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## **Analysis of 4-mm DSR tests: calibration, sample preparation, and evaluation of repeatability and reproducibility**

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# **Analysis of 4-mm DSR tests: calibration, sample preparation, and evaluation of repeatability and reproducibility**

Rheological characterization methods are important when it comes to a performance-based selection system for bituminous binders for road applications. Standardized methods exist for the determination of bitumen rheological behaviour based on a dynamic shear rheometer (DSR) at intermediate and high service temperatures and a bending beam rheometer (BBR) at low temperatures. Recently, a dynamic shear rheometer with 4 mm parallel plates (4-mm DSR), has been proposed by Western Research Institute (WRI), as a promising method to determine the rheological behaviour at low temperatures. Clear advantages of the 4-mm DSR are related to the small sample size, especially important for recovered and aged binders, and the ability to work with adequate stresses at very low temperatures. Furthermore, if 4-mm DSR can be used to assess the low temperature behaviour of bitumen, then the performance of a binder in the whole range of service temperatures, could be assessed by just one equipment, a DSR. This paper focuses on the challenges related to 4-mm DSR tests regarding equipment preparation, with a specific attention to the temperature assessment and calibration. Furthermore, a sample preparation method, assuring good adhesion to both plates is proposed and the repeatability and reproducibility of 4-mm DSR tests is evaluated.

Keywords: dynamic shear rheometer, DSR, low temperature, repeatability, reproducibility, 4-mm parallel plates

## **1. Introduction**

Rheological characterization methods are extremely important to develop a performance-based selection system for bituminous binders for road applications. In Europe, standardized methods exist for the determination of bitumen rheological behaviour. For example, EN 14770 describes test procedures for the determination of complex modulus and phase angle based on a dynamic shear rheometer (DSR) using a parallel plate geometry, focusing on 25 mm and 8 mm plates diameters. These plate diameters cover intermediate and high service temperatures, typically between 5 °C to

85 °C. They enable to evaluate the bitumen resistance to rutting and to fatigue cracking. Furthermore EN 14771 describes the use of a bending beam rheometer (BBR) suitable to test the bitumen behaviour at low service temperatures (from 0 °C to -30 °C) and its resistance to low temperature cracking.

BBR focuses on two criteria: the flexural stiffness (S) and the change of this stiffness over loading time (the m-value) at a specific loading time of 60 s. The m-value criterion is based on the idea that a high m-value leads to a faster relaxation of the thermal stresses induced at low temperature (Lytton et al., 1993). The questionable usefulness of m-value criterion has been discussed in detail by Marasteanu (2004) and Marasteanu and Basu (2004). The BBR criteria, S and m-value, were initially developed for neat binders, it has been shown that these may underestimate the performance of modified binders (Bouldin et al., 1999; Dongré, Button, Kluttz, & Anderson, 1997; Kluttz & Dongré, 1997). The BBR method needs a lot of bitumen: one test beam contains approximately 15 g, and to determine the limiting temperatures, at least two temperatures need to be tested, requiring at least two beams at each temperature.

Many tests and methods have been developed to evaluate bitumen low temperature properties. But, they have one or more disadvantages, e.g. data have a low repeatability and/or reproducibility, results strongly depend on the specimen geometry and are highly affected by physical hardening, results do not relate to field behaviour, the equipment is expensive, and etc. (Gražulytė & Vaitkus, 2017). A dynamic shear rheometer with 4 mm parallel plates (4-mm DSR), proposed by Western Research Institute (WRI) in 2015, seems like a promising method (Farra, Sui, Salmans, & Qin, 2015; C. Sui, Farrar, Tuminello, & Turner, 2010; Changping Sui, Farrar, Harnsberger, Tuminello, & Turner, 2011; Laukkanen, 2017; Lu, Uhlback, & Soenen, 2017). The main advantage is that it requires only a small amount of sample, 25 mg of bitumen is

needed for one specimen. This becomes significant when evaluating recovered or aged bitumen. A DSR is more versatile compared to a BBR with regard to parameters like the temperature and frequency range or the stress and strain range. It also allows a follow up of stiffness and elasticity isothermally. And finally, the 4-mm DSR test method would enable to have the same device for the determination of bitumen rheological properties at high, intermediate and low temperatures. But, the use of 4-mm DSR also sets a number of challenges. For example, obtaining a correct temperature control of such a small (4 mm in diameter) sample is challenging, as well as its preparation and correct placement between these small plates. It is of particular importance to obtain a correct sample radius. Repeatability and reproducibility have been under discussion. Sui et al. (2010) concluded that 4-mm DSR tests have a good repeatability and reliability. They investigated the repeatability by comparing master curves, measured in 4 repeats, including a numerical comparison of these master curves based on the Christensen-Anderson-Marasteanu model. The reliability of 4 mm data was confirmed by comparing data collected on different size plates, and by comparing BBR versus 4 mm plate data. Farrar, Salmans and Planche (2013) as well as Farrat et al. (2016) reported that the repeatability of the 4-mm DSR is currently under investigation and is part of ruggedness and round robin testing and will be published in the near future.

The main objective of this paper is to evaluate the challenges when using 4-mm DSR on a material like bitumen, and to determine the repeatability and reproducibility of 4-mm DSR tests. In addition, procedures how to perform the necessary calibration steps are discussed, as well as sample preparation methods. Finally, some crucial steps, specific for the rheometer type used in this study are included.

## 2. Experiment

Four bitumen types were selected, of different origin and penetration grades, empirical properties are shown in Table 1. The tendency of the bitumen to show physical hardening, taken from previous experiences with these binders is also indicated.

Bitumens designated as B1-B3 are neat while the polymer modified bitumen (PMB) is an SBS modified bitumen, according to FT-IR the modification level is between 3-5%. This was derived from the FT-IR signals at  $699\text{ cm}^{-1}$  and  $966\text{ cm}^{-1}$ , in accordance to Zofka and Błażejowski (2019).

Regarding the preparation of the rheometers and the rheological tests, an overview is presented in Table 2; indicating which tests were performed in each lab. All tests were executed on MCR rheometers produced by “Anton Paar”. Two software versions were used, “Rheoplus (v.3.62)”, and “RheoCompass (v.1.21)”. The specifications of the DSR devices are given in Table 3.

For the temperature calibration, two thermocouples were used, connected to a “Hanna Instruments HI 93532” measuring unit. These thermocouples were calibrated in a temperature range from  $20^{\circ}\text{C}$  to  $-40^{\circ}\text{C}$ , to a maximum deviation of  $0.1^{\circ}\text{C}$ .

In Lab 1 several temperature control systems, from “Anton Paar” for MCR rheometers were evaluated. Initially, an older Peltier hood (PHH-120), bought in 2001, was used. This was disregarded because of ice formation upon cooling. A second option was a CTD180, a Peltier heating – cooling oven, able to cool to  $-20^{\circ}\text{C}$ . This device was disregarded because of very long temperature equilibrations times ( $> 60\text{ min}$ ). Finally, a new Peltier hood, PDT-200 was acquired in Lab 1. It was able to prevent ice formation, still keeping reasonable equilibrium times, as will be shown in the paper.

Table 1. Empirical properties of the selected binders

Bitumen designation	Bitumen grade	Penetration at 25 °C, dmm	Softening point, °C	Physical hardening
B1	50/70	64	47.7	very little
B2	50/70	67	46.8	high
B3	160/220	187	36.9	very little
PMB	25/55-60	37	66.0	unknown

Table 2. Rheometer calibrations and test plan

4-mm DSR tests:	Lab 1	Lab 2
Instrument calibrations (temperature, compliance, inertia, gap setting)	tested	tested
Evaluation of a suitable sample preparation and sample loading procedure (4 binders, frequency sweep at -10 °C, followed by a stress sweep, 5 repeats)	tested	-
Determination of the linear visco-elastic range	tested	tested
Testing of frequency-temperature sweeps (from 0 °C to -24 °C in steps of 6 °C, 10 Hz-0.01 Hz, fixed strain level, 3 repeats)	tested	tested

Table 3. The peculiarities of the DSR devices used in two laboratories

Characteristic	Laboratories	
	LAB 1 (Antwerp, Belgium)	LAB 2 (Vilnius, Lithuania)
DSR type	MCR 102	MCR 302
Manufacturer	Anton Paar	Anton Paar
Software	Rheoplus (v.3.62) RheoCompass (v.1.21)	RheoCompass (v.1.21).
Type of temperature control system	Water-cooled double Peltier system TEK150PA-CF (underplate) + H-PTD200 (hood)	Water-cooled double Peltier system P-PTD200 + H-PTD200 (hood)
Temperature range, °C	+200 to -40	+200 to -40
Temperature accuracy, °C	0.1	0.1
Type of counter-cooling system	VT2: Cooling / heating bath, cooling capacity 260W, to -28°C.	VT2: Cooling / heating bath, cooling capacity 260W, to -28°C.
Fluid of counter-cooling system	50/50 Mixture of water and glycol	anti-freeze up to -35 °C
Device purchase year	The rheometer in 2013, The Peltier hood (H-PTD200) in 2018	2016

### **3. Equipment and sample preparation, and linear viscoelastic (LVE) range**

#### ***3.1. Equipment preparation***

##### ***3.1.1. Temperature calibration***

An accurate temperature control is a main requirement to get reliable results, especially when working with bitumen. To assess thermal gradients, two calibrated thermocouples were used. Before starting the temperature calibration, all temperature corrections in the software were turned off. In a first test, the thermocouples were glued to the centre of the respective plates. Subsequently, a bitumen sample was placed between the plates, as illustrated in Figure 1, with a gap of 2 mm. For this calibration test, a hard bitumen, penetration of 5 dmm, was used, it was easier to handle than softer samples. Preliminary tests on bitumen, indicated that thermal equilibrium is reached after less than 1000 s (16 min). In Figure 2 the changes in  $G^*$  for bitumen B1 are followed over time at -10 °C. At time zero, the temperature was set from 0 °C to -10 °C, and the graph shows that  $G^*$  reaches a stable level after about 1000 s indicating that thermal equilibrium is reached.

During the calibration procedure, the temperature was decreased from 0 °C to -40 °C, in steps of 5°C, and samples were at rest (no oscillations). The thermocouple temperatures were recorded after 20 min. The Anton Paar PTD 200 hood, applies an airflow through the upper Peltier element, to guaranty a more homogenous temperature distribution in and around the test specimen. The temperature calibration was conducted at two air flow rates (200 l<sub>N</sub>/h and 100 l<sub>N</sub>/h).



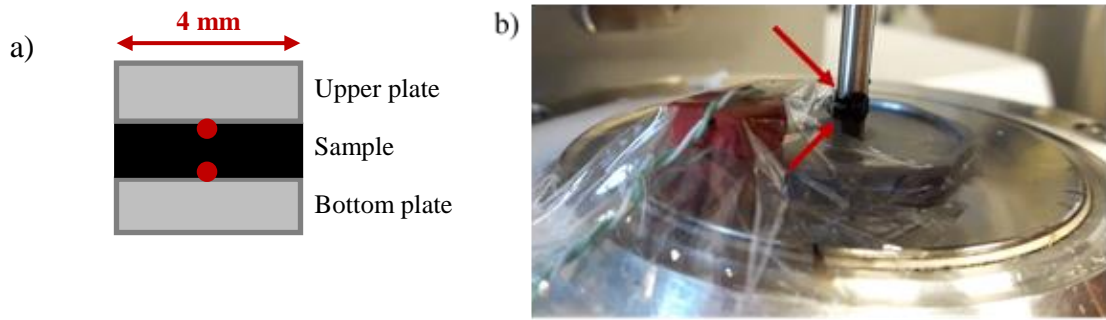


Figure 1. Thermocouples position: a) principal scheme; b) glued thermocouples within the sample

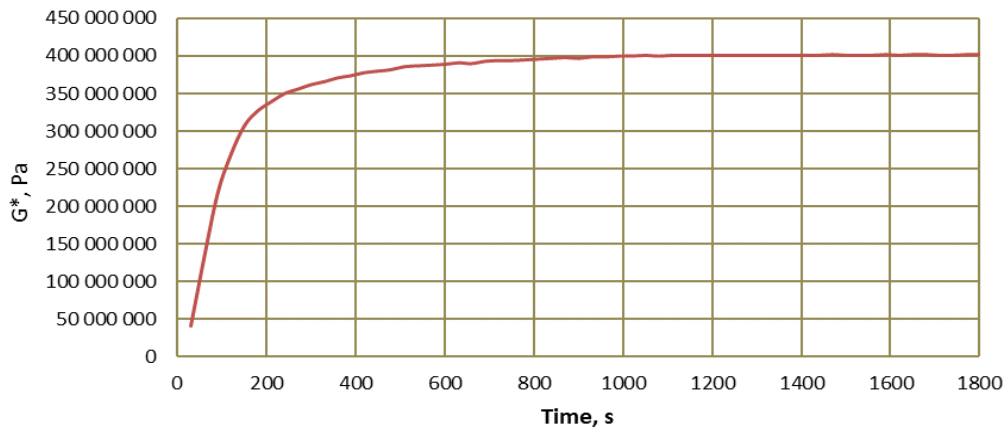


Figure 2. Time to reach thermal equilibrium (bitumen B1) at -10 °C (at time zero, the temperature was set from 0 °C to -10 °C)

The temperature measurements of the external thermocouples are given in Table 3. At the air flow of 200 l<sub>N</sub>/h, the thermal gradients were quite low and almost constant (0.3-0.5 °C) up to -25 °C. At lower temperatures, thermal gradients significantly increased and reached levels of 3.2 °C and 5.3 °C, at -35 °C and -40 °C, respectively. The lower air flow of 100 l<sub>N</sub>/h resulted in higher thermal gradients than the air flow of 200 l<sub>N</sub>/h and these gradients gradually increased when the temperature decreased. Already at -20°C, there was a gradient of about 1°C. Thus, these measurements show the importance of using the same and high enough air flow during calibration and

testing with this geometry and rheometer type. The data also show that below  $-30^{\circ}\text{C}$ , gradients become very large even if using the larger air flow. The data at an air flow of  $200\text{ l}_\text{N}/\text{h}$  from Table 4 were used to determine the coefficients for temperature calibration.

Table 4. Temperatures measured with the external thermocouples at an air flow of  $200\text{ l}_\text{N}/\text{h}$  and  $100\text{ l}_\text{N}/\text{h}$

Set temperature to DSR, $^{\circ}\text{C}$	Upper plate, $^{\circ}\text{C}$	Bottom plate, $^{\circ}\text{C}$	Thermal gradient, $^{\circ}\text{C}$	Average, $^{\circ}\text{C}$	Correction, $^{\circ}\text{C}$
Air flow $200\text{ l}_\text{N}/\text{h}$					
0	-0.5	0.0	0.5	-0.3	0.3
-5	-5.6	-5.1	0.5	-5.4	0.4
-10	-10.5	-10.1	0.4	-10.3	0.3
-15	-15.5	-15.1	0.4	-15.3	0.3
-20	-20.5	-20.2	0.3	-20.4	0.4
-25	-25.3	-25.0	0.3	-25.2	0.1
-30	-28.4	-29.6	-1.2	-29.0	-1.0
-35	-30.4	-33.6	-3.2	-32.0	-3.0
-40	-32.5	-37.8	-5.3	-35.2	-4.9
Air flow $100\text{ l}_\text{N}/\text{h}$					
0	0.4	0.2	-0.2	0.3	-0.3
-5	-4.3	-4.7	-0.4	-4.5	-0.5
-10	-8.9	-9.6	-0.7	-9.3	-0.7
-15	-13.5	-14.4	-0.9	-14.0	-1.0
-20	-18	-19.3	-1.2	-18.7	-1.3
-25	-22.6	-24.1	-1.5	-23.4	-1.6
-30	-27	-29.0	-1.9	-28.0	-2.0
-35	-29.7	-33	-3.2	-31.4	-3.6
-40	-31.5	-37	-5.3	-34.3	-5.7

The temperature calibration procedure was also repeated with 25 mm diameter parallel plates at air flow of  $200\text{ l}_\text{N}/\text{h}$  in the same temperature range in order to check the temperature and thermal gradient. The measured temperatures were similar to those determined with 4 mm diameter plates, the thermal gradient was almost constant ( $0.4\text{--}0.5^{\circ}\text{C}$ ) up to  $-25^{\circ}\text{C}$ . Thus, it seems that the 25 mm calibrated setup from Anton Paar can be used for the temperature calibration of the 4 mm plates, even at these very low temperatures, but there will be no information with regard to thermal gradients.

After setting the temperature correction parameters in the software, as discussed before, a more severe temperature check was conducted. In this case, one of the thermocouples was centred in the sample, and the second thermocouple was measuring the air around the sample (Fig. 3). The air flow was again set to 200 l<sub>N</sub>/h. The results of this check are shown in table 5. These data indicate that the temperature in the sample is acceptable, taking into account the precision of the thermocouples. The air temperature is somewhat lower as compared to the requested set temperature. It indicates that there could still be minor horizontal gradients in the sample, even after performing the temperature calibration.

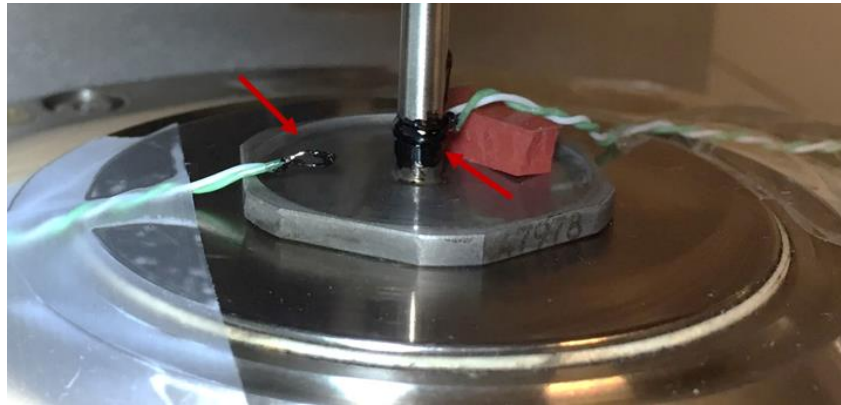


Figure 3. Thermocouples positions

Table 5. Results of the comparison sample versus air temperature around the sample at an air flow of 200 l<sub>N</sub>/h after 20 min

Set temperature to DSR, °C	Air around the sample, °C	Sample, °C
Air flow 200 l <sub>N</sub> /h		
6	6.0	6.5
0	-0.2	0.3
-6	-6.6	-5.7
-12	-12.9	-11.7
-18	-19.0	-17.7
-24	-25.7	-23.8

### *3.1.2. Other calibrations: Inertia, Rheometer radial compliance, and thermal expansion/contraction of the measuring system*

The inertia of the device and measuring system was re-adjusted before the measurements, as recommended by Anton Paar and saved accordingly in the software.

The rheometer radial compliance was determined as described by Farrar et al (2015) and in the Extended Application Report: Measurement of glycerol in the glassy state prepared by Anton Paar. Upper and lower parallel plates were glued together at a small gap (10  $\mu\text{m}$ ) and a torque ramp from 0.5 mN·m to 150 mN·m was conducted. A linear regression between the measured encoder deflection angle and the torque resulted in the rheometer radial compliance of 0.0203 rad/N·m and 0.0242 rad/N in Lab 1 and Lab 2, respectively, which was set in the software. These measurements were repeated at several low temperatures, but temperature did not show a significant difference.

Changes in the gap -setting were determined in temperature range from -30 °C to 0 °C according to procedure described by Farrar et al (2015). The gap deviation from the zero position was 0.007 mm for a change in temperature of 30 °C. This distance was small, compared to the measuring gap of 1.75 mm, so that in this study the thermal expansion/contraction of the measuring system was not corrected. In the experiments described in the following sections, the zero gap was set at an intermediate temperature of -10°C.

### **3.2. Sample preparation**

The main challenges with the 4-mm DSR is to get a sample with the correct diameter and to have a good adhesion between bitumen and both plates. For this purpose, silicone moulds with a cavity of approximately 4 mm in diameter and 2 mm in depth were made. A technique that gave good adhesion results is proposed, consisting of the following steps:

- (1) Bitumen was preheated (according to EN 12594) and poured in the silicon mould, this filled mould was covered directly with a second flat silicon mould (Figure 4). These moulds were allowed to cool to room temperature for about 5 to 10 min;
- (2) The sample was carefully taken out of the mould (Figure 5), for very soft bitumens it is recommended to place the moulds in the fridge, before demoulding.
- (3) The upper plate was preheated outside the rheometer, by keeping it in contact to a standard electrical heating plate (its temperature was about 150-180°C). By pressing the preheated upper plate gently to the sample, a good adhesion between upper plate and sample was obtained, as illustrated in Figure 6 (some preliminary tests may be needed, if the upper plate is too hot, the bitumen may lose its shape).
- (4) The bottom plate was preheated in the DSR to 70 °C, 60°C for the soft bitumen (B3).
- (5) The upper plate with the sample attached to it was placed in the DSR and brought into contact with the preheated bottom plate to the trim position of 1.870 mm. The hood was also closed around the sample for a fixed time of 2 min. This allowed the sample to soften and reach a good adhesion to the bottom plate.
- (6) After 2 min the hood was lifted, and the temperature was decreased to 10 °C. When this temperature was reached in the sample, it was trimmed (Figure 7).
- (7) After trimming, the gap was decreased to its measurement position, 1.750 mm, as recommended also in the WRI proposed draft version of AASTHO standard.

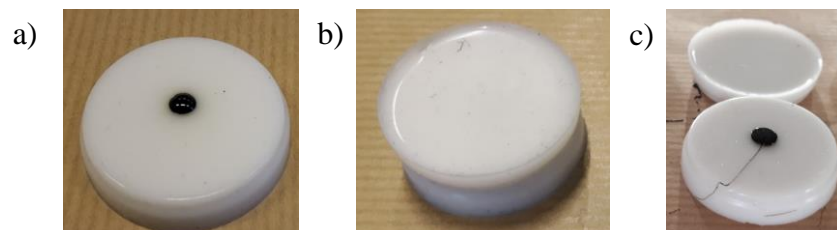


Figure 4. Sample preparation a) mould filled with bitumen; b) sample pressed with another flat silicon mould to get a flat surface; c) prepared sample in mould

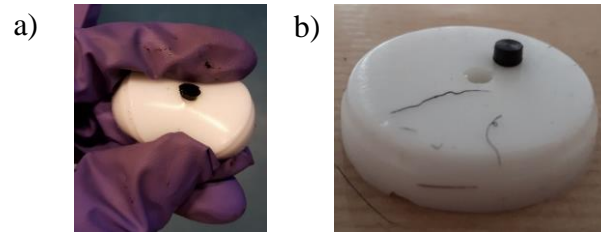


Figure 5. Taking out the sample from the mould a) slightly bending the mould b) the sample out of the mould

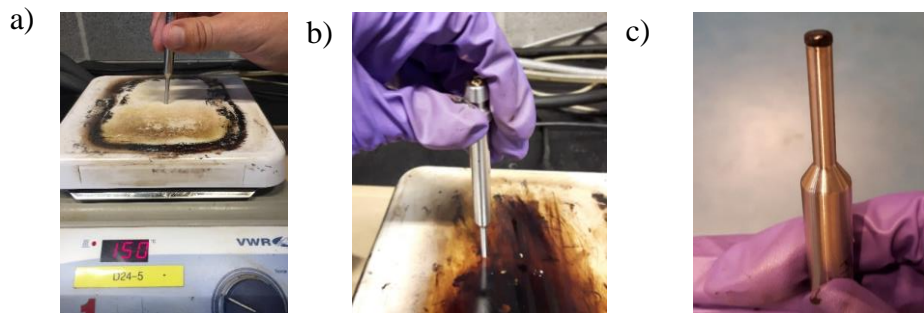


Figure 6. Sample attachment to the upper plate a-b) preheating of the upper plate on the portable electrical heating plate; c) sample attached to the upper plate

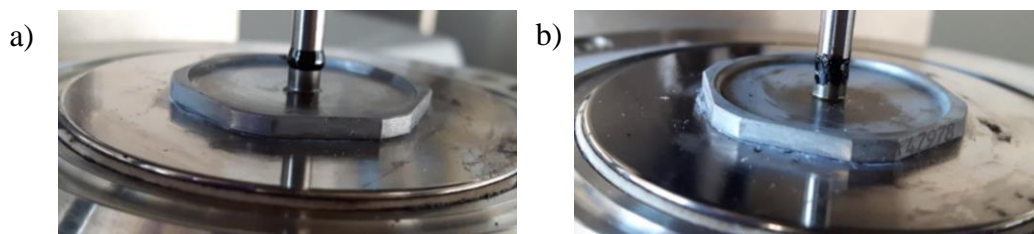


Figure 7. Sample preparation in DSR a) the sample after step 5, when it was kept at a temperature slightly above the softening point for 2 min with the hood in a closed position; b) sample after trimming

It should be noted that the temperature to which the bottom plate is preheated and the temperature at which the sample is trimmed should be selected based on the softening point or on preliminary (trial) tests. The preheating temperature (of the plates and in the rheometer) is critical, it should not be too high, otherwise, the sample may start to flow and loses its shape, but it should be high enough so that a good adhesion

can be reached. The trimming temperature should not be too low, since it is the intention to create a nice outward bulge of the sample and for a very stiff sample this will not happen, and it should not be too high to reduce the risk of deforming the sample by the trimming action.

The adhesion between bitumen and both plates was determined in a strain sweep until failure. If there was good adhesion, both plates were covered with bitumen. In this study, strain sweeps were conducted at  $-10\text{ }^{\circ}\text{C}$  at a frequency of 1 Hz and a normal force of 0.3 N. The strain varied from 0.0001 to 10 (in 31 logarithmic measuring points). A temperature equilibrium period of 20 min was applied, before the testing.

During the strain sweep, the sample “broke”. After the test, both plates were checked. If there was still bitumen visible on both plates (Figure 8), this was taken as an indication that good adhesion had been obtained. In all further tests this method was followed to prepare samples for 4 mm DSR test.



Figure 8. Photograph of both plates after the strain sweep: a) The sample after the test; b) The upper plate; c) the bottom plate

### ***3.3. Determination of the linear viscoelastic (LVE) range***

Typically, DSR tests are conducted within the linear viscoelastic (LVE) range, which is defined as the stress or strain (deformation amplitude) range, in which the complex modulus  $G^*$  and the phase angle are constant. In this study, a deviation of 5% in  $G^*$  from the low strain complex modulus  $G^*$  was used to determine the LVE range.

The LVE range is dependent upon the bitumen stiffness, so also on the test temperature and the loading frequency. The LVE was determined at the highest temperature (0 °C), at an intermediate temperature (-10 °C) and at the lowest temperature (-30 °C). For each specific temperature, the following frequencies, i.e. 0.1 Hz, 1 Hz and 10 Hz, were selected. The strain (deformation amplitude) range varied from 0.00001 to 10. When performing DSR tests at these low temperatures, the bitumen is shrinking during the cooling cycles, and in the period of reaching thermal equilibrium. A constant normal force value between 0.2-0.3 N was set to allow the gap to adapt slightly according to this shrinkage. Thermal equilibrium periods of at least 20 min (typically 30 min) were applied and up to 5 repeats were tested.

In Figures 11-13, strain sweeps at 0 °C, -10 °C and -30 °C are plotted, one repeat is shown. At 0°C, complex modulus  $G^*$  gradually decreased from the constant low strain (plateau) value with increasing strain for all the bitumens. At lower temperatures, i.e. at -10 °C and -30 °C, samples broke very sudden when the strain increased, this happened even within the LVE range. This was clearly seen in the data as a sudden decrease in complex modulus  $G^*$  and was also noticed by a breaking sound, generated by the sample. This is typical for a brittle fracture. At very low strains ( $<8 \cdot 10^{-4}$ ) deviations in  $G^*$  were observed, related to the lower detection limit of the equipment, these values were not used to establish the low strain modulus levels. Table 6 summarizes the information on the determination of the LVE range.



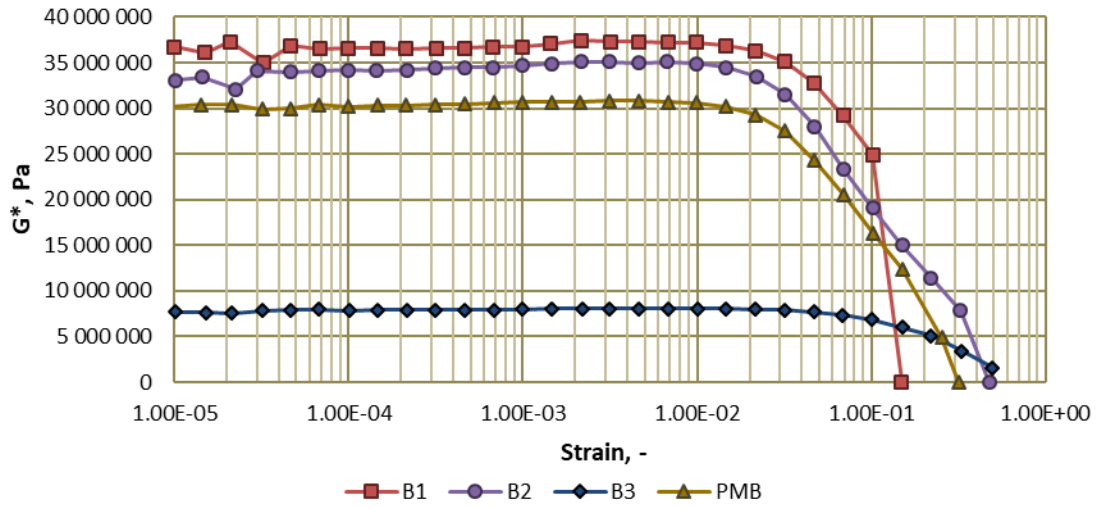


Figure 9. Strain sweeps at 0 °C and 0.1 Hz.

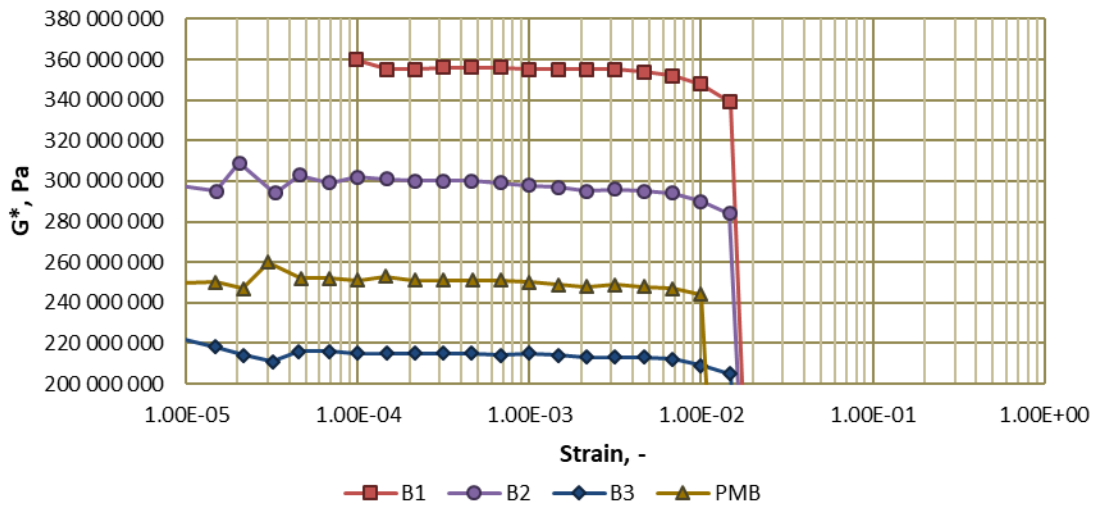


Figure 10. Strain sweeps at -10 °C and 1 Hz

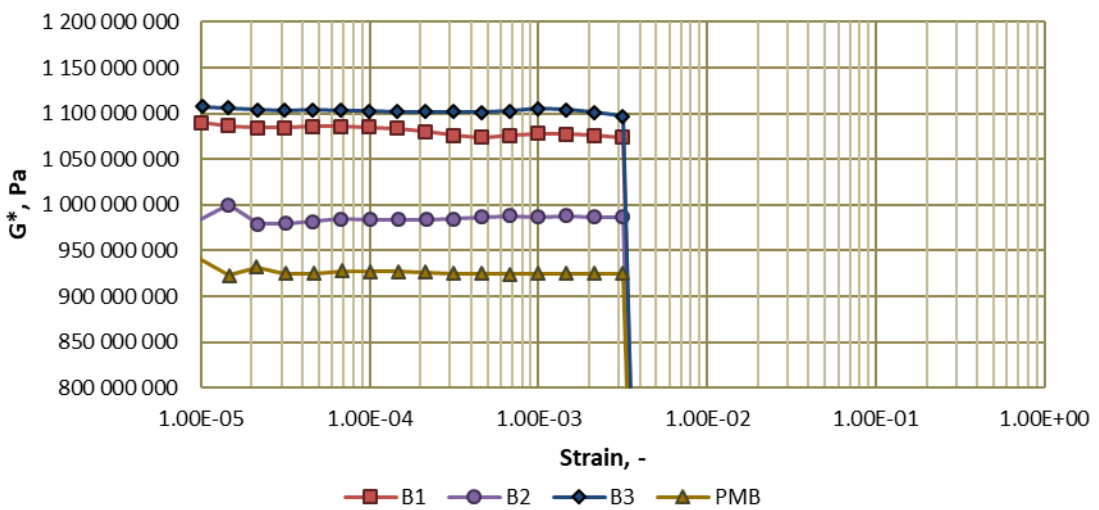


Figure 11. Strain sweeps at -30 °C and 10Hz

Table 6. LVE range at 0 °C, -10 °C and -30 °C

Parameter	Bitumen			
	B1	B2	B3	PMB
0 °C, 0.1Hz				
LVE ( $G^*$ decreased $\geq 5\%$ ), -	3.20E-02	2.18E-02	4.67E-02	2.16E-02
-10 °C, 1 Hz				
Fracture strain, -	8.12E-03	1.22E-02	1.24E-02	1.32E-02
-30 °C, 10 Hz				
Fracture strain, -	3.15E-03	3.16E-03	3.15E-03	3.16E-03

#### 4. Repeatability and reproducibility analysis

Repeatability and reproducibility coefficients of variation for  $G^*$  and phase angle were determined according to standard ISO 5725-2+AC1:2006 “Accuracy (trueness and precision) of measurement methods and results Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method (identical ISO 5725-2:1994 including technical corrigendum ISO 5725:1994/Cor.1:2002)”. In this dataset, data were obtained from 4 bitumen types, B1-B3 and PMB, at 5 temperature points (0, -6, -12, -18, -24) and at 10 frequency points (between 0.01 Hz and 10 Hz) – this are 200 data points. Each binder was tested in 3 replicate test results under repeatability conditions, resulting in 600 observed values, and this was tested in each laboratory.

The overall (average of all replicate tests, including all binder types, frequencies, temperatures and averaged over both labs) repeatability COV was 4.06% for  $G^*$  and 2.50% for the phase angle. The overall reproducibility COV was 5.61% and 3.42% for  $G^*$  and phase angle, respectively. In the EN 14770:2012 standard “Bitumen and bituminous binders – Determination of complex modulus and phase angle – Dynamic Shear Rheometer (DSR)” is stated that the reproducibility of  $G^*$  and phase angle should be in the range below 10% and 5%, respectively, independently from bitumen type, modification and aging. So, the overall repeatability and reproducibility levels, obtained

here fulfil this requirement. Again, with the notation that in this case the reproducibility is in fact a very limited study.

In the next steps the repeatability and reproducibility COVs, respectively denoted as COV-r and COV-R for  $G^*$  and phase angle are calculated as a function of bitumen type, temperature and frequency. The repeatability levels were further separated per laboratory. The results are shown in Figs. 12-14.

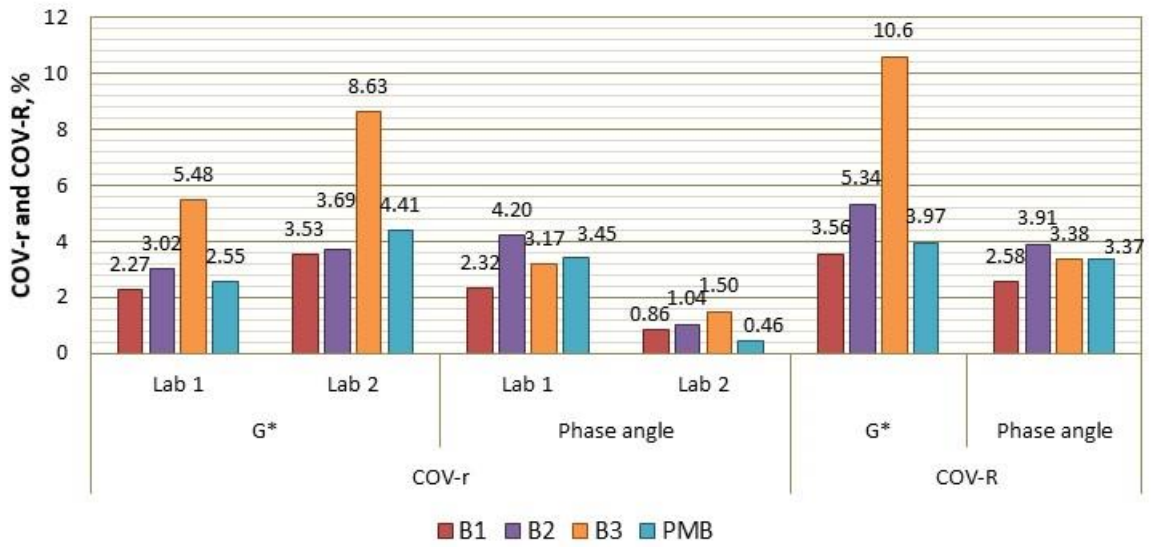


Figure 12. Repeatability and reproducibility COVs for  $G^*$  and phase angle as a function of binder type

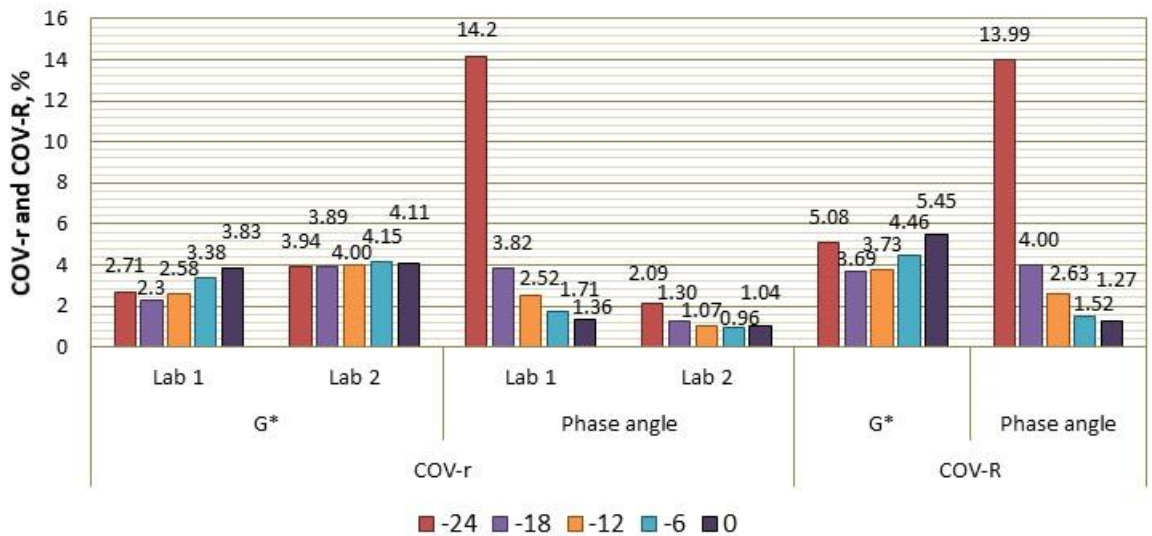


Figure 13. Repeatability and reproducibility COVs for  $G^*$  and phase angle as a function of temperature

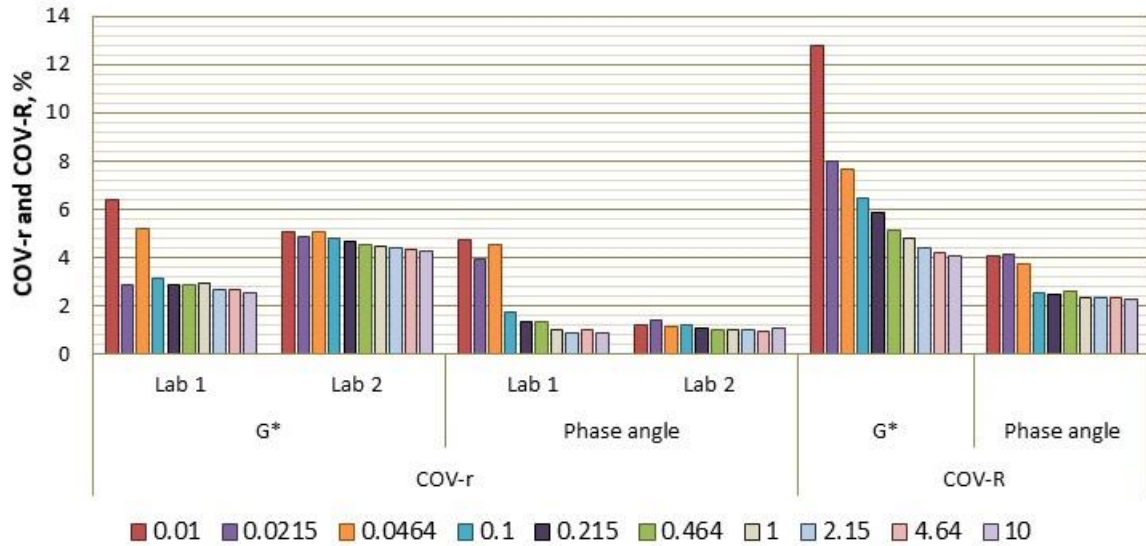


Figure 14. Repeatability and reproducibility COVs for  $G^*$  and phase angle as a function of frequency

Regarding the influence of the binder type, Fig. 12, it is clear that B3, the softest binder has the largest COV-r in both labs with regard to  $G^*$ . So, it seems that this soft binder is the most critical one to measure in a repeatable way. It is even more critical than the PMB. For the phase angle, which has a lower COV-r, this effect is observed only in Lab 2. In Lab 1 B2, the binder with a high physical hardening has the largest COV-r, but the effect is small in both laboratories. One can also note that for B3, COV-R on  $G^*$  is  $> 10\%$ .

Regarding the influence of temperature, Fig. 13, for the modulus  $G^*$ , the COV-r is rather constant with temperature, this is somewhat more clear for Lab 2. For the phase angle, both laboratories show an increase in the COV-r towards lower temperatures. This is more clear for Lab 1 and it has a repeatability issue at the lowest temperature of

-24°C, the COV-r of the phase angle is 14.20%, while this is not observed in Lab 2 (2.09%). Regarding the reproducibility, there is no clear trend for  $G^*$  with temperature, but for the phase angle the reproducibility is clearly deteriorating towards lower temperatures. The large COV-R for the phase angle at -24°C, is most likely related to the repeatability problem of the phase angle in Lab 1 at -24°C.

Regarding the influence of frequency, Fig. 14, for Lab 1, the COV-r of  $G^*$  and phase angle increases towards lower frequencies. This is not observed for Lab 2. The COV-R values of  $G^*$  and to a smaller extend of the phase angle increase towards lower frequencies.

A further graphical analysis of the data was conducted, In Figure 15 the COV-R of the phase angle is plotted as a function of temperature. Each temperature consists of 40 data points, this number is composed of 10 frequencies recorded for 4 binders. From the graph, it is clear that as the temperature decreases the COV-R increases and reproducibility gets worse. The reproducibility is worst at -24°C. One can also note a few very high values for COV-R, as high as 20%. This are data points related to the lowest frequency data, at the lowest temperature in Lab 1.

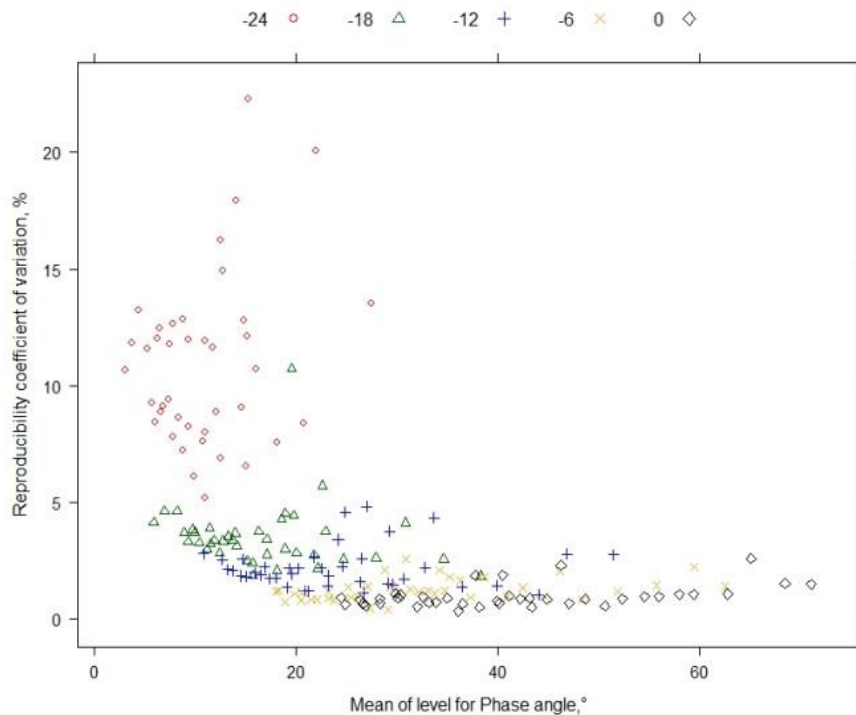


Figure 15. Reproducibility COV-R for the phase angle at different temperatures. (Each temperature consists of 40 data points which refer to one out of 10 frequencies and one out of 4 binders).

As a conclusion, it is obvious from the repeatability and reproducibility parameters that the softest binder (B3) is the most difficult one to measure. The data also show that especially at the lowest temperature of  $-24^{\circ}\text{C}$  the low frequency data (typically from 0.1 Hz and lower) gave a problem in Lab 1. When comparing the data at various temperatures, one can notice that, especially for the phase angle the repeatability and reproducibility levels become worse, at lower temperatures. A possible reason could be the temperature control, which is more difficult at lower temperatures.

The lower repeatability and reproducibility levels at low frequencies could be related to settings in the software, more precisely to the time that the software allows to reach the set value of the strain, and to the number of oscillations used in the calculation. In both laboratories, changing these parameters improved the low frequency (typically from 0.1 Hz and lower) variability.

## 5. Conclusions and recommendations

From the analysis of 4-mm DSR tests presented in this paper, the following conclusions and recommendations can be drawn:

- A very versatile temperature control unit based on Peltier elements allows quick temperature changes, and thermal equilibrium is reached within a time range of 15-20 minutes. Besides, vertical gradients in the bitumen specimen can be reduced to acceptable levels, at temperatures of 0°C to -25°C, by selecting the appropriate air flow. However, with this setup it is not possible to measure at temperatures below -30°C. The comparison of the temperature under the Peltier hood, surrounding the sample, and the temperature in the sample indicated that minor horizontal temperature gradients may be present in the sample, which are currently not corrected by the temperature calibration. Temperature calibration depends strongly on the value of the air flow. The air flow should be at the same level during calibration and testing. In this respect a value of 180 l<sub>N</sub>/h and 200 l<sub>N</sub>/h provided the best results, and ensured the lowest vertical thermal gradients.
- In the sample preparation method, in order to get good adhesion between bitumen and both plates, the plates and the sample should be preheated. The temperature and time of this preheating depends on the bitumen grade. It is recommended to perform preliminary (trial) tests for each sample. The adhesion can be assessed in a stress sweep test until failure.
- At the lowest temperatures, -30°C in this study, all the samples indicated a brittle fracture when tested in strain sweeps, the samples broke while still in the LVE range. At higher temperatures, -10°C and 0°C in this study, this was no longer observed, and the samples moved out of the LVE range, before failing.

- The repeatability analysis showed that in general, the 4-mm DSR test have a high repeatability (< 5%). The test precision depends on the bitumen type, temperature and frequency. The worst repeatability was observed for the softest binder (160/220). Tests at the lowest temperature (-24 °C) and frequency (0.01 Hz) also resulted in a low repeatability. In this case, coefficients of variation higher than 6% for G\* and 5% for phase angle were obtained.
- The trends observed in the limited reproducibility (within two laboratories) with bitumen type, temperature and frequency were similar to the repeatability analysis. In general, the reproducibility levels, expressed as the coefficient of variation, were within the limits specified in EN standard for measurements with 8 and 25 mm plate tests.

From a test design point, the 4-mm DSR tests can be used for bitumen samples within assured repeatability and reproducibility. However, the temperature control, especially the elimination of vertical and horizontal thermal gradients in the sample should be addressed in future researches.

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