also compared with maltene viscosities determined based on flocculation rates. Figure 16, which depicts a plot of maltene viscosities based on the mix rule (Eq 45) compared with maltene viscosities based on flocculation rate constants (Eq 43) show these values to reasonably line up in one:to:one correspondence, unlike predicted and measured asphaltenes.

A notable peculiarity may be pointed for measured p-values for the 64-28 Control as compared to typical p-values of SHRP when data in tables 1 and 2 are compared. It has been generally noted that  $p_a$  values decrease and  $p_o$  values increase with increasing oxidation of a binder. It has also been observed in other types of heavy residuum that high  $p_o$  values (>1.5) are indicative of high concentration of polar aromatics, and sometimes non-organic particulate matter. Based on the resulted reported in table 2 for the 64-28 Control sample, material blends with RAP after mix plant conditions appear to result in excessive oxidation, and hence, excessive asphaltene production. As a result of this, predicted viscosities of the blends may be observed to be much higher than predicted based on a simple mixture formulation, as depicted in Figure 17.

| Sample                 | $V_{solvent}(mL)$ | $V_{titrant}(mL)$ | $\phi_{\scriptscriptstyle solvent}$ | $\phi_{\scriptscriptstyle solvent}$ | $\phi_{\scriptscriptstyle titrant}$ | $\eta_{\scriptscriptstyle maltene}$ |
|------------------------|-------------------|-------------------|-------------------------------------|-------------------------------------|-------------------------------------|-------------------------------------|
| 64-28 Control          | 3                 | 7.02              | 0.278                               | 0.651                               | 0.071                               | 3225                                |
| 64-28 15%RAP HMA       | 3                 | 6.54              | 0.294                               | 0.641                               | 0.065                               | 3263                                |
| 64-28 25%RAP HMA       | 3                 | 6.69              | 0.291                               | 0.650                               | 0.059                               | 4184                                |
| 64-28 35%RAP HMA       | 3                 | 6.57              | 0.295                               | 0.646                               | 0.059                               | 3698                                |
| 64-28 50%RAP HMA       | 3                 | 6.97              | 0.284                               | 0.659                               | 0.058                               | 5035                                |
| RAP Stockpile Material | 3                 | 7.34              | 0.273                               | 0.669                               | 0.058                               | 5416                                |

**Table 4.** AFT and FKT parameters determined for 10 UMASS binder samples.

| Sample                 | $1/k = \eta(k)$ | $\left(\eta_{\scriptscriptstyle malt} ight)_{\scriptscriptstyle mix}$ | $\eta = \eta_m (1 - 3.3 \chi_{mix})^{-2.5}$ | $\eta = \eta_m (1 - 3.3 \chi_a)^{-2.5}$ |
|------------------------|-----------------|---|---|---|
| 64-28 Control          | 228             | 3225  | 4975  | 4975                                    |
| 64-28 15%RAP HMA       | 213             | <u>3553</u>   | 6922  | 27364                                   |
| 64-28 25%RAP HMA       | 246             | <u>3773</u>   | 8697  | 205558                                  |
| 64-28 35%RAP HMA       | 220             | <u>3992</u>   | 11024                                       | 247661                                  |
| 64-28 50%RAP HMA       | 290             | <u>4320</u>   | 16075                                       | 215515                                  |
| RAP Stockpile Material | 316             | 5416  | 79376                                       | 79376                                   |



Figure 16. Maltene viscosities based on a mix rule compared with Maltene viscosities based on flocculation rate constants.

**Table 1.** Heithaus compatibility parameters: asphaltene peptizability (p<sub>a</sub>), maltene peptization (p<sub>o</sub>), and state of peptization (P) measured by AFT and asphaltene mass fractions based on n-heptane and 2,2,4-trimethylpentane precipitation.

| Asphalt | Heithaus<br>asphaltene<br>peptizability<br>parameter, pa | Heithaus<br>maltene<br>peptization<br>parameter, p₀ | Heithaus state<br>of peptization<br>parameter, P | n-Heptane<br>precipitated<br>asphaltene<br>mass fraction | iso-Octane<br>precipitated<br>asphaltene mass<br>fraction |
|---------|--|---|--|--|---|
| AAA-1   | 0.701  | 0.825   | 2.80   | 0.158  | 0.221   |
| AAB-1   | 0.697  | 0.872   | 2.88   | 0.173  | 0.224   |
| AAC-1   | 0.765  | 0.759   | 2.23   | 0.099  | 0.155   |
| AAD-1   | 0.660  | 0.888   | 2.61   | 0.202  | 0.274   |
| AAF-1   | 0.685  | 0.732   | 2.32   | 0.134  | 0.187   |
| AAG-1   | 0.802  | 0.817   | 4.12   | 0.05   | 0.102   |
| AAK-1   | 0.692  | 0.976   | 3.16   | 0.201  | 0.247   |
| AAM-1   | 0.902  | 0.759   | 7.73   | 0.037  | 0.112   |

**Table 2.** AFT and FKT parameters determined for 10 UMASS binder samples.

| Sample                 | pa                | p₀                | Р     | Abs (0.8-g) | k(0.8-g) | % asphaltene |
|------------------------|-------------------|-------------------|-------|-------------|----------|--------------|
| 64-28 Control          | <mark>0.35</mark> | <mark>4.98</mark> | 7.72  | 0.521       | 4.39E-03 | 4.8          |
| 64-28 15%RAP HMA       | 0.46              | 3.26              | 6.02  | 0.519       | 4.71E-03 | 16.9         |
| 64-28 25%RAP HMA       | 0.62              | 1.28              | 3.34  | 0.548       | 4.06E-03 | 24.2         |
| 64-28 35%RAP HMA       | 0.57              | 1.84              | 4.27  | 0.615       | 4.55E-03 | 24.5         |
| 64-28 50%RAP HMA       | 0.58              | 1.89              | 4.50  | 0.549       | 3.45E-03 | 24.0         |
| RAP Stockpile Material | 0.62              | 1.55              | 4.07  | 0.527       | 3.17E-03 | 20.0         |
| 64-28 50%RAP WMA       | 0.70              | 0.15              | 0.50  | 0.561       | 3.11E-03 | 20.6         |
| 64-28 35%RAP WMA       | 0.62              | 1.11              | 2.95  | 0.503       | 4.43E-03 | 17.8         |
| 52-34 50%RAP HMA       | 0.47              | 3.52              | 6.63  | 0.452       | 2.99E-03 | 20.1         |
| 52-34 35%RAP HMA       | -1.16             | 28.23             | 13.08 | 0.496       | 2.11E-03 | 23.4         |



Figure 17. Predicted binder viscosities based on mix model calculations.

## CONCLUSIONS AND RECOMMENDATIONS

Evaluation of the compatibility parameters measured specifically for the 64-28 data sets suggests that the RAP Stockpile Material is characterized by typical p-values for a mildly aged binder material, but the 64-28 Control material (Base binder) is measured to have atypical p-values, specifically, the low  $p_a$  value is more often characteristic of high asphaltene content, but in the present case asphaltene content is low, and high  $p_o$  values are indicative of high concentrations of polar aromatic materials, and or a more complex chemical make (polymer or other type of modification) than is typical for a straight run binder. It was further noted that the increase in asphaltene content for RAP blends was related to simulated mix plant conditioning, thus it was observed that 64-28 WMA materials at the same RAP content showed less asphaltene content than 64-28 HMA materials. It is speculated based on the results presented that this particular blend of 64-28 Control material with RAP is highly susceptible to mix plant condition oxidation. Blending theories of residua also suggest that blending of materials with vastly different compatibility characteristics may lead to very incompatible resultant blends.

Additional testing may involve IR-spectrometric investigation to evaluation the extent of oxidation and to better characterize the starting and blended materials. Chromatographic separation (SARA, GPC, IEC) and material fraction physicochemical characterization (IR) of the 64-28 Control material may also provide additional information regarding this material's

peculiar compatibility characteristics. Atomic force microscopic (AFM) studies may also provide further information regarding wax content, polymer modification, and presence of recycled motor oil bottoms.

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