Bitumen quality recording - A German approach

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Digital Object Identifier (DOI): dx.doi.org/10.14311/EE.2016.339

ABSTRACT

The requirements of the majority of bitumen in Europe are described in EN 12591 and EN 14023. European regulation allows each country to adopt a suitable description of these in their local norms. In Germany the requirements for EN 12591 and 14023 normed bitumen have been transferred into TL Bitumen (TL means technical conditions of delivery) as the national guidance paper for these standards.
In an attempt to improve the description of bitumen quality, the German government has approved a paper with additional requirements to be measured by both supplier and user. The paper, named ARS 11/2012, contains investigations with DSR and BBR. It was implemented into the TL Bitumen in 2013. At the time of implementation most of the test laboratories in Germany were not used to the methods.
In this paper we describe the results from a two-step comparative study with participants along the whole supply chain. In the first step, measurements were done with the authorities original test procedures. Based on these results the test descriptions were modified. In the second step, improvement of the reproducibility of the methods was investigated. A benefit for the participants was to gain more experience with these kinds of equipment and test methods. In this paper the results and conclusions are discussed.

Keywords: Performance testing, Performance based standards

1. Introduction

The requirements for the majority of bitumen in Europe are described in EN 12591 [1] and EN 14023 [2]. CEN rules allow each country to adopt a suitable description of these in their local standards. In Germany the requirements for bitumen described in EN 12591 and 14023 have been transferred into TL Bitumen [3] (TL means technical conditions for delivery) as the national guidance paper for these standards.

In an attempt to improve durability of asphalt roads, the German road authorities have approved a program with additional requirements to be measured by both supplier and user. The program, introduced as ARS 11/2012 [4], includes some changes in asphalt requirements (referring to TL Asphalt-StB 07 and ZTV Asphalt-StB 07,Ausgabe 2013) and additional tests for bitumen requirements (according to TL Bitumen-StB 07, Ausgabe 2013). While all changes are transferred into German asphalt guidance papers and additional tests are introduced into TL Bitumen since 2013 most of the German test laboratories are in process of developing the relevant competences. It is seen as necessary that reliable test results are found at all points of the whole supply chain

This paper deals with the additional tests required for bitumen. The results from a two-step comparative study with participants along the whole supply chain are described. In the first step, measurements were performed following the original test procedures. Based on these results the test descriptions particularly have been rendered towards more precision. In the second step, improvement of the reproducibility of the methods was investigated. A benefit for the participants was to gain more experience with an often new kind of equipment and often unfamilliar test methods. In this paper the results and conclusions are summarized.

2. Bitumen Quality

Within this paper the expression “Bitumen Quality” has been used from a practical perspective of a bitumen supplier only. There is to consider, how safe the handover of the product can be guaranteed regarding to the specification dealt with. So there is interest in traceability and repeatability / reproducibility of conventional and performance related test methods.

Based on EN 12591 for paving grade bitumen and EN 14023 for polymer modified binders the bitumen delivery specifications in Germany are transferred into TL Bitumen as the national guidance paper for these standards. Tables 1 and 2 show the properties and the margins of this national guidance paper. Predominantly both tables define the requirements for the conventional tests.

Table 1: Requirements for paving grade bitumen

Property	Unit	Method	Bitumen				
			20/30	30/45	50/70	70/100	160/220
Needle penetration at 25°C	1/10 mm	DIN EN 1426	20 to 30	30 to 45	50 to 70	70 to 100	160 to 220
Softening point ring and ball	°C	DIN EN 1427	55 bis 63	52 bis 60	46 bis 54	43 bis 51	35 bis 43
Flash point	°C	DIN EN ISO 2592	≥ 240	≥ 240	≥ 230	≥ 230	≥ 220
Solubility	M.-%	DIN EN 12592	≥ 99,0	≥ 99,0	≥ 99,0	≥ 99,0	≥ 99,0
Fraass breaking point	°C	DIN EN 12593	-	≤ -5	≤ -8	≤ -10	≤ -15
Resistance against hardening (RTFOT)		DIN EN 12607-1					
Retained penetration after RTFOT	%		≥ 55	≥ 53	≥ 50	≥ 46	≥ 37
Change of softening point after RTFOT	°C		≤ 8	≤ 8	≤ 9	≤ 9	≤ 11
Change of mass after RTFOT	M.-%		≤ 0,5	≤ 0,5	≤ 0,5	≤ 0,8	≤ 1,0
Additional methods for gathering of experience							
DSR Temperature sweep 30°C to 90°C	-	TL Bitumen Chapter 5.3	No requirement but value has to be documented				
BBR – Flexural creep stiffness	-	TL Bitumen Chapter 5.4					

Table 2: Requirements for elastomer modified bitumen

Property	Unit	Method	PmB				
			120/200-40 A	45/80-50 A	25/55-55 A	10/40-65 A	40/100-65 A
Needle penetration at 25°C		DIN EN 1426	120 bis 200	45 bis 80	25 bis 55	10 bis 40	40 bis 100
Softening point ring and ball	°C	DIN EN 1427	≥ 40	≥ 50	≥ 55	≥ 65	≥ 65
Force ductility	J/cm²	DIN EN 13589	≥ 2	≥ 2	≥ 3	≥ 2	≥ 3
Temperature for force ductility		DIN EN 13703	at 0°C	at 5°C	at 5°C	at 10°C	at 5°C
Flash point	°C	DIN EN ISO 2592	≥ 220	≥ 235	≥ 235	≥ 235	≥ 235
Fraass breaking point	°C	DIN EN 12593	≤ -20	≤ -15	≤ -10	≤ -5	≤ -15
Elastic recovery at 25°C	%	DIN EN 13398	≥ 50	≥ 50	≥ 50	≥ 50	≥ 70
Storage stability		DIN EN 13399					
Difference in softening point	°C	DIN EN 1427	≤ 5	≤ 5	≤ 5	≤ 5	≤ 5
Stability against hardening (RTFOT)							
Change of mass after RTFOT	M.-%	DIN EN 12607-1	≤ 0,5	≤ 0,5	≤ 0,5	≤ 0,5	≤ 0,3
Retained penetration after RTFOT	%	DIN EN 1426	≥ 60	≥ 60	≥ 60	≥ 60	≥ 60
Maximum increase in softening point after RTFOT	°C	DIN EN 1427	≤ 8	≤ 8	≤ 8	≤ 8	≤ 8
Minimum increase in Softening point after RTFOT	°C	DIN EN 1427	≤ 2	≤ 2	≤ 2	≤ 2	≤ 5
Elastic recovery at 25°C after RTFOT	%	DIN EN 13398	≥ 50	≥ 50	≥ 50	≥ 50	≥ 50
Additional methods for gathering of experience							
Force ductility	-	TL Bitumen Chapter 5.2	No requirement but value has to be documented				
DSR Temperature sweep 30°C to 90°C	-	TL Bitumen Chapter 5.3					
BBR – Flexural creep stiffness	-	TL Bitumen Chapter 5.4					

All tests with requirements are based on empirical test methods. The additional methods making use of Dynamic Shear or Bending Beam Rheometers provide fundamental values which do not depend on the test conditions. These test methods are well-known to the bitumen producers and some other experts but not necessarily everyone involved in the supply chain.

With the ARS 11/2012 the German road authorities have introduced a program with minimum requirements for test frequency for a range of methods into the TL Bitumen. The bitumen suppliers have to test paving grade bitumen 30/45, 50/70, 70/100, 160/220 and polymer modified binder grades 25/55-55, 10/40-65 and 40/100-65 at least once every quarter of a year and upload them into a data base. The data base with all results from the whole supply chain will be evaluated in a research program at the Ruhr-Universität Bochum. Asphalt producers' test frequency and test scope are described in table 3.

Table 3: Test methods and test frequency for the asphalt producer in ARS 11/2012

<table>
<tr><th rowspan="2">Property</th><th rowspan="2">Method</th><th colspan="2">Bitumen</th><th rowspan="2">Test frequency</th></tr>
<tr><th>NPG</th><th>PmB</th></tr>
<tr><td>Needle penetration at 25°C</td><td>DIN EN 1426</td><td>X</td><td>X</td><td rowspan="2">Once every 300 tons</td></tr>
<tr><td>Softening point ring and ball</td><td>DIN EN 1427</td><td>X</td><td>X</td></tr>
<tr><td>DSR Temperature sweep 30°C to 90°C</td><td>TL Bitumen – Chapter 5.3</td><td>-</td><td>X</td><td rowspan="3">Once every 1.500 tons, started from 50 tons in the current year</td></tr>
<tr><td>MSCRT at 60°C</td><td>AL MSCR-Prüfung (DSR)</td><td>-</td><td>X</td></tr>
<tr><td>BBR - Flexural creep stiffness</td><td>TL Bitumen – Chapter 5.4</td><td>-</td><td>X</td></tr>
<tr><td>Stability against hardening (RTFOT)
- Needle penetration at 25°C
- Softening point ring and ball</td><td>DIN EN 12607-1
DIN EN 1426
DIN EN 1427</td><td>X</td><td>X</td><td rowspan="2">Once every 900 tons, started from 50 tons in the current year</td></tr>
<tr><td>Stability against hardening after RTFOT and PAV
- Needle penetration at 25°C
- Softening Point Ring and Ball</td><td>DIN EN 12607-1
DIN EN 14769
DIN EN 1426
DIN EN 1427</td><td>X</td><td>X</td></tr>
</table>

3. Comparative study

For the handover of products on a specification basis it is self-evident to be interested in evaluating the characteristics\` precision. Therefore a transfer of factory production control\`s reproducibility – if carried out in the past on voluntary basis – into a precision all over the supply chain has been requested.

In 2013 a comparative study has been started in the German market. There were 16 participants representing the whole supply chain from a binder supplier over the paving industry, authorities, universities and independent test laboratories. The intention of the study was to not to question the usefulness of the data collection itself but how much the values vary between those involved and whether it is possible to improve these variations. The tested binders were 50/70 and elastomer modified 25/55-55 supplied by BP. Step 1 of the program is described in table 4. Sample preparation has been carried out following EN 58 and EN 12594.

Table 4: Test methods for the comparative study

Test methods				Number of results
	Fresh binder	After RTFOT	After RTFOT + PAV	
Conventional methods	Softening point ring an ball (EN 1427)	Softening Point Ring and Ball (EN 1427)	Softening Point Ring an Ball (EN 1427)	2
	Needle Penetration (EN 1426)	Needle Penetration (EN 1426)	Needle Penetration (EN 1426)	3
	Force ductility (EN 13589 + EN 13703)	-	-	3
	Dynamic viscosity at 60°C (EN 12596)	-	-	1
	Kinematic viscosity at 135°C (EN 12595)	-	-	1
Performance related methods	Complex shear modulus and phase angle (EN 14770 and AL DSR-Prüfung)	-	-	3
	Multiple stress and creep recovery test (MSCRT) (AL MSCR-Prüfung)	-	-	3
	Flexural creep stiffness (EN 14771)	-	-	3

The reason to carry out step 2 was to check whether comparability could be improved by defining some test conditions more precisely. Furthermore each participant could verify if conditions rendered more precisely would improve the repeatability of their results (data gathered in step 1). Therefore the ARBIT recommendation [5] for conventional tests and an updated instruction sheet for DSR [6] issued by FGSV have been used in step 2. All participants were asked to consider compliance with the test conditions and the calibration of their test devices required there. The test material has been provided from the same batch as for step 1.

4. Results

While participants in the annual ARBIT Round Robin tests could have gathered a lot of experience with conventional tests for decades, the corresponding results from the comparative study have been excluded in this paper. The focus in this paper is on results of performance related test methods only, e.g. those from DSR and BBR tests. Figure 1 shows the results for 50/70 in blue, those for 25/55-55 are orange colored.

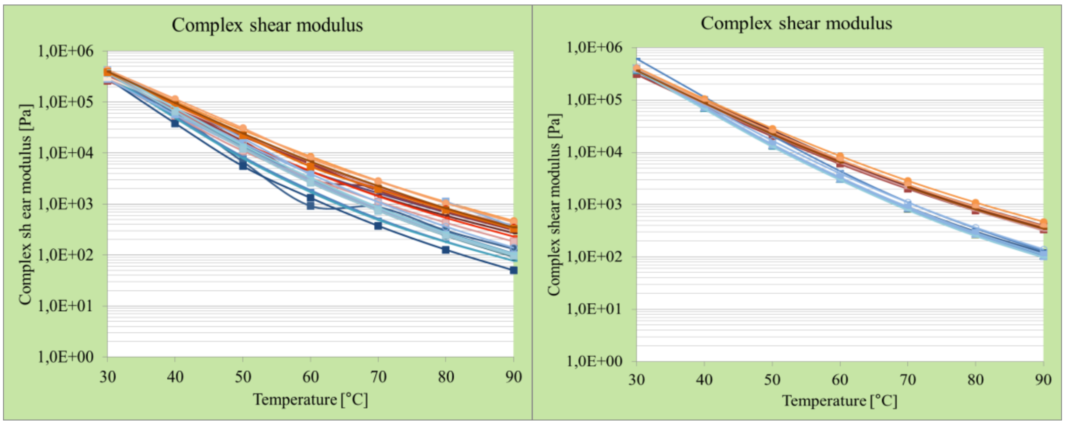

Figure 1: Complex shear modulus (G*) – Step 1 (left) and step 2 (right)

If we compare the course of the curves of step 1 and 2 it can be observed that the variation is much smaller in step 2. For step 1 the curves of 50/70 and 25/55-55 partially overlap. Up to about 50 °C the complex shear modulus of both binder types overlaps too. From temperatures of about 50°C onwards the complex shear modulus of these two binders spreads.

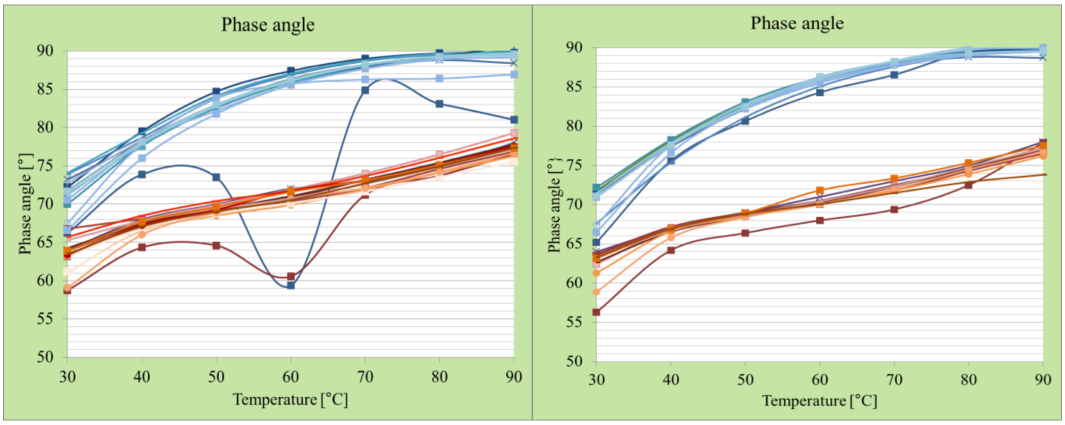

Figure 2: Phase angle (δ) – Step 1(left) and step 2 (right)

In figure 2 on the left we can see two sets of data which apparently drop out. Comparing the rest of the curves we can exactly differentiate the two different types of binder. Between step 1 and step 2 it can be observed that the variation in range for the single binder slightly decreases analog to the complex shear modulus.

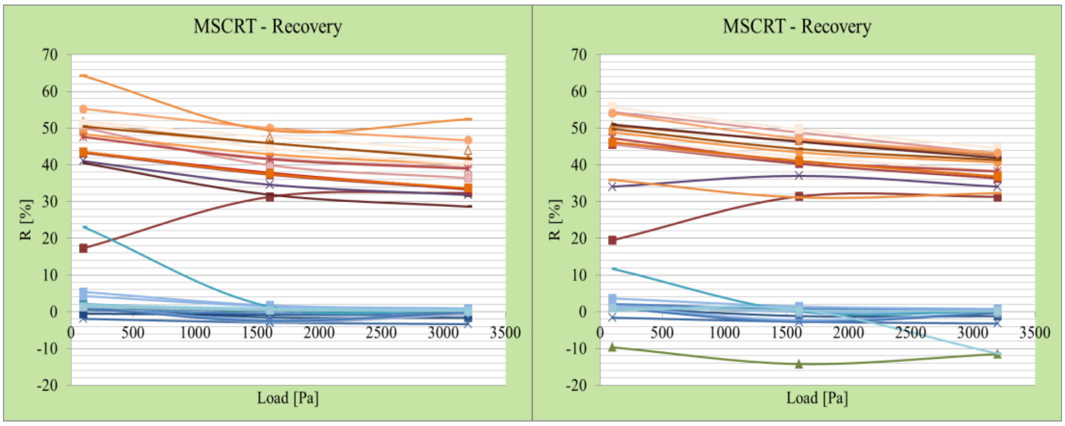

Figure 3: MSCRT Recovery – Step 1 left - step 2 right

Figure 3 shows the results of the MSCR test. Between step 1 and step 2 it can be observed that there is no significant improvement of reproducibility of the test data.

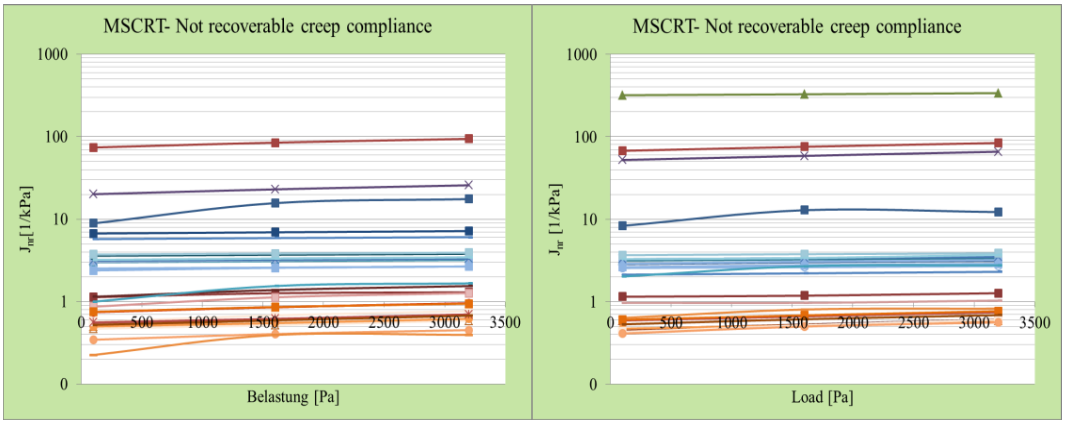

Figure 4: MSCRT Not recoverable creep compliance – Step 1 left - step 2 right

Figure 4 shows significant differences between the single measurements. This presentation is not useless to compare data sets. If we delete obvious outliers, we get the curves in figure 5.

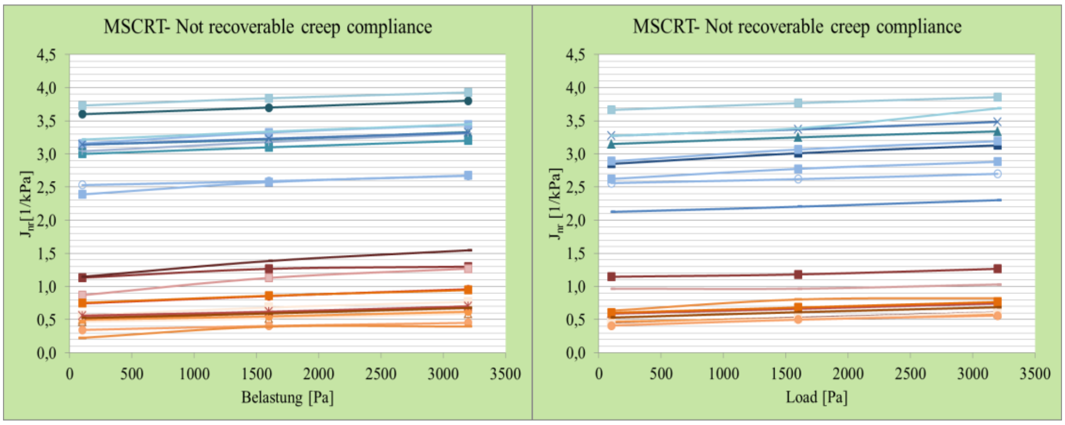

Figure 5: MSCRT Not recoverable creep compliance (corrected) – Step 1 left - step 2 right

Between step 1 and step 2 no significant improvement of the reproducibility of the test data can be observed. Figure 6 and 7 show the measured flexural creep stiffness.

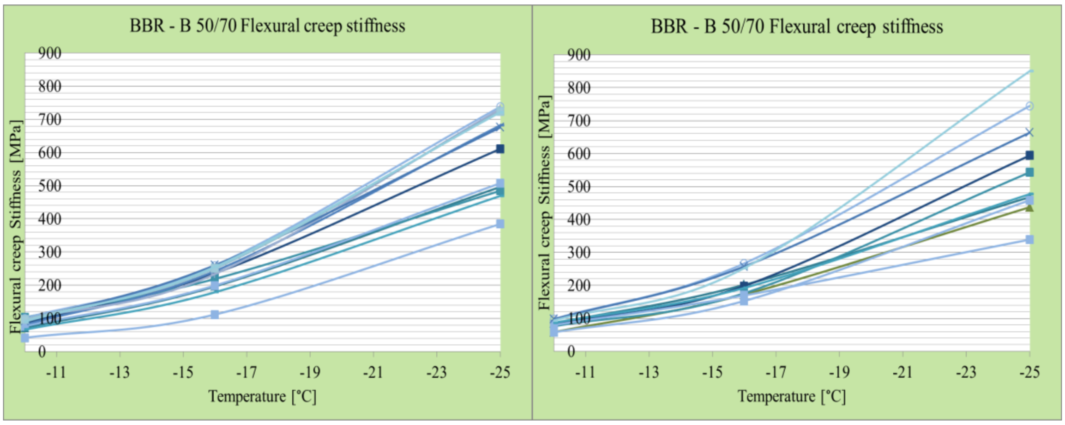

Figure 6: 50/70 Flexural creep stiffness – Step 1 left - step 2 right

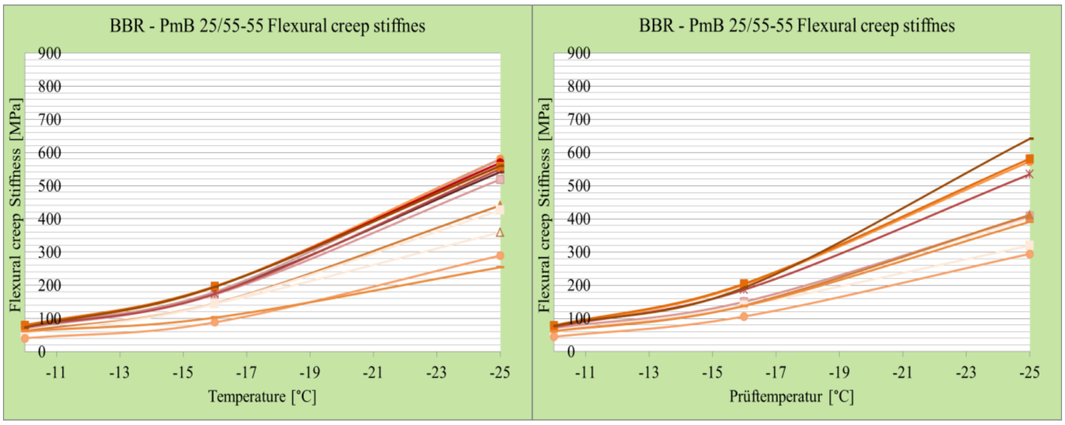

Figure 7: 25/55-55 Flexural creep stiffness – Step 1 left - step 2 right

Comparing the curves of both binders between step 1 and step 2 no significant improvement of reproducibility can be found.

5. Conclusion and recommendation

The comparative study reflects the current capacity regarding new testing`s precision and cooperation within the supply chain.

This study proves that the reproducibility of performance related test methods over the whole supply chain can be optimized. Based on these results it could be useful to perform a statistical analysis of the data to get the reproducibility and repeatability of the performance related test methods.

Analytical infrastructure in the German market has developed since the data collection started. As one could never be completely sure when using specifications based on conventional tests only, we are well advised to train new test methods wherever possible.

6. Acknowledgements

Special thanks go out to all participants in the study. There were a lot of resources needed to generate this data base.

7. References

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