# Test Report No. 48/2005

Client:	Degussa AG Paul-Baumann-Straβe 1 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			

This test report may be reproduced only in its full version. Reproduction and copying in excerpts require our approval. The results relate solely to the tested specimen.

Roggentin, 11/25/2005

# **Table of Contents**

- 1 Test assignment
- 2 Production of the laboratory mixtures
- 3 Determination of physical properties and macroscopic structure
- 4 Sample preparation
- 5 Test results
- 6 Assessment of test results

# Appendices

Appendix 1: Test results – ductility under load

# 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:

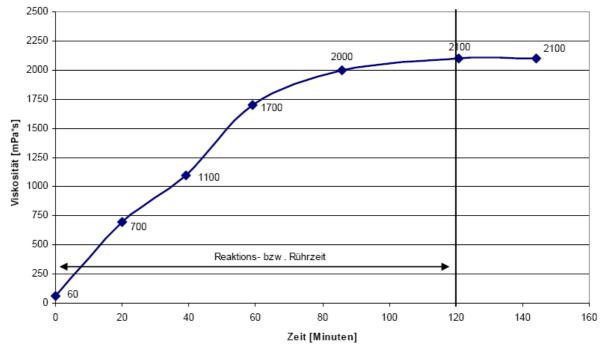
- Deformation behavior (thermal stability) with a dynamic shear rheometer (DSR)
- Low-temperature behavior with the bending beam rheometer (BBR) and
- Deformation work

## 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.

Viskosität = Viscosity Reaktions-bzw.Rührzeit = Reaction time and stirring time Zeit [Minuten] = Time [Minutes]



Viskosität bei = Viscosity at

Figure 2-1: Viscosity of a 70/100 bitumen-Road+ mixture at a ratio of 85:15 wt% as a function of reaction/stirring time at a constant temperature of 180°C.

#### 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.

Prop	perty	Unit	Tested according to		Tes	Required value according to TL PmB		
Sample				50/70	50/70 (5% Road+)	50/70 (10% Road+)	50/70 (15% Road+)	(PmB 45 A)
Mixture				-	Ι	II	III	
Viscosity at 177°C		mPa*s	-	-	900	1100	2900	-
Needle penetration (100 g, 5 sec, 25°C)		0.1 mm	DIN EN 1426	55	41	34	29	20 - 60
Ring and ball	softening point	C°	DIN EN 1427	49.6	56.2	64.2	72.8	55.0 - 63.0
Elastic recovery in thread tear test		%	DIN V 52021-1	-	65	78	83	≥ 50
Bent beam	Stiffness	MPa		142	128	120	114	<u>&lt;</u> 300
rheometer (BBR) at - 16°C	m value	-	AASHTO TP 1 <sup>1)</sup>	0.380	0.363	0.341	0.293	-
Ductility under load at 25°C	Deformation work to minimum ductility	J	DIN 52013 and TL PmB Appendix B	-	0.231	0.524	1.028	≥ 1
	Deformation work to thread tear			0.079	0.231	0.524	1.028	-
	Ductility	cm	DIN 52013	140 (no thread tear)	13	12	10	<u>≥</u> 40
Dynamic shear rheometer	Complex shear modulus	Ра	AASHTO TP 5 and TL PmB Appendix C	2916	8768	13753	28213	≥ 7000
(DSR) at $60^{\circ}C^{2)}$	Phase shift angle	0		82.1	59.6	53.5	44.9	<u>&lt;</u> 75

American Association of State Highway and Transportation Officials
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

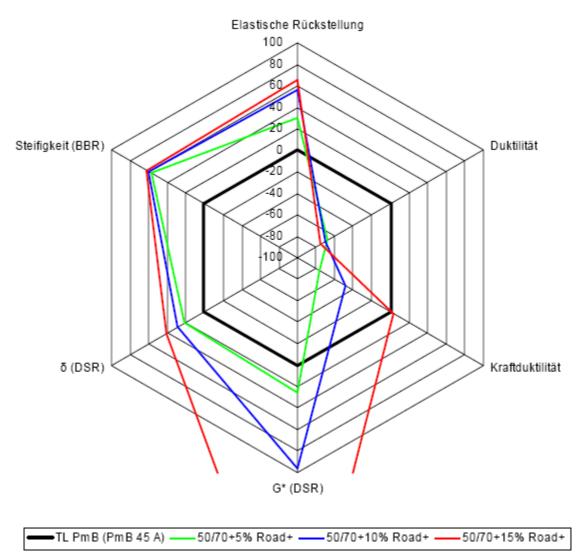


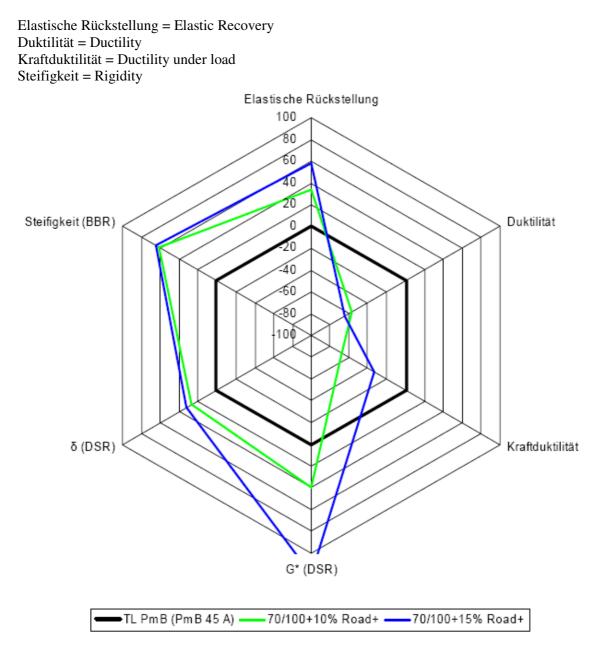
Figure 5-1: Test results for the 50/70 bitumen modified with Road+ compared with the requirements for a PmB 45 A according to TL PmB

The following Table 5-2 contains the tested properties and their results for the 70/100 bitumen modified with Road+.

Prop	perty	Unit	Tested according to	Test Value			Required value according to TL PmB (PmB 45 A)
Sample				70/100	70/100 (10% Road+)	70/100 (15% Road+)	,
Mixture				-	IV	V	
Viscosity	Viscosity at 177°C		-	60	700	2300	-
Needle penetration (100 g, 5 sec, 25°C)		0.1 mm	DIN EN 1426	76	41	37	20 - 60
Ring and ball s	Ring and ball softening point		DIN EN 1427	46.2	58.2	65.6	55.0 - 63.0
Elastic recovery in thread tear test		%	DIN V 52021-1	-	67	79	≥ 50
Bent beam	Stiffness	MPa		139	117	107	<u>&lt;</u> 300
rheometer (BBR) at - 16°C	m value	-	AASHTO TP 1 <sup>1)</sup>	0.423	0.348	0.319	-
Ductility under load at 25°C	Deformation work to minimum ductility	J	DIN 52013 and TL PmB Appendix B	-	0.327	0.670	<u>≥</u> 1
	Deformation work to thread tear			0.042	0.327	0.670	-
	Ductility	cm	DIN 52013	140 (no thread tear)	17	14	≥ 40
Dynamic shear	Complex shear module	Ра	AASHTO TP 5 and TL PmB	1870	9789	15337	≥ 7000
rheometer (DSR) at $60^{\circ}C^{2)}$	Phase shift angle δ	o	Appendix C	85.4	55.2	50.8	<u>≤</u> 75

3) American Association of State Highway and Transportation Officials4) 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.



## 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 (70/100starting 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 gemä $\beta$  TL PmB = Requirements for a PmB 45 A according to TL PmB

Anteil Road+ [M.-%] = Proportion of Road+ (wt%)

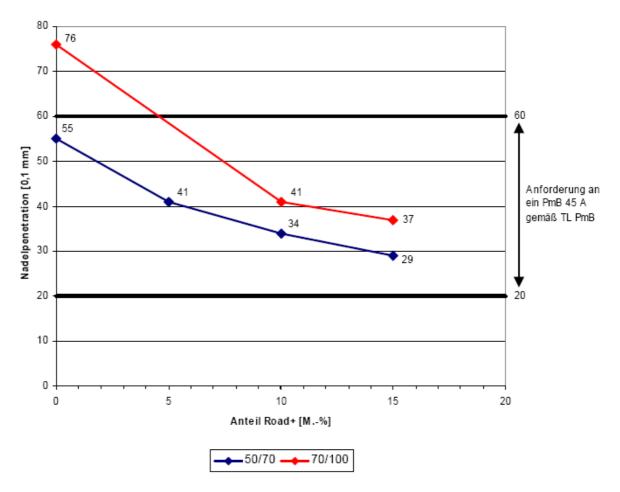


Figure 6-1: Test results for needle penetration

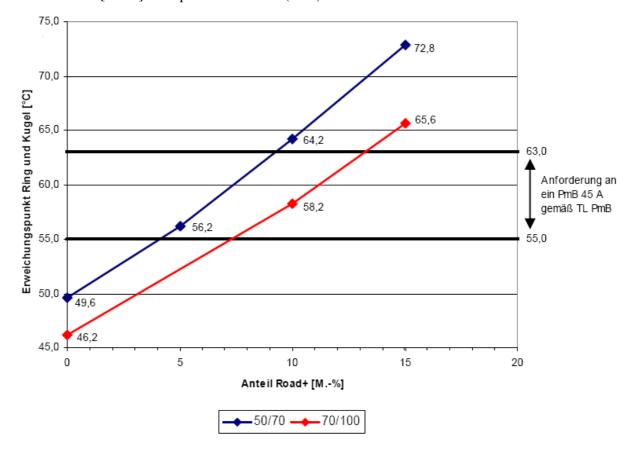
#### **Ring and Ball Softening Point**

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 gemä $\beta$  TL PmB = Requirements for a PmB 45 A according to TL PmB



Anteil Road+ [M.-%] = Proportion of Road+ (wt%)

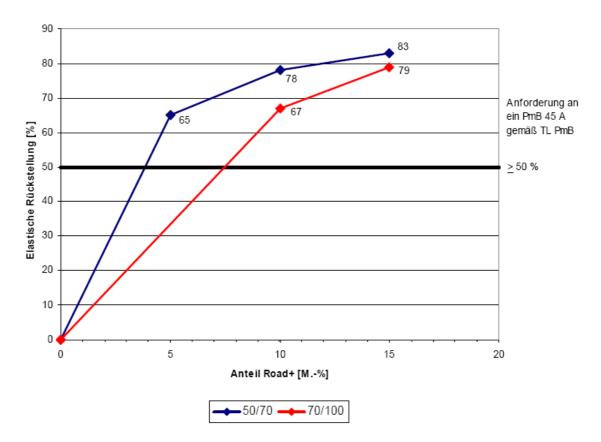
Figure 6-2: Test results for ring and ball softening point

## Elastic Recovery

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 gemä $\beta$  TL PmB = Requirements for a PmB 45 A according to TL PmB



Anteil Road+ [M.-%] = Proportion of Road+ (wt%)

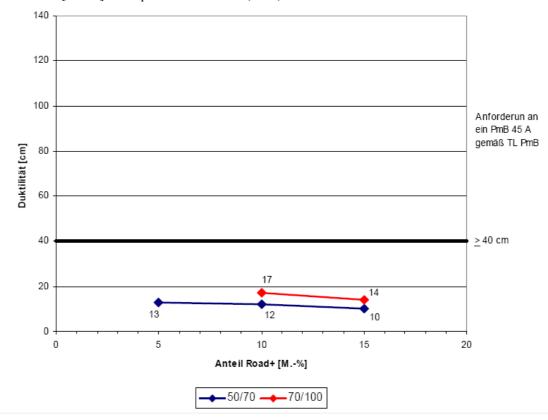
Figure 6-3: Test results for elastic recovery

#### 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 gemä $\beta$  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

#### Ductility Under Load

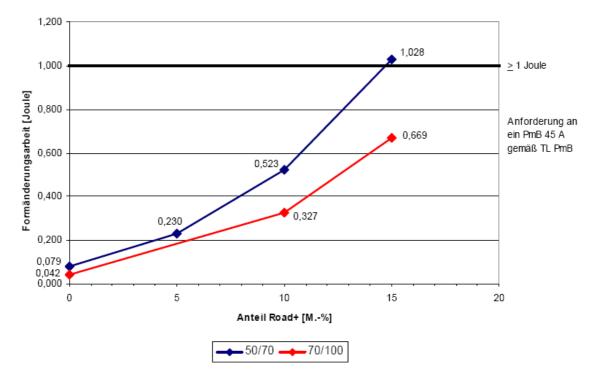
Only Mixture III, with a value of 1,028 Joules, exceeds the minimum requirement of 1 Joule (deformation work to minimum ductility) for a PmB 45 A according to TL PmB. Because the binder modified with Road+ does not reach the minimum ductility of 40 cm, the deformation work for these binders is reported 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 gemä $\beta$  TL PmB = Requirements on a PmB 45 A according to TL PmB



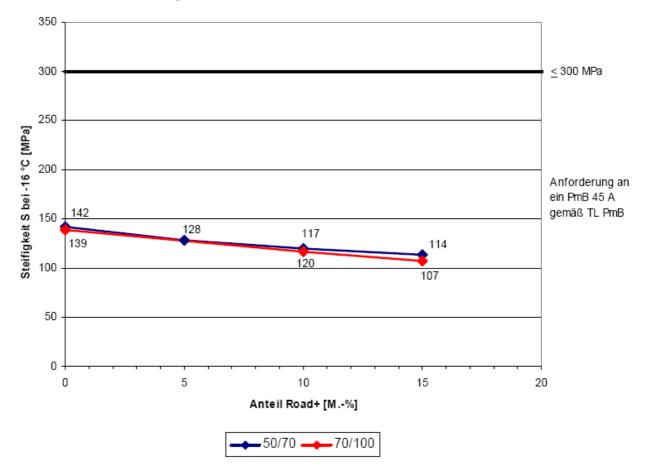
Anteil Road+ [M.-%] = Proportion of Road+ (wt%)

Figure 6-5: Test results for ductility under load

#### Low-Temperature Behavior

The behavior of the bitumen at low temperatures  $(-16^{\circ}C)$  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  $-16^{\circ}$ C = Stiffness S at  $-16^{\circ}$ C

Anforderung an ein PmB 45 A gemä $\beta$  TL PmB = Requirements for a PmB 45 A according to TL PmB



Anteil Road+ [M.-%] = Proportion of Road+ (wt%)

Figure 6-6: Test results for stiffness at -16°C

## 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 $60^{\circ}$ C = Complex Shear Modulus G\* at $60^{\circ}$ C Anforderung an ein PmB 45 A gemä $\beta$ TL PmB = Requirements on a PmB 45 A according to TL PmB

Anteil Road+ [M.-%] = Proportion of Road+ (wt%)

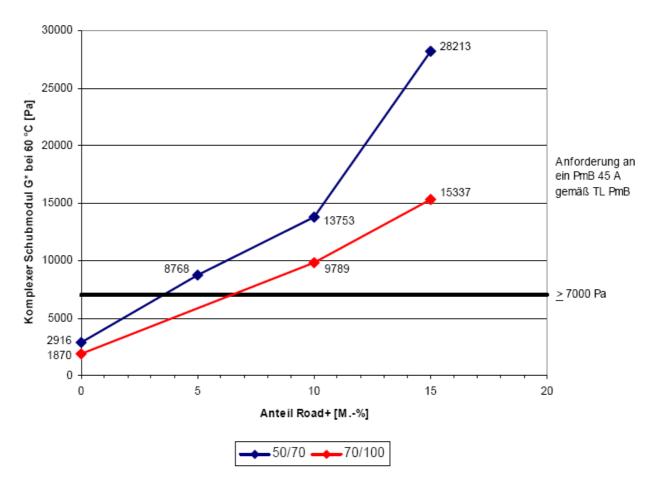
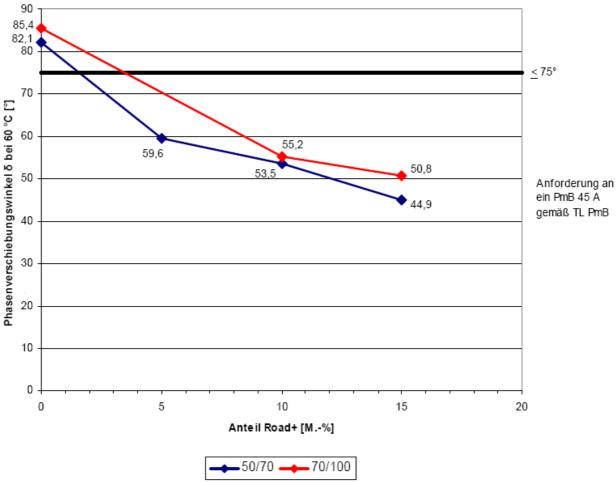


Figure 6-7: Test results for complex shear modulus at 60°C

Phasenverschiebungswinkel  $\delta$  bei 60°C = Phase shift angle  $\delta$  at 60°C Anforderung an ein PmB 45 A gemä $\beta$  TL PmB = Requirements on a PmB 45 A according to TL PmB



Anteil Road+ [M.-%] = Proportion of Road+ (wt%)

Figure 6-8: Test results for the phase shift angle 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.

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 Umweltprüfung GmbH

Zielke Official in Charge Mahnke 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