EVALUATION OF VESTENAMER REACTIVE MODIFIER IN CRUMB RUBBER ASPHALT

Performance of Asphalt Binder and Asphalt Concrete Modified by Ground Tire and VESTENAMER® – A Laboratory Study

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### Abstract
VESTENAMER® “Polyoctenamer rubber” is used in the asphalt industry as a cross-linkable dispersant and compatibilizer, which, along with crumb rubber, can modify asphalt cement with the goal of making it a high-performance binder. The laboratory work presented in this document was conducted to evaluate the effectiveness of VESTENAMER® in providing a better rubber/asphalt mix for pavement construction. The work included characterization of VESTENAMER®-modified binders, selection and design of a reference asphalt mix, providing crumb rubber/asphalt mixes with different levels of the modifier, performing a series of laboratory tests, analyzing data, and providing information regarding the potential performance of asphalt mixtures containing VESTENAMER®-modified binders. This report presents the test results, findings, conclusions, and recommendations based on the concluded work.

### Key Words
- Asphalt cement
- asphalt mix
- crumb rubber
- high-performance binder
- pavement construction
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PREFACE

This project was carried out from January 2002 through June 2003. An extensive amount of laboratory testing was performed during this period to provide sufficient information for the project. This report presents the test results, findings, conclusions, and recommendations based on the concluded work.

INTRODUCTION

VESTENAMER® “Polyoctenamer rubber” is used in the asphalt industry as a cross-linkable dispersant and compatibilizer, which, along with crumb rubber, can modify asphalt cement with the goal of making it a high-performance binder. The laboratory work presented in this document was conducted to evaluate the effectiveness of VESTENAMER® in providing a better rubber/asphalt mix for pavement construction. The work included characterization of VESTENAMER®-modified binders, selection and design of a reference asphalt mix, providing crumb rubber/asphalt mixes with different levels of the modifier, performing a series of laboratory tests, analyzing data, and providing information regarding the potential performance of asphalt mixtures containing VESTENAMER®-modified binders.

MATERIALS

A Superpave performance-grade binder PG 58-28 was used for this study. The binder was provided by the Northeast Center of Excellence for Pavement Technology (NECEPT) and was approved by Degussa Corporation. The source of the binder was Koch Pavement Solutions from Wichita, Kansas. The crumb rubber was a mesh 14 ground tire rubber (GTR) and was selected and provided by Degussa Corporation. The reactive modifier was also provided to the research team by Degussa Corporation. As an extension to the work conducted under this project, a mesh 30 GTR was also included in the study. The aggregate used in the mixture is dolomite in nature from a quarry in Curtin Gap, PA, and is produced by HRI, Inc.

TESTING PROGRAM

The laboratory investigation included two phases. Phase one of the study was fully concentrated on the binder evaluation. Phase two of the project dealt with evaluation of the asphalt-aggregate mixtures when modified with GTR and VESTENAMER®. The conducted tests, the results, and discussion of results are presented in the subsequent sections of this report.
Binder Evaluation

Originally, two levels of crumb rubber modification were used: 10 percent and 20 percent (by weight of the asphalt binder). For those binders modified by VESTENAMER®, the polymer was added at a level of 4.5 percent (based on the weight of GTR). Preparing specimens for testing proved to be difficult for binder with 20 percent GTR after aging. Therefore, investigation was conducted on 5 percent and 10 percent levels of mesh 14 GTR. The extended work on mesh 30 GTR was conducted only at the 10 percent level. A testing matrix is presented in Table 1.

Table 1. Binder Treatment Matrix Applied in This Study.

<table>
<thead>
<tr>
<th>Type</th>
<th>Binder PG Grade</th>
<th>GTR % by Weight of Binder</th>
<th>VESTENAMER Modifier % by Weight of GTR</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Mesh 14</td>
<td>Mesh 30</td>
</tr>
<tr>
<td>A</td>
<td>58-28</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>B-1</td>
<td>58-28</td>
<td>5</td>
<td>NU*</td>
</tr>
<tr>
<td>B-2</td>
<td>58-28</td>
<td>5</td>
<td>NU*</td>
</tr>
<tr>
<td>C-1</td>
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<td>10</td>
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</tr>
<tr>
<td>C-2</td>
<td>58-28</td>
<td>10</td>
<td>10</td>
</tr>
</tbody>
</table>

* NU: Not used. Mesh 30 GTR was used only at 10 percent level.

The same binder was used throughout the study. As shown in Table 1, two levels of treatment with VESTENAMER® (0 and 4.5 percent) were used with mesh 14 GTR, providing a total of five different combinations.

The binder performance tests were conducted on all modified binders as presented in Table 2.

Table 2. Tests and Practices Conducted on the Binders for This Study.

<table>
<thead>
<tr>
<th>Test and Practice</th>
<th>Description of Test</th>
</tr>
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<td>Dynamic Shear Rheometer on Unaged Binder</td>
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<tr>
<td>Rolling Thin Film Oven (RTFO)</td>
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<tr>
<td>Dynamic Shear Rheometer on Short-Term Aged Binder (after RTFO)</td>
<td></td>
</tr>
<tr>
<td>Pressure Aging Vessel (PAV)</td>
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<tr>
<td>Dynamic Shear Rheometer on PAV Aged Binder</td>
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<tr>
<td>Bending Beam Rheometer on Long-Term Aged Binder (after RTFO+PAV)</td>
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</tr>
<tr>
<td>Direct Tension Test</td>
<td></td>
</tr>
<tr>
<td>Rotational Viscosity*</td>
<td>Conducted on mesh 14 GTR only.</td>
</tr>
</tbody>
</table>

* Conducted on mesh 14 GTR only.

Preparation of the Modified Binder

There are two stages to preparation of the modified binder. Stage one requires preparation of crumb rubber modifier (CRM) using GTR, and stage two requires addition of the reactive modifier VESTENAMER®.
For preparation of GTR, it is important to pay attention to the reaction time. The finer the material, the quicker it will "react." Basically, for a given weight of CRM, the reaction time is directly proportional to the diameter squared of the CRM particles. In addition, the reaction time is inversely proportional to the temperature of the material. In general, the reaction time approximately doubles with every 10 °C (18 °F) decrease in asphalt-cement temperature. Adding CRM to the asphalt drops the temperature of the asphalt cement due to the ambient temperature of the CRM. For example, the addition of 20 percent CRM material to asphalt cement at about 204 °C (400 °F) will cause the combined temperature to drop to about 177 °C (350 °F). With the smaller material and lower concentrations, the "reaction" time can be shorter and is generally about 15 minutes. However, in most cases, the reaction time is selected between 30 to 60 minutes.

For mixing GTR with the PG 58-28 binder of this study, a target temperature of 150 °C (302 °F) and a reaction time of 30 minutes were used for both 5 percent and 10 percent GTR concentration levels. A Ross™ mixer was used for blending. The Batch Model High Shear Rotor-Stator mixer design consists of a single-stage rotor that turns at high speed within a stationary stator. As the rotating blades pass the stator, they mechanically shear the contents. With the mixer rpm set at 4,000, temperature spiked at 170 °C within the first 7 minutes. Reducing the mixer rpm to 2,000 brought the temperature back to 150 °C. The authors believe that most of the increase in temperature in the initial stage of mixing binder with GTR was a result of physical action of shearing and generated heat from friction. However, part of this increase in temperature could have been the result of exotherm, the heat generated as a result of chemical reaction. For those modifications requiring VESTENAMER®, the modifier was added to the binder at the same time the GTR was being added. The required amount of modifier was added to the binder at 150 °C and was allowed to react with the binder for 30 minutes while being mixed in the shear mixer.

Discussion of Results from Binder Tests

The summary of results from binder tests is presented in graphical form in Figures 1 through 7. Figures 1 and 2 present the results from the dynamic shear rheometer testing on unaged binder and short-term aged binder, respectively, for different blends. The result is the ratio of complex modulus over the phase angle from the test. A higher ratio is desirable since this property indicates rutting resistance properties of the binder. The dashed red line indicates the minimum acceptable level at a certain temperature. The first observation is that the binders with GTR and GTR+ VESTENAMER® provide higher rutting resistance compared to the control binder. The second observation is that modification with 5 percent GTR provides one grade bump in the performance grade (PG) of the binder. In other words, it turns a PG 58 binder into a PG 64. This is true for both GTR-modified binders with and without VESTENAMER®. Ten percent GTR with and without VESTENAMER® results in three grades of bump. This means that, at the high end of temperatures, a PG 76 binder is produced with 10 percent GTR from a PG 58.

Figure 3 shows the results for the binder complex modulus at 19 °C. A lower value is desirable since it implies less susceptibility to fatigue cracking. Both GTR and GTR+ VESTENAMER®
provide stiffness values comparable with the control binder at this temperature. The five percent level modification has provided slightly higher stiffness compared to the control binder, while the ten percent level modification has resulted in a slightly lower stiffness compared with the control binder. In all cases, the stiffness is lower than the maximum limit of 5,000 KPa at 19 ºC.

Figure 4 shows the viscosity results for the binders at a temperature of 135 ºC. These results are obtained from tests with the rotational viscometer. While with GTR and GTR+ VESTENAMER® an increase in viscosity is observed, the result is considerably lower than the limiting value of 3 Pa-s. The binder would not meet the current binder specification, AASHTO M320-02, if the viscosity exceeds 3 Pa-s at 135 ºC. A lower value is desirable to ensure that the binder can be properly pumped and worked with.

Figure 5 presents the test results from the bending beam rheometer. It can be seen that at low temperatures (-18 ºC and -24 ºC) both GTR and GTR+ VESTENAMER® modifications result in reduction of the binder stiffness. This is indeed desirable at low temperatures, since it makes the binder more resistant to low-temperature cracking. As shown in Figure 6, not much change is observed in the slope of the stiffness-time relationship (i.e., m-value when both stiffness and time are plotted in log-scale) with modification. The slope presents the ability of the binder to relax stresses with time. Therefore, a higher value is desirable, since it means that at a given temperature, the binder can release induced thermal stresses at a faster rate.

Figure 7 exhibits the strain at failure for different blends at low temperature (-18 ºC) from direct tension tests. The results in this figure indicate that while no significant difference is observed between failure strains for different blends at 5 percent GTR level, significant improvement is observed at 10 percent GTR level when VESTENAMER® is used. A higher strain at failure is desirable since it implies a more ductile behavior, one that should be more resistant to low-temperature cracking and fatigue.
Figure 1. Results from Tests with *Dynamic Shear Rheometer* on *Unaged* Binder for Different Blends of Binder with mesh 14 GTR.

Figure 2. Results from Tests with *Dynamic Shear Rheometer* on *Short-Term Aged* Binder for Different Blends of Binder with Mesh 14 GTR.
Figure 3. Results from Tests with *Dynamic Shear Rheometer* on *Long-Term Aged* Binder for Different Blends of Binder with Mesh 14 GTR.

Figure 4. Results from Tests with *Rotational Viscometer* on *Unaged* Binder for Different Blends of Binder with mesh 14 GTR.
Figure 5. Stiffness Results from Tests with *Bending Beam Rheometer* on *Long-Term Aged* Binder for Different Blends of Binder with mesh 14 GTR.

Figure 6. Slope Results from Tests with *Bending Beam Rheometer* on *Long-Term Aged* Binder for Different Blends of Binder with mesh 14 GTR.
Comparison of Binders: Mesh 14 GTR versus Mesh 30 GTR

The major testing under this project was conducted for the PG 58-28 binder modified with mesh 14 GTR. However, at a later time, evaluation was extended to mesh 30 GTR. The idea for this extended testing was to determine how the results are changed when a finer mesh GTR is utilized. However, due to time and budget constraints, rotational viscosity tests were not conducted on the binders modified with mesh 30 GTR. The practices and tests conducted at this phase of the study included short- and long-term aging, dynamic shear rheometer on unaged, short-term aged and long-term aged binder, and tests with the bending beam rheometer on the long-term aged binder.

The results shown indicate that, in general, finer mesh GTR has provided a softer unaged binder (Figure 8) and a softer long-term aged binder (Figure 11) based on the tests with DSR at high temperatures of testing. However, no significant difference is observed between mesh 30 and mesh 14 GTR for short-term aged binder (Figures 9 and 10). Nor is a significant difference observed between GTR and GTR+ VESTENAMER® modification for unaged or short-term aged binders (Figures 8, 9, and 10) when 30 mesh GTR is used. However, the VESTENAMER® did result in reduction of the stiffness for the long-term aged binder tested at 19 °C (Figure 11). This is a desirable effect in regard to fatigue resistance behavior.

Testing with the bending beam rheometer at -24 °C and -18 °C indicates that both 14 mesh GTR and 30 mesh GTR significantly reduce the binder stiffness compared with the neat
binder. However, 14 mesh GTR has a higher influence in reducing this stiffness compared with 30 mesh GTR (Figures 12 and 13). VESTENAMER® makes the reduction in stiffness even more pronounced (Figures 12 and 13). This is again a desirable effect, since it reduces the potential for low-temperature cracking. The m value shown in Figure 14 is also increased consistently for both fine and coarse mesh GTR at -18 °C testing when VESTENAMER® is added. This is also a desirable effect, since as m increases it implies better ability of the modified binder to relieve stresses (relaxing under load). However, both GTR modified and GTR+VESTENAMER® modified binders present lower m-values compared to the base asphalt at -18°C. There is not a significant difference among the m-values at -24 °C testing temperatures. Figure 15 shows that none of the binders meets requirements for m value or stiffness at -24 °C. All binders pass low-temperature requirements at -18 °C and are performance graded as PG xx -28 °C.

A comparison of results from direct tension tests is presented in Figures 16 and 17. There is no significant difference between the stress at failure for GTR-modified and GTR+VESTENAMER® modified binders when 30 mesh GTR is used. However, for 14 mesh GTR, VESTENAMER® has increased the stress at failure compared to the case where only GTR is used. In addition, for both mesh 14 and mesh 30 GTRs, using VESTENAMER® has provided higher failure strain compared with the base PG 58-28 binder. Higher strain at failure is desirable, presenting a more ductile behavior.
Figure 8. \( G*/\text{Sin} (\delta) \) for Different Blends of Unaged Binder from Tests with DSR.

Figure 9. \( G*/\text{Sin} (\delta) \) for Different Blends of Short-Term Aged Binder from Tests with DSR.
Figure 10. $G^*/\sin(\delta)$ for Different Blends of Short-Term Aged Binder from Tests with DSR.

Figure 11. $G^*.\sin(\delta)$ for Different Blends of Long-Term Aged Binder from Tests with DSR.
Figure 12. Stiffness at 60 s and -18 °C for Long-Term Aged Binder from Tests with BBR.

Figure 13. Stiffness at 60 s and -24 °C for Long-Term Aged Binder from Tests with BBR.
Figure 14. m Value at 60 s and -18 °C for Long-Term Aged Binder from Tests with BBR.

Figure 15. m Value at 60 s and -24 °C for Long-Term Aged Binder from Tests with BBR.
Figure 16. Failure Stress for Long-Term Aged Binder from Tests with Direct Tension.

Figure 17. Failure Strain for Long-Term Aged Binder from Tests with Direct Tension.
**Mixture Evaluation**

The study on the asphalt-aggregate mixture characteristics was conducted after the major part of binder testing and analysis was complete for mesh 14 GTR. One aggregate gradation was selected. This gradation was for a 19.0 mm maximum nominal size Superpave mix used as a reference mixture at NECEPT. The aggregate was selected and provided by NECEPT. The mixture modification was conducted only through dry process with mesh 14 GTR.

The mixture study was limited to three levels presented in Table 3.

<table>
<thead>
<tr>
<th>Type</th>
<th>Binder PG Grade</th>
<th>Aggregate Gradation</th>
<th>GTR % by weight of Binder</th>
<th>VESTENAMER Modifier % by weight of GTR</th>
<th># of Specimens</th>
<th>Tests</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>58-28</td>
<td>SP(1) 19.0 mm</td>
<td>0</td>
<td>0</td>
<td>6 for mix design 2 for performance tests 2 for Gmm(3)</td>
<td>Gmm, Gmb(4), RSCHT</td>
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<tr>
<td>C-1</td>
<td>58-28</td>
<td>SP 19.0 mm(2)</td>
<td>10</td>
<td>0</td>
<td>6 for mix design 2 for performance tests 2 for Gmm</td>
<td>Gmm, Gmb, RSCHT(5)</td>
</tr>
<tr>
<td>C-2</td>
<td>58-28</td>
<td>SP 19.0 mm(2)</td>
<td>10</td>
<td>4.5</td>
<td>6 for mix design 2 for performance tests 2 for Gmm</td>
<td>Gmm, Gmb, RSCHT</td>
</tr>
</tbody>
</table>

1. SP: Superpave.
2. For the mixes with GTR, an equivalent volume of #8 mesh was removed from the aggregate to provide sufficient space for GTR.
3. Gmm: Maximum Theoretical Specific Gravity.
5. RSCHT: Repeated Shear Constant Height Test.

The aggregate gradation and binder information is provided in the appendix. The mix design had provided a design binder content of 4.6 percent for the control mix and 5.1 percent for the modified mixes. Compaction of the mixes took place at the design binder content. Two specimens were prepared for each mix. However, the average resulting air voids for the control mix and the mix modified with GTR were considerably higher than the expected air void level of 4 percent. While no justification could be found for this deviation in air voids from the target values, no adjustments were applied and testing continued on the prepared specimens. The mix study was a very small part of this research, and was carried out to provide some preliminary measure of the performance of these mixes. It is highly recommended that a more elaborate study be undertaken for testing mixes with GTR+VESTENAMER® before any reasonable conclusions can be drawn regarding the mix behavior.
For preparation of the modified mixtures, the GTR was batched with the rest of the aggregate and subsequently heated as a normal batch. The gradation of the control mix was modified to provide space for the 14 mesh GTR used with the aggregate. An equivalent volume of material retained on sieve #8 was removed to allow space for the required GTR at 10 percent level. A temperature of 160 °C was used as the mixing temperature. Prior to adding the binder and mixing, the batch was thoroughly hand mixed while VESTENAMER® was being added. After the addition of the binder the entire batch was vigorously mixed in a bucket mixer for 120 s. All the prepared mix batches were cured at 135 °C for 3 hours, followed by half an hour at 160 °C before compaction. The specimens were then compacted to 75 gyrations at 160 °C and then were allowed to cool in the molds before being extruded.

After determination of the bulk density of the specimens, they were tested in the Superpave shear tester (SST). The repeated shear constant height tests were conducted for evaluation of performance of the mixes. The SST is a closed-loop feedback, servo-hydraulic system that consists of four major components: the testing apparatus, the test control unit and data acquisition system, the environmental control chamber, and the hydraulic system (Figure 18).

The equipment is capable of applying repeated loads on the specimen in both axial and horizontal directions at controlled temperatures. The responses of asphalt concrete to these loads can be used as inputs to performance prediction models or as mix design and performance evaluation criteria. The results are used to evaluate permanent deformation (rutting) and fatigue cracking susceptibilities in asphalt mixtures.

For the repeated shear test, a load cycle consists of 0.7 s, which is comprised of a 0.1-s shear load in the horizontal direction followed by a 0.6-s rest period. Test specimens are subjected to 5,000 load cycles or until the permanent shear strain reaches 5 percent.

The first step in specimen preparation is to trim the test specimen to a thickness of 50 mm. The specimen is then glued between two platens using a two-component epoxy and a gluing
device (Figure 19).

Figure 19. SST Gluing Device.

Linear variable differential transducers (LVDTs) are affixed to the specimen and measure the deformation response of the specimen to the applied testing loads (Figures 20 and 21).

Figure 20. Specimen Instrumentation for Unconfined SST Tests (Side View).
The test temperature used is $T_{\text{max}}$, which is the seven-day maximum pavement temperature at 20 mm depth. During the test, axial and shear loads and deformations are measured and recorded (Figure 22).

The tests were conducted at 52 °C, under a shear stress level of 69 KPa (10 psi) for 5,000 cycles. The results are presented in Figure 23.
Figure 23. Comparison of the Performance of Different Mixes from Shear Tests.

From Figure 23, it is clear that the smallest value for maximum permanent deformation is obtained for the mix modified by GTR+VESTENAMER® and highest is obtained for the control mix with no modification. This is an interesting finding because the GTR+V mix (the mix with GTR and VESTENAMER®) is even at a higher binder content than the control mix. However, part of this reduced deformation in the GTRV mix could be the result of lower air voids compared to the control mix. The difference in permanent deformations is large enough to indicate that most of the difference is coming from the GTR and VESTENAMER® effect rather than smaller air void level.
Observations made during the preparations of the binders indicate that the VESTENAMER® definitely reacts with the ground tire rubber. With an equal percentage of ground tire rubber the binder with and without the VESTENAMER® has a very different physical appearance. The VESTENAMER® reduces the granular appearance that is typical of asphalt cements containing unreacted ground tire rubber. This effect is more pronounced as the size of the ground tire rubber becomes smaller.

The VESTENAMER® significantly influenced the properties of the asphalt cement at the upper pavement temperatures. Generally, combination of GTR and VESTENAMER®, at 5 percent level of mesh 14 GTR, resulted in one grade increase in the high-temperature binder grade. The binder grade was bumped three levels at 10 percent level of mesh 14 GTR. The stiffness of short-term aged binder modified with 30 mesh GTR+VESTENAMER® is comparable with that modified with 14 mesh GTR+VESTENAMER®. However, for unaged binder, 30 mesh GTR did not increase the binder stiffness so much as 14 mesh GTR did.

The VESTENAMER® did not adversely affect the low-temperature properties; instead, a noticeable improvement was observed in some of the blends/properties through reduction of binder stiffness at low temperature as seen from the tests with the bending beam rheometer. This improvement was more pronounced for mesh 14 GTR+VESTENAMER® compared to mesh 30 GTR+VESTENAMER. The rate at which the binder can relieve thermal stresses, as reflected in an increase in the m-value, is also increased when VESTENAMER® is added to GTR modified binder. This is a desirable effect as obtained from the BBR tests. Results from direct tension tests also indicate that the failure strain at low temperature increases as a result of using VESTENAMER®. This is also a desirable finding.

Both GTR modified as well as GTR + VESTENAMER® modified binders showed decreased long-term aging when compared to the control binder as seen from the DSR tests on the PAV residue. In the case of 30 mesh GTR, the stiffness is even further reduced when VESTENAMER® is added. This is indeed a desirable finding and an encouraging result, but it is premature to conclude that this will result in enhanced pavement life. The long-term PAV aging test was developed for plain asphalt cement and its use for modified binders, especially those modified with crumb rubber, must be interpreted with caution.

The results with the mixtures were also very encouraging. The presence of the VESTENAMER® demonstrated improved resistance to rutting based on the results obtained from the repeated shear tests on the specimen while keeping the height of the specimen constant.

Thus, it can be clearly concluded that, based on the materials and test procedures used in this study, the addition of crumb rubber modified with VESTENAMER® should enhance pavement performance at the upper range of service temperatures where rutting is the more dominant distress mode.
APPENDIX A

AGGREGATE AND BINDER INFORMATION
Aggregate and Binder Information

Mix Type: 19-mm Coarse

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<thead>
<tr>
<th>Sieves</th>
<th>%Pass</th>
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<td>1</td>
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<td>#30</td>
<td>0.6</td>
</tr>
<tr>
<td>#50</td>
<td>0.3</td>
</tr>
<tr>
<td>#100</td>
<td>0.15</td>
</tr>
<tr>
<td>#200</td>
<td>0.075</td>
</tr>
<tr>
<td>pan</td>
<td>0</td>
</tr>
</tbody>
</table>

Percentages of Different Aggregates

A67  A8  B3  Scrn
41.8 27  28.3 2.9

Aggregate Nominal Max Size 19 mm
Aggregate Type Dolomite
Binder Grade and Source PG 58-28
Binder Source Koch Pavement

<table>
<thead>
<tr>
<th>N_{des.}</th>
<th>75</th>
<th>N_{initial}</th>
<th>7</th>
<th>N_{max}</th>
<th>115</th>
</tr>
</thead>
<tbody>
<tr>
<td>G_b (Binder Specific Gravity)</td>
<td>0.991</td>
<td>G_{ab} (Agg. Bulk Sp. Gr.)</td>
<td>2.785</td>
<td></td>
<td></td>
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