Test Report No. 48/2005

Client: Degussa AG
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45764 Marl

Project: Binder tests of bitumen modified with Road+

Subject of the Study: Production of 50/70 and 70/100 mixtures of bitumen and Road+, determination of various binder characteristics and comparison with the requirements on PmB 45 A according to TL PmB.

The report comprises 18 pages and 1 attachment
Roggentin, 11/25/2005

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1 Assignment

Heiden Labor received from Degussa AG the assignment to produce the following bitumen/Road+ mixtures:
- Bitumen 50/70 + Road+ - 95:5 wt% ratio (Mixture I)
- Bitumen 50/70 + Road+ - 90:10 wt% ratio (Mixture II)
- Bitumen 50/70 + Road+ - 85:15 wt% ratio (Mixture III)
- Bitumen 70/100 + Road+ - 90:10 wt% ratio (Mixture IV)
- Bitumen 70/100 + Road+ - 85:15 wt% ratio (Mixture V)

The bitumen was purchased from TOTAL Bitumen Deutschland GmbH from Brunsbüttel.

The bitumen/Road+ mixtures and the 50/70 and 70/100 starting bitumens were then to be tested by conventional tests:
- Needle penetration
- Ring and ball softening point
- Ductility
- Elastic recovery

and performance-oriented test methods:
- Deformation behavior (thermal stability) with a dynamic shear rheometer (DSR)
- Low-temperature behavior with the bending beam rheometer (BBR) and
- Deformation work

2 Production of the Laboratory Mixtures

The bitumen/Road+ mixtures were prepared using a laboratory mixer (low shear mixer: LSM). The Road+ was added to the hot (180-190°C) bitumen and stirred in by hand. Subsequently, the mixture was homogenized with an LSM (paddle mixer) at a medium speed and constant temperature of 180°C.

A sample mixture with a 70/100 bitumen and Road+ at a ratio of 85:15 wt% was prepared to determine the reaction time and stirring time. The viscosity was measured regularly at a temperature of 177°C (Figure 2-1). After 120 min, no significant increase in viscosity was observed. The mixing time for the subsequent mixing was determined as 120 min.
3 Determination of Physical Properties and Macroscopic Structure

The samples were investigated for their physical properties and macroscopic structure at room temperature according to DIN EN 1425:

- Surface appearance: dark, dull black uneven surface
- Foreign substances: none
- Consistency: solid
- Odor: mild rubber-bitumen odor

4 Sample Preparation

The samples were prepared for the test according to DIN EN 12594. The samples were prepared immediately after production of the binders modified with Road+.
5 Test Results

The following Tables 5-1 show the tested properties and their results for the 50/70 bitumen modified with Road+ and the starting bitumen as well as the requirements for a PmB 45 A according to TL PmB (technical delivery conditions for ready-to-use polymer-modified bitumen, 2001 version). The results relate solely to the tested samples. The test values are arithmetic means of at least 2 individual values.
<table>
<thead>
<tr>
<th>Property</th>
<th>Unit</th>
<th>Tested according to</th>
<th>Test Value</th>
<th>Required value according to TL PmB (PmB 45 A)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Mixture</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Viscosity at 177°C</td>
<td>mPa*s</td>
<td>-</td>
<td>900</td>
<td>1100</td>
</tr>
<tr>
<td>Needle penetration (100 g, 5 sec, 25°C)</td>
<td>mm</td>
<td>DIN EN 1426</td>
<td>55</td>
<td>41</td>
</tr>
<tr>
<td>Ring and ball softening point</td>
<td>°</td>
<td>DIN EN 1427</td>
<td>49.6</td>
<td>56.2</td>
</tr>
<tr>
<td>Elastic recovery in thread tear test</td>
<td>%</td>
<td>DIN V 52021-1</td>
<td>-</td>
<td>65</td>
</tr>
<tr>
<td>Bent beam rheometer (BBR) at -16°C</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Stiffness</td>
<td>MPa</td>
<td>AASHTO TP 1 ¹</td>
<td>142</td>
<td>128</td>
</tr>
<tr>
<td>m value</td>
<td></td>
<td></td>
<td>0.380</td>
<td>0.363</td>
</tr>
<tr>
<td>Deformation work to minimum ductility</td>
<td>J</td>
<td>DIN 52013 and TL PmB Appendix B</td>
<td>-</td>
<td>0.231</td>
</tr>
<tr>
<td>Deformation work to thread tear</td>
<td></td>
<td></td>
<td>0.079</td>
<td>0.231</td>
</tr>
<tr>
<td>Ductility</td>
<td>cm</td>
<td>DIN 52013</td>
<td>140 (no thread tear)</td>
<td>13</td>
</tr>
<tr>
<td>Dynamic shear rheometer (DSR) at 60°C²</td>
<td>Pa</td>
<td>AASHTO TP 5 and TL PmB Appendix C</td>
<td>2916</td>
<td>8768</td>
</tr>
<tr>
<td>Phase shift angle</td>
<td>°</td>
<td></td>
<td>82.1</td>
<td>59.6</td>
</tr>
</tbody>
</table>

¹) American Association of State Highway and Transportation Officials
²) Temperature control with electric hotplate (ETC) and air chamber
The following net diagram contains the results of the binder studies for the 50/70 bitumen modified with Road+. Shown here is the percentage (relative) of improvement (positive) or worsening (negative) of the properties in comparison to the requirements on a PmB 45 A according to TL PmB.

Elastische Rückstellung = Elastic Recovery
Duktilität = Ductility
Kraftduktilität = Ductility under load
Steifigkeit = Rigidity

![Net Diagram](image)

**Figure 5-1:** Test results for the 50/70 bitumen modified with Road+ compared with the requirements for a PmB 45 A according to TL PmB

The following Table 5-2 contains the tested properties and their results for the 70/100 bitumen modified with Road+.
<table>
<thead>
<tr>
<th>Property</th>
<th>Unit</th>
<th>Tested according to</th>
<th>Test Value</th>
<th>Required value according to TL PmB (PmB 45 A)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sample</td>
<td></td>
<td>70/100</td>
<td>70/100</td>
<td></td>
</tr>
<tr>
<td>Mixture</td>
<td></td>
<td></td>
<td>70/100 (10% Road+)</td>
<td>70/100 (15% Road+)</td>
</tr>
<tr>
<td>Viscosity at 177°C</td>
<td>mPa*s</td>
<td>-</td>
<td>IV</td>
<td>V</td>
</tr>
<tr>
<td>Needle penetration (100 g, 5 sec, 25°C)</td>
<td>0.1 mm</td>
<td>DIN EN 1426</td>
<td>76</td>
<td>41</td>
</tr>
<tr>
<td>Ring and ball softening point</td>
<td>°C</td>
<td>DIN EN 1427</td>
<td>46.2</td>
<td>58.2</td>
</tr>
<tr>
<td>Elastic recovery in thread tear test</td>
<td>%</td>
<td>DIN V 52021-1</td>
<td>-</td>
<td>67</td>
</tr>
<tr>
<td>Bent beam rheometer (BBR) at -16°C</td>
<td>Stiffness</td>
<td>MPa</td>
<td>139</td>
<td>117</td>
</tr>
<tr>
<td></td>
<td>m value</td>
<td></td>
<td>107</td>
<td>≤ 300</td>
</tr>
<tr>
<td></td>
<td></td>
<td>AASHTO TP 1(^1))</td>
<td>0.423</td>
<td>0.348</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.319</td>
<td>-</td>
</tr>
<tr>
<td>Ductility under load at 25°C</td>
<td>Deformation work to minimum ductility</td>
<td>DIN 52013 and TL PmB Appendix B</td>
<td>-</td>
<td>0.327</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.670</td>
<td>≥ 1</td>
</tr>
<tr>
<td></td>
<td>Deformation work to thread tear</td>
<td></td>
<td>0.042</td>
<td>0.327</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0.670</td>
<td>-</td>
</tr>
<tr>
<td></td>
<td>Ductility</td>
<td>cm</td>
<td>140 (no thread tear)</td>
<td>17</td>
</tr>
<tr>
<td></td>
<td></td>
<td>DIN 52013</td>
<td>14</td>
<td>14</td>
</tr>
<tr>
<td>Dynamic shear rheometer (DSR) at 60°C(^2))</td>
<td>Complex shear module</td>
<td>AASHTO TP 5 and TL PmB Appendix C</td>
<td>1870</td>
<td>9789</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>15337</td>
<td>≥ 7000</td>
</tr>
<tr>
<td></td>
<td>Phase shift angle δ</td>
<td></td>
<td>85.4</td>
<td>55.2</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>50.8</td>
<td>≤ 75</td>
</tr>
</tbody>
</table>

3) American Association of State Highway and Transportation Officials
4) Temperature control with electric hot plate (ETC) and air chamber
The following net diagram contains the results of the binder studies for the 70/100 bitumen modified with Road+. Shown here is the percentage (relative) of improvement (positive) or worsening (negative) of the properties in comparison to the requirements on a PmB 45 A according to TL PmB.

Elastische Rückstellung = Elastic Recovery  
Duktilität = Ductility  
Kraftduktilität = Ductility under load  
Steifigkeit = Rigidity

6 Evaluation of the Test Results

Needle Penetration
With regard to needle penetration, all five tested mixtures are within the specification for a PmB 45 A according to TL PmB. In comparison to Mixtures II and III (starting bitumen 50/70) and Mixtures IV and V (70/100 starting bitumen), we see that the penetration values are lower for the harder 50/70 starting bitumen when the same amounts of Road+ are added. A larger amount of Road+ causes a reduction in penetration. As a result, we infer that needle penetration decreases with increasingly hard starting
bitumen and increasing Road+ proportions. Compared to the starting bitumens, a reduction in penetration is provided by the Road+ modification.

Nadelpenetration = Needle Penetration
Anforderung an ein PmB 45 A gemäß TL PmB = Requirements for a PmB 45 A according to TL PmB
Anteil Road+ [M.-%] = Proportion of Road+ (wt%)

Figure 6-1: Test results for needle penetration

**Ring and Ball Softening Point**
The ring and ball values of Mixtures I and IV are within the specification for a PmB 45 A according to TL PmB. The softening points of Mixtures II, III and V, however, are too high. This is due to the influence of the starting bitumen. In comparison with Mixtures II and III (50/70 starting bitumen) and Mixtures IV and V (70/100 starting bitumen), we see that the softening points for the harder 50/70 starting bitumen are higher when the same amounts are added. A larger amount of Road+ increases the ring and ball values. As a result, we see that the ring and ball softening point increases as the starting bitumen becomes harder and as the Road+ proportion increases.

In comparison to the starting bitumens, the Road+ modification causes an increase in the softening point.
Erweichungspunkt Ring und Kugel = Ring and ball softening point
Anforderung an ein PmB 45 A gemäß TL PmB = Requirements for a PmB 45 A according to TL PmB
Anteil Road+ [M.-%] = Proportion of Road+ (wt%)

Unlike their starting bitumens, all three tested mixtures have, with values in the range of 65-83\% (at thread tear), an elastic recovery in excess of the minimum requirement of 50\% for a PmB 45 A according to TL PmB. We see from the following diagram that both an increase in Road+ proportion and the harder 50/70 starting binder have positive effects on the elastic recovery.
Elastische Rückstellung = Elastic recovery
Anforderung an ein PmB 45 A gemäß TL PmB = Requirements for a PmB 45 A according to TL PmB
Anteil Road+ [M.-%] = Proportion of Road+ (wt%)

Figure 6-3: Test results for elastic recovery

Ductility
At values of 10-17 cm, all mixtures with Road+ fall distinctly below the minimum requirement of 40 cm for a PmB 45 A according to TL PmB. We infer from the following diagram that the higher Road+ proportion and the softer 70/100 starting bitumen have a positive effect on ductility. In comparison to the starting bitumens, the Road+ modification produces a distinct decrease in ductility.
Duktilität = Ductility
Anforderung an ein PmB 45 A gemäß TL PmB = Requirements for a PmB 45 A according to TL PmB
Anteil Road+ [M.-%] = Proportion of Road+ (wt%)

![Figure 6-4: Test results for ductility](image)

**Ductility Under Load**

Only Mixture III, with a value of 1,028 Joules, exceeds the minimum requirement of 1 Joule (deformation work to minimum ductility) for a PmB 45 A according to TL PmB. Because the binder modified with Road+ does not reach the minimum ductility of 40 cm, the deformation work for these binders is reported up to thread tear. The ductility under load was influenced by the higher Road+ proportion and the harder 50/70 starting binder.

In comparison with the starting bitumens, the Road+ modification produces a distinct increase in deformation work.

The force-deformation diagrams (Appendix 1) show that, in comparison to the starting bitumens and with increasing Road+ proportions, considerably higher forces are needed to pull the samples apart.
Formänderungsarbeit = Deformation work
Anforderung an ein PmB 45 A gemäß TL PmB = Requirements on a PmB 45 A according to TL PmB
Anteil Road+ [M.-%] = Proportion of Road+ (wt%)

Figure 6-5: Test results for ductility under load

Low-Temperature Behavior
The behavior of the bitumen at low temperatures (-16°C) is characterized with the BBR-bent beam rheometer, which determines the stiffness $S$ and relaxation, expressed by the $m$ value of the binder. The lower the stiffness and the higher the $m$ value, the better is the low-temperature behavior of bitumen and the asphalt made from it. For all of the samples, the stiffness is less than the maximum value of 300 MPA for a PmB 45 A according to TL PmB. We can infer from the results that the softer 70/100 starting binder improves the low-temperature behavior. Further, a positive influence on the low-temperature behavior is observed with increasing Road+ proportions due to the lower stiffness, but a negative influence is observed due to the lower $m$ value. In comparison to the starting bitumens, the stiffness and the $m$ value decrease. That is, the stiffness is positively influenced and the relaxation negatively influenced by the Road+ modification.
Steifigkeit S bei -16°C = Stiffness S at -16°C
Anforderung an ein PmB 45 A gemäß TL PmB = Requirements for a PmB 45 A according to TL PmB
Anteil Road+ [M.-%] = Proportion of Road+ (wt%)

Deformation Behavior (Thermal Stability)
The complex shear modulus $G^*$ determined with the DSR – Dynamic Shear Rheometer – in the upper service temperature range at 60°C characterizes the thermal stiffness of the binder. High $G^*$ values result in a high thermal stability of the asphalt and less rutting. The values of the complex shear modulus, in the range of 8768-28213 Pa, are in some cases far above the value of at least 7000 Pa according to TL PmB for a PmB 45 A. We can infer from the results that the stiffer 50/70 starting binder and the higher Road+ proportion have a positive effect on thermal stability.
Komplexer Schubmodul $G^*$ bei 60°C = Complex Shear Modulus $G^*$ at 60°C
Anforderung an ein PmB 45 A gemäß TL PmB = Requirements on a PmB 45 A according to TL PmB
Anteil Road+ [M.-%] = Proportion of Road+ (wt%)

Figure 6-7: Test results for complex shear modulus at 60°C
The phase shift angle $\delta$ determined with the DSR at 60°C, characterized the elastic or viscous behavior of the binder. A small phase shift $\delta$ characterize an elastic binder (high deformation and fatigue resistance). The values of the phase shift angles $\delta$ at 60°C, lying in the range of 44.9-59.6°, are distinctly lower than the required value of max 75°C according to TL PmB for a PmB 45 A. These results show that the stiffer 50/70 starting binder and the higher Road+ proportion have a positive effect on the phase shift angle. In comparison to the starting bitumens, and the complex shear module increases and the phase shift angle decreases; that is, the Road+ modification produces an appreciable improvement in the deformation and fatigue resistance.

Figure 6-8: Test results for the phase shift angle at 60°C
The evaluation of the test results was based solely on the results of the tested samples (starting bitumen and SBS proportions).

HEIDEN LABOR
für Baustoff- und Umweltpreßung GmbH

Zielke Mahnke
Official in Charge Study Director
Test Result No. 48/2005

Appendix 1

Test Results for Ductility Under Load
Appendix 1

To Test Report No. 48/2005

Prüfung der Formänderungsarbeit mit Hilfe der Kraftduktilität = Test of Deformation work Based on Ductility Under Load
Anhang = Appendix
Probe = Sample
Labor Nr. = Laboratory No.
Mindestduktilität = Min. ductility
Formänderungsarbeit bis Mindestduktilität = Deformation work to min. ductility
Duktilität = Ductility
Formänderungsarbeit bis zum Fadenriss = Deformation work to thread tear
Prüftemperatur = Test temperature
Bemerkung = Comment
kein Fadenriss = no thread tear