Road and Transportation Research Association

Working Group on Asphalt Pavements

FGSV

Test description for determining the phase transition temperature of viscosity modified binders using a Dynamic Shear Rheometer (DSR) – Part 3: Testing with constant shear rate

AL DSR-Prüfung (konstante Scherrate)

W 1

Edition 2016/Translation 2018

ISBN 978-3-86446-166-8

Road and Transportation Research Association

Working Group on Asphalt Pavements

FGSV

Test description
for determining the phase transition temperature of viscosity modified binders using a Dynamic Shear Rheometer (DSR)
– Part 3: Testing with constant shear rate

AL DSR-Prüfung (konstante Scherrate)

W 1

Edition 2016/Translation 2018

Working Group on Asphalt Pavements
Working Committee on Binders
Subcommittee on Bitumen and Modified Bitumen

Chair:

Dr.-Ing. Tobias Hagner, Brunsbüttel

Members:

Dipl.-Ing. Alexander Alisov, Braunschweig
Dipl.-Ing. Mohammed Ammadi, Illkirch-Graffenstaden (F)
Dipl.-Ing. Rudolf Barth, Leipzig
Dipl.-Ing. Arnd Bartholomäus, Hannover
Dipl.-Ing. Lars Driske, Diegem (B)
BDir. Dipl.-Ing. Jens Ehmke, Krumbach
Dipl.-Ing. Mario Kowsky, Soest
Dipl.-Ing. Dipl. Umweltwiss. Tatjana Kunsic, Großmehring
Dipl.-Ing. Heinz-Peter Louis, Essen
Dipl.-Ing. Carsten Oelkers, Hamburg
Dipl.-Ing. Robert M. Riegler, Wesseling
Dipl.-Ing. Gabriele Sauerhering, Halberstadt
Dipl.-Lab. Chem. Silke Sielaff, Bergisch Gladbach
Dipl.-Ing. (FH) Ludger Vienenkötter, Gelsenkirchen
Dr.-Ing. Bernd Wallner, Munich
Dipl.-Ing. Anne Wittenberg, Karlsruhe
M.Sc. Kathrin Zumsande, Pinneberg

Preliminary remark:

The ‘Test description for determining the phase transition temperature of viscosity modified binders using a Dynamic Shear Rheometer (DSR), Part 3: Testing with constant shear rate (AL DSR-Prüfung (konstante Scherrate)), Edition 2016’, was drafted by the Road and Transportation Research Association’s Subcommittee on Bitumen and Modified Bitumen and finalised by the Working Committee on Binders (chaired by Dr.-Ing. Anja Sörensen).

Table of contents

page

1 Introduction 5

2 General remarks 5

3 Abbreviations and definitions 5

4 Test principles 6

5 Test equipment 7

5.1 Dynamic Shear Rheometer (DSR) 7

5.2 Moulds 8

5.3 Oven 8

6 Preparation of the Dynamic Shear Rheometer (DSR) . . . 8

7 Sample preparation 8

7.1 General remarks 8

7.2 Preparation of test specimens and storage conditions 9

8 Conducting the test 9

8.1 Placing the test specimen in the rheometer 9

8.2 Test conditions 10

9 Expression of results 10

10 Precision 11

11 Test report 11

Annex A: How to use a graph to determine the phase transition temperature of viscosity modified binders T_{PT} 12

Annex B: How to calculate the phase transition temperature of viscosity modified binders T_{PT} 13

Annex C: Technical standards and specifications 15

1 Introduction

This document describes a procedure for determining the phase transition temperature of viscosity modified bituminous binders. Testing is carried out using a Dynamic Shear Rheometer (DSR), with a parallel plate test geometry in rotational mode.

2 General remarks

This test description outlines how to test viscosity modified bituminous binders in accordance with the 'Recommendations on the classification of viscosity-modified binders' (E KvB).

Note: The use of this European Standard can involve hazardous materials, operations and equipment. This European Standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this European Standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

3 Abbreviations and definitions

For general definitions, see DIN EN 12597 'Bitumen and bituminous binders – Terminology'.

Viscosity modified paving grade bitumen: Paving grade bitumen that complies with DIN EN 12591 whose rheological properties are modified using suitable organic additives in a way that the handling temperatures can be reduced.

Viscosity modified polymer-modified bitumen: Polymer-modified bitumen (PmB) that complies with DIN EN 14023 whose rheological properties are modified using suitable organic additives in a way that the handling temperatures can be reduced.

Rate of measurement ($\dot{n}$): Number of values measured over a specified period, given in s^{-1}.

Phase transition temperature (T_{PT}): Temperature at which viscosity modified binders that have been modified using suitable organic additives demonstrate discernibly different material behaviour as a result of shear stress, given in °C, than non viscosity modified bituminous binders.

Note: The index of the phase transition temperature T_{PT} stands for the English term 'phase transition'. The phase transition temperature can only be determined if the binder contains suitable organic additives.

Shear rate (γ): Relative speed of the plates depending on the distance between the plates, given in s^{-1}.

Note: Other terms used in litterature for 'shear rate' are shear speed, shear gradient, or speed gradient in the gap.

Shear stress (τ): Quotient between shear force and shear surface, given in Pa.

Temperature rate ($\dot{T}$): Change in temperature over a defined period, given in K/s.

4 Test principles

This test procedure is used to determine the temperature at which viscosity modified binders in accordance with E KvB demonstrate discernibly different material behaviour when exposed to shear load.

Parallel plate systems with plate diameters of 25 mm and a gap of 1 mm are used.

The rheometer is operated in rotational mode.

The test is conducted at a constant shear rate of 2 s^{-1}.

During the test, the temperature is decreased over a temperature range of 150 °C to 70 °C. The temperature rate is –0,02 K/s (Fig.1).

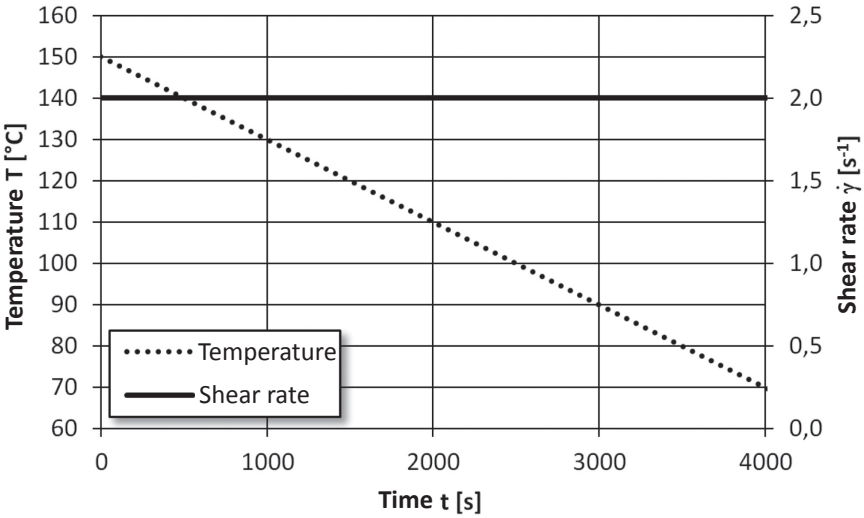


Fig. 1: Test parameters for temperature and shear rate

During the test, record the shear stress at a measurement rate of 1 s^{-1}.

The test result is the temperature at which an over-proportional increase in shear stress is determined on a double logarithmic scale (Fig. 2).

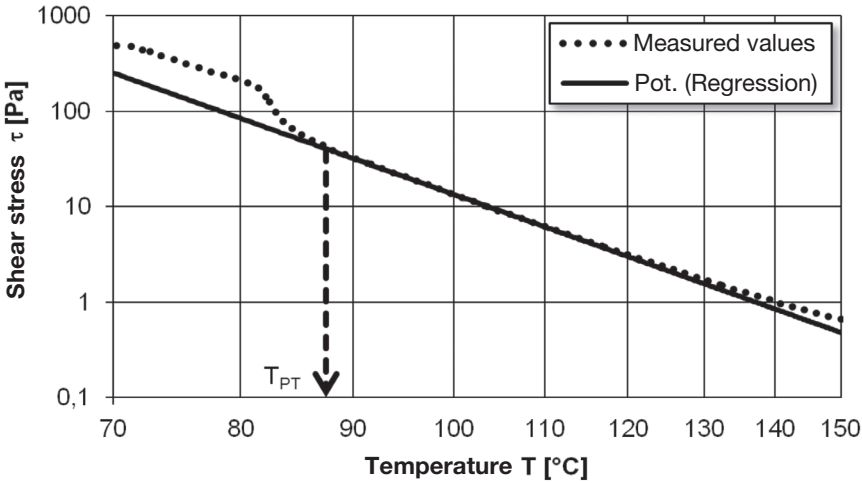


Fig. 2: Example of data measured and the determination of phase transition temperature T_{PT}

At least two individual valid results must be obtained on two separate test specimens. The test result is the mean value of these individual results.

5 Test equipment

Standard laboratory equipment as well as:

5.1 Dynamic Shear Rheometer (DSR)

A Dynamic Shear Rheometer that complies with DIN EN 14770 with parallel plates of a diameter of 25 mm and a temperature control system that allows temperatures to be controlled with an accurary of ± 0,3 °C over the course of the entire test. In order to avoid a temperature gradient between the plates, the temperature control system must include both plates.

In execptional cases, if the temperature control system does not include both plates, the non-heated plate may be heated by contact with the heated plate.

5.2 Moulds

Moulds that comply with AASHTO T 315, made of silicone or a similar material to which the test specimen does not adhere. The use of release agents is not permitted because they could impair the adherence of the specimen to the rheometer plates.

5.3 Oven

Oven capable of being controlled at temperatures between 50 °C and 200 °C with an accuracy of ± 5 °C.

6 Preparation of the Dynamic Shear Rheometer (DSR)

Prepare the rheometer according to the manufacturer's instructions. Select the test configuration and gap in accordance with this test description.

Note: The rheometer and the temperature control system should be checked, adjusted, and, if necessary, calibrated at regular intervals (monitoring of measuring and test equipment). It is recommended that test results are regularly compared by measuring a control substance such as polyisobutene (PIB) and documenting the outcome on a mean value control card. Oppanol B10N (BASF) has proven to be a suitable control substance. Guidance on how to keep a mean value control card is given in Annex B of the 'Test description for determining the deformation behaviour of bitumen and bituminous binders using a dynamic shear rheometer (DSR) – Part 1: Conducting the test with a temperature sweep (AL DSR-Prüfung (T-Sweep)).

Carefully prepare the rheometer by cleaning the rheometer plates with a suitable solvent and a soft cleaning cloth or paper.

7 Sample preparation

7.1 General remarks

ATTENTION! This test description covers the handling of equipment and bituminous binders at very high temperatures. When handling very hot bitumen, always wear suitable protective gloves and goggles and avoid all contact with exposed skin.

Take samples in accordance with DIN EN 58 and prepare them in accordance with DIN EN 12594.

Avoid unnecessarily long heating of the test sample. The heating times given in DIN EN 12594 should be considered maximum times before one or more test samples are taken. Homogenise the laboratory test sample before taking test samples. Protect the test samples with a cover against the admission of air and place them in the oven, which is heated to between 180 °C and 200 °C. Do not exceed 200 °C.

The samples may only be reheated twice.

The following reheating times for test samples must be adhered to:

– 50 g to 100 g: max. 30 min;

– 100 g to 500 g: max. 60 min;

– 500 g to 1000 g: max. 120 min.

7.2 Preparation of test specimens and storage conditions

When the bitumen has reached the required temperature, stir the sample with a suitable tool to ensure the required homogeneity.

Pour the test sample into the moulds, avoiding air bubbles.

Use a new test specimen for each test in the Dynamic Shear Rheometer.

Note: It is recommended that all specimens are prepared at once.

Cover the test specimens and allow them to cool to ambient temperature.

The prepared test specimens must rest for at least 12 h, but no longer than 36 h.

To ensure demoulding with minimum deformation, the test specimens may be placed for a maximum of 30 minutes in a fridge where the temperature has been set to between 5 °C and 10 °C. Demould and transfer the test specimen to the rheometer immediately after cooling.

Note: In order to avoid contamination of the test specimen surface by the skin, wear clean gloves to demould and place the test specimen in the DSR.

Note: If the sample is stored in the fridge, water may condensate on the cold test specimen when it is removed from the fridge, thereby impairing the adhesion of the test specimen and the plates. Ensure that there is no condensation on the test specimen (visual check) before putting it in the rheometer.

8 Conducting the test

8.1 Placing the test specimen in the rheometer

In order to ensure that the test specimen adheres to the plates, preheat the plates of the rheometer to (100 ± 5) °C. Maintain this temperature for at least 5 minutes.

Now insert the test specimen in the pre-heated system.

At the temperature described above, bring the plates of the rheometer to a gap of between 1,025 mm and 1,050 mm, and keep them at this temperature for at least 5 minutes.

Note: If the test specimen does not fill the entire gap, stop the test, remove the test specimen and start again with a new specimen.

Trim any excess bitumen using a suitable tool that has been preheated to no more than 90 °C.

Move the plates to the testing gap of 1,00 ± 0,01 mm.

The time between the insertion of the test specimen and establishment of the required gap should not exceed 10 minutes.

8.2 Test conditions

Operate the rheometer in rotational mode at a constant shear rate of 2 s^{-1}.

Maintain a constant gap of (1,00 ± 0,01) mm throughout the test.

The test specimen must be allowed to reach a state of thermal equilibrium. To this end, once the starting temperature of 150 °C has been reached, allow (5 ± 1) min for equilibrium to be reached.

The test begins at a starting temperature of 150 °C and ends at 70 °C. During the test, decrease the test temperature continuously at a rate of 0,02 K/s.

Note: The time needed to reach a constant temperature rate depends on the equipment being used and may take several minutes.

Note: Due to the specified temperature range and the stipulated temperature rate, the test lasts 4.000 s.

Throughout the entire test, record the shear stress to within exactly 1 Pa at a measurement rate of 1 s^{-1}.

9 Expression of results

Determine the individual phase transition temperature values in °C, rounded to 0,5 °C. The phase transition temperature is the temperature at which the shear stress over temperature increases over proportionally on a double-logarithmic scale.

The permissible difference of individual values for the phase transition temperature is 2 K.

Individual phase transition temperature values can be determined either graphically or by calculation. See appendices A and B for guidance on both methods.

Give the results of phase transition temperature as the arithmetic mean of the individual values, in °C and rounded off to 1,0 °C.

10 Precision

The precision of this test procedure has not yet been determined.

11 Test report

The test report must shall reference to this test description and shall contain at least the following information:

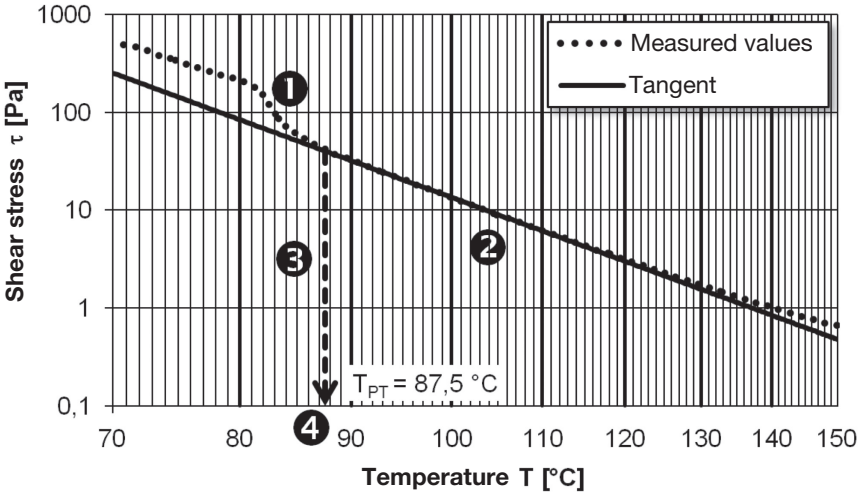
- type and grade of bitumen, including information on conditioning stage, as well as the full identification of the tested sample;
- the date of the test;
- individual values in °C to within exactly 0,5 °C as well as the test result for the phase transition temperature T_{PT} in °C to within exactly 1,0 °C;
- evaluation method used;
- any deviation from this test description and the reason for it.

Annex A

(informative)

How to use a graph to determine the phase transition temperature of viscosity modified binders T_{PT}

Plot the shear stress against temperature on a graph with a double logarithmic scale. Apply a tangent to the curve in the upper temperature range. At the point at which the shear stress deviates from the tangent, drop a perpendicular and read the phase transition temperature of the viscosity modified binder T_{PT}.



1 Plot the shear stress against temperature on a graph with a double logarithmic scale.

2 Apply a tangent to the curve in the upper temperature range.

3 Drop a perpendicular at the point at which the tangent deviates from the measured values.

4 Read the phase transition temperature T_{PT} to within exactly 0,5 °C.

Fig. A1: Using a graph to determine the phase transition temperature T_{PT}

Annex B

(informative)

How to calculate the phase transition temperature of viscosity modified binders T_{PT}

When calculating the phase transition temperature, a reference range with an almost linear relationship between log(τ)/log(T) is defined. Subsequently, the temperature at which a specified deviation of the curve of measured values from the regression calculated in the reference range occurs is determined.

Abbreviations and definitions

Reference range: Measured values within the temperature range (120,0 ± 0,1) to (135,0 ± 0,1) °C.

Regression range: Section of measured values at a temperature of T <120 °C over a temperature range of (2,0 ± 0,1) K each with approx. 100 measured values.

Exponential function: Mathematical function $\tau(T) = a \cdot e^{b \cdot T}$

Gradient: Coefficient b of the exponential function, which describes the gradient on a double logarithmic scale.

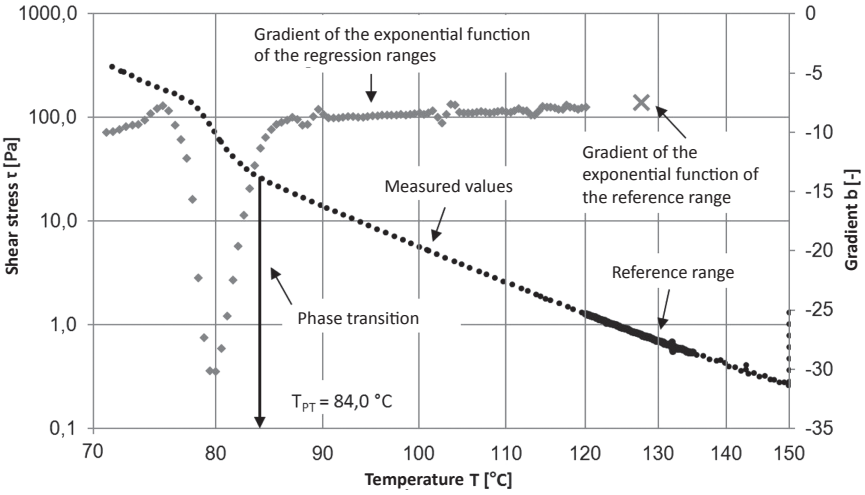


Fig. B1: How to calculate the phase transition temperature TPT of viscosity modified binders

Map the reference range using regression and an exponential function and determine the coefficient b.

Regression ranges with a temperature span of (2,0 ± 0,1) K each are specified. The first regression range comprises the temperature range (120,0 ± 0,1) °C to (118,0 ± 0,1) °C. Each subsequent regression range is shifted by ΔT = (–0,5 ± 0,1) K so that it overlaps with the previous regression range over a temperature range of (1,5 ± 0,1) K. Consequently, the second regression range covers the temperature range (119,5 ± 0,1) °C to (117,5 ± 0,1).°C, the third temperature range (119,0 ± 0,1) °C to (117,0 ± 0,1) °C. Each regression range is mapped using an exponential function by means of regression, the coefficient b of the exponential function is determined and set in relation to the exponential function coefficients b in the reference range.

In the range of the phase transition temperature, the shear stress rises overproportionally and the gradient in the regression ranges fall. The temperature at which the coefficient b of a regression range falls more than 40% below the coefficient b of the exponential function of the reference range for the first time is the phase transition temperature T_{PT} to within exactly 0,5 °C.

Note: A calculation tool for the phase transition temperature of viscosity modified binders, based on an Excel table is available free of charge on the website of FGSV Verlag.

Technical standards and specifications

DIN	DIN EN 58	Bitumen and bituminous binders – Sampling bituminous binders	1)
	DIN EN 12591	Bitumen and bituminous binders – Specifications for paving grade bitumens	1)
	DIN EN 12594	Bitumen and bituminous binders – Preparation of test samples	
	DIN EN 12597	Bitumen and bituminous binders – Terminology	1)
	DIN EN 14023	Bitumen and bituminous binders – Specification framework for polymer-modified bitumens	1)
FGSV	AL DSR-Prüfung (T-Sweep)	Test description for determining the deformation behaviour of bitumen and bituminous binders using a dynamic shear rheometer (DSR) – Part 1: Conducting the test with a temperature sweep (FGSV 722)	2)
	E KvB	Recommendations for the classification of viscosity modified binders (FGSV 727)	2)
	TL Bitumen-StB	Technical delivery terms for paving grade bitumen and ready-to-use polymer-modified bitumen (FGSV 794)	2)
AASHTO	AASHTO T 315	Standard Method of Test for Determining the Rheological Properties of Asphalt Binder Using a Dynamic Shear Rheometer (DSR), 2012	3)

Sources of supply:

1) **Beuth Verlag GmbH**
Address: Burggrafenstraße 6, 10787 Berlin, Germany
Tel.: +49 (0)30 26 01-13 31, Fax: +49 (0)30 26 01-12 60
e-mail: kundenservice@beuth.de, Internet: www.beuth.de

2) **FGSV Verlag GmbH**
Address: Wesselinger Straße 17, 50999 Köln, Germany
Tel.: +49 (0)2236 38 46 30, Fax: +49 (0)2236 38 46 40
e-mail: info@fgsv-verlag.de, Internet: www.fgsv-verlag.de

3) **American Association of State Highway Transportation Officials**
Internet: www.transportation.org

All of the FGSV publications listed above can also be obtained in digital form for the FGSV Reader and are included in the comprehensive subscription service 'FGSV – Technisches Regelwerk – Digital

Remarks on the system of technical publications of the FGSV

R stands for regulations:

These publications either specify the technical design or realization (R1) or give recommendations on the technical design or realization (R2).

W stands for information documents:

These publications represent the current state-of-the-art knowledge and define how a technical issue shall be practicably dealt with or has already been successfully dealt with.

Category R1 indicates 1st category regulations:

R1-publications contain the contractual basis (Additional Technical Conditions of Contract and Guidelines, Technical Conditions of Delivery and Technical Test Specifications) as well as guidelines. They are always coordinated within the FGSV. R1-publications – in particular if agreed on as integral part of the contract – have a high binding force.

Category R2 indicates 2nd category regulations:

R2-publications contain information sheets and recommendations. They are always coordinated within the FGSV. Their application as state-of-the-art technology is recommended by the FGSV.

Category W1 indicates 1st category documents of knowledge:

W1-publications contain references. They are always coordinated within the FGSV but not with external parties. They represent current state-of-the-art knowledge within the respective responsible boards of the FGSV.

Category W2 indicates 2nd category documents of knowledge:

W2-publications contain working papers. These may include preliminary results, supplementary information and guidance. They are not coordinated within the FGSV and represent the conception of an individual board of the FGSV.

Production and sales:
FGSV Verlag GmbH
Wesselinger Straße 15-17, 50999 Köln, Germany
Tel: 0 22 36 / 38 46 30 · Fax: 0 22 36 / 38 46 40
Internet: www.fgsv-verlag.de
ISBN 978-3-86446-166-8

W 1

721 E/8/18