Assessment of asphalt binder viscosities with a new approach

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ABSTRACT.

A well acquainted method for measuring the viscosity of bitumen, the rotational viscometer, has undergone major technical improvements in recent years. In several Theses at LTH, the Faculty of Engineering at Lund University, the use of this technique has been developed step by step. Along with the WLF (Williams, Landel, Ferry) equation and a correctly digitized Heukelom's BTDC (Bitumen Test Data Chart) it has become a useful tool for many purposes.
The utility, reliability and accuracy are demonstrated by testing different types of binders compared with other methods. A new procedure makes it possible to replace conventional viscosity methods, even Ring & Ball (Softening Point), with a series of programmed measurements. The precision is the same as for glass capillaries, even better than for Ring & Ball, and they seem to coincide well. Special features of modified bitumen are illustrated graphically and are easily understood, even by an untrained observer.
The temperature dependence of binder viscosities is described with a mathematical relationship based on the WLF equation. Among many uses the viscosity can be predicted for any temperature and temperatures may be calculated for any viscosity, especially the Softening Point. Sufficient space between spindle and container allows testing of asphalt mastics in the same way as bitumen, which should improve predictions of workability and pavement behavior.
In the daily testing of bitumen, especially at road laboratories, costs are cut and the working environment is improved, since the use of solvents is reduced and the cleaning up is easy to do. The measuring process creates no emissions and could even take place on a desk at the office. It could also be operated from another location. The LTH protocol takes viscosity measurements into the digital and the mathematical world in tune with modern road design.

Keywords: viscosity, testing, temperature susceptibility, modified binders, rheology

1. INTRODUCTION

This paper presents a developed usage of the rotational viscometer and examines its pros and cons.
Measuring bitumen viscosity is an essential part of categorizing bitumen and predicting asphalt properties since the beginning of the asphalt technology. The measuring techniques, however, need to be replaced since they do not measure up to modern demands. They are slow, laborious and are burdened with dependence on solvents. From practical and environmental points of view, even when it comes to costs, modernized versions of the rotational viscometer has become an interesting alternative.
It is based on Master Theses presented at the Faculty of Engineering at Lund University (LTH) in Sweden. Results from these Theses will illustrate the utility of this new approach, the LTH protocol for measuring asphalt binder viscosities.
The word *binder* in the title represents both bitumen and bitumen-filler mastic. Mastics display viscous properties similar to those of bitumen. These characteristics may predict asphalt behavior even better than neat bitumen viscosities.
The procedures are described only in basic parts on this limited space. The operators need to be duly instructed in the details to receive the full benefit of this new technology.

2. BACKGROUND AND ELEMENTS OF THE LTH PROTOCOL

2.1 The instrument

The measurement of viscosity using rotating spindles, here referred to as RVB (Rotational Viscometer model B) has been practiced since the 1930s. It is used in many industrial applications all over the world, including the bitumen industry. However, since it has to compete with well-established methods for bitumen the break through as a fully accepted alternative has yet to come.
The usability of the instruments is much appreciated but for many years the weak spot was the temperature. Since precise temperature is crucial to measurements of bitumen, there were doubts whether the technique was sufficiently reliable.
Some years ago the instrument was equipped with, besides computerized control, an electrical heating device of high accuracy at level with oil baths for glass capillaries. It should have eliminated any concerns about that. Nevertheless, the old skepticism is hard to eradicate due to lack of knowledge about the latest improvements.

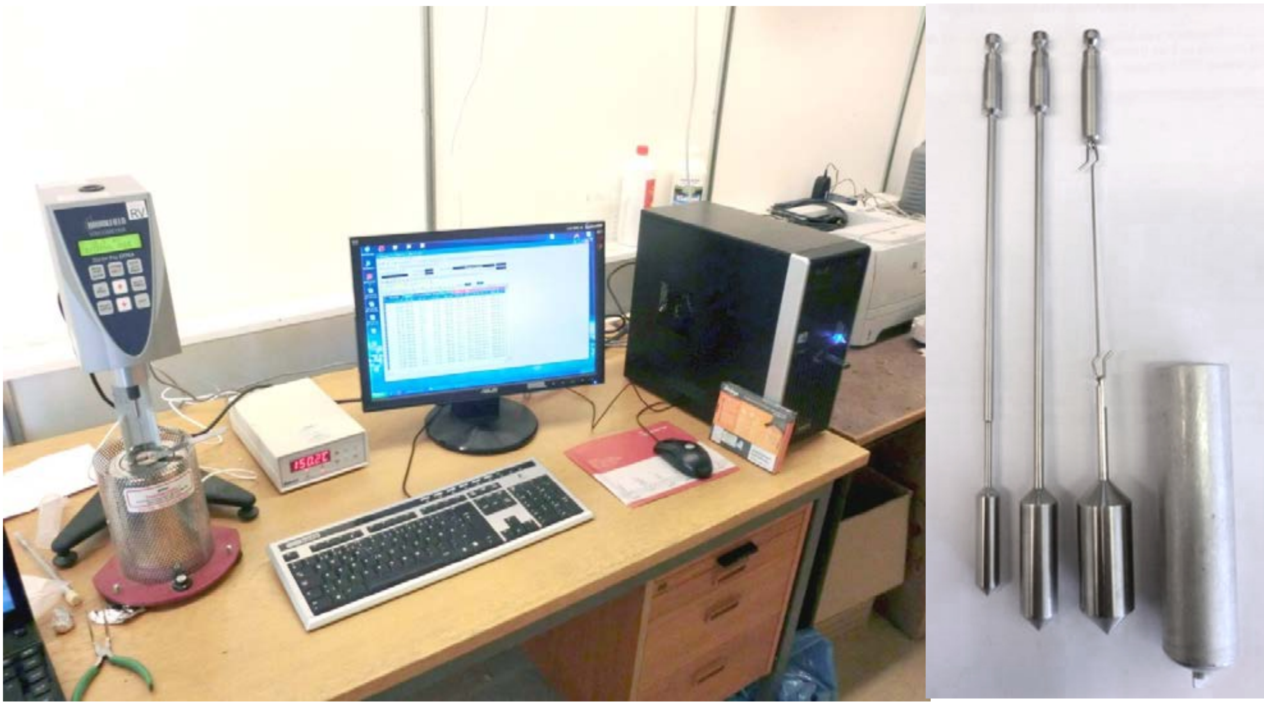

Figure 1: The RVB-equipment: viscometer, electrical heating device and computerized control of running the measurements and recordings. To the right rotating spindles, from left #29, #27, #21 and a cylindrical test container.

2.2. The Heukelom Bitumen Test Data Chart, BTDC

Willem Heukelom presented the final version of his BTDC in 1973 [1]. This outstanding education tool has helped generations of asphalt engineers to a better understanding of the nature of bitumen and the relationship between different properties. It has to some extent lost its significance after the search for more functionally based tests. As long as the methods of Fraass, penetration, Ring and Ball (Softening Point) and capillary viscometers are in use the chart still has a role to play and there are numerous test results to refer to.
To be of practical use the chart needed to be digitized. It has been tried before but not very successfully due to incorrect axis scales. Therefore the first step was to find the original scales to create a correctly digitized version of the BTDC (see Figure 3).
Owing to the accuracy of this chart it became clear that resulting lines connecting measured viscosity values are not straight (see Figure 3). Heukelom assumed the C1constant in the WLF equation (Equation 2 below) to be 8,5 while the correct value is around 7,8 for neat bitumen. Only that case of C1= 8,5 will form a straight line in the viscosity diagram in the BTDC, all others will be slightly curved in either direction. However, the Heukelom BTDC still works well enough as intended and the chart should remain as it is since it is well known all over the world.
Another of Heukeloms assumptions appears to be fairly right, though, the viscosity of the Softening Point being 1,3 kPa·s. This value seems to apply to different binders, including those hard to measure using the Ring & Ball method (see Figure 2, Figure 4 and Table 4-6).

2.3. The WLF equation

In 1955 Williams, Landel and Ferry introduced an equation to describe a relationship between temperature and rheological response for polymeric substances [2]. The original equation is written:

$$\log(a_T) = -C1(T-Ts)/(C2+T-T_s) \quad (1)$$

a_T relaxation time at a given temperature
T_s reference temperature (constant)
T temperature (variable).

Willem Heukelom presented in [1] a version designed for bitumen:

$$\log(\eta/\eta_{ref}) = -C1(T-T_{ref})/(C2+T-T_{ref}) \quad (2)$$

η_{ref} chosen reference viscosity, normally 1,3 kPa·s representing the Softening Point viscosity
η calculated viscosity
T_{ref} reference temperature (constant), normally the Softening Point
T temperature (variable).

C1 and C2 are constants determined by a fitting calculation based on a given set of data. The relationship between viscosity and temperature is usually described with good accuracy this way, much better than using single or double logarithms, which are often used of convenience. The resulting WLF equation of blended grades can be calculated from the WLF constants of the constituents [3]. Even low viscous petroleum oils can be described this way [4].

3. RVB PRECISION AND COMPARISONS WITH CONVENTIONAL METHODS

The rotational viscometer is described in ASTM D4402 and in EN 13302. The expected precision given in each standard is specified in Table 1 along with the corresponding values for Ring & Ball and capillary viscometers.

Table 1: Precision requirements for standardized viscosity methods

Maximum difference between two measurements		
Standard	**Repeatability ***	**Reproducibility ****
ASTM D4402 Rotational Viscometer	3,5 %	12,1 %
EN 13302 Rotational Viscometer	5 %	15 %
EN 12596 Glass Capillary Dynamic Viscometer	6 %	10 %
EN 12595 Glass Capillary Kinematic Viscometer	4 %	6 %
EN 1427 Ring & Ball	1,0 °C (17 % ***)	2,0 °C (34 % ***)

* Same operator, same substance ** Different laboratories, same substance

*** Expressed in % of the viscosity value 1,3 kPa·s

The conservative precision for the RVB in EN 13302 will probably be adjusted in future updates. The precision of the Ring & Ball is low compared to the viscometers, which becomes apparent after converting the allowed temperature variation to the corresponding change of viscosity using the WLF equation.

Table 2 shows different precision values concerning RVB measurements based on results from Theses at LTH [5].

Table 2: Precision in RVB measurements using the LTH protocol

Precision parameter	Determined precision *
Repeatability for RVB readings	3 %
Coefficient of variation for RVB readings	1 %
Average coefficient of variation between readings and the computed WLF equation	1 %

* Rounded figures

The *coefficient of variation* (COV) is the standard deviation expressed in percent of the average value. The *repeatability* is COV multiplied by 2,8, which is used for the difference between two variables with equal normal distribution at 95 % confidence level.
The *determined RVB repeatability* in Table 2 agrees with the corresponding value in the ASTM standard in Table 1. The average COV of the *computed WLF equation* is comparable to that of *RVB readings*, as shown in Table 2.
To compare the RVB versus conventional methods samples were picked out and tested from round robin studies in 2013 and 2014. These surveys are carried out yearly in Scandinavia and Finland, arranged by the Finnish company Neste Oil. They include i.a. 14 viscosity tests and 42 Ring & Ball on each occasion. The results are presented in Table 3.

Table 3: Comparisons between average values of conventional viscosity methods and the RVB to be referred to in the following

Year	Grade	Method	Softening Point °C	Dynamic Viscosity at 60 °C %	Kinematic Viscosity at 135 °C %
2013	B200[1]	Conventional	39,1	100	100
		RVB difference	-1[4]	+11,9	+5,1
	B70[2]	Conventional	47,2	100	100
		RVB difference	-0,1[4]	+12,3	+3,5
2014	B200[1]	Conventional	39,7	100	100
		RVB difference	-1,6[4]	+10,2	-1,7
	B50[3]	Conventional	51,6	100	100
		RVB difference	0,9[4]	+16,5	+5,9
		Average difference	**-0,5**	**+12,7**	**+3,2**
		Accepted difference	± 2[5]	± 10[5]	± 6[5]

Finnish grades: [1] ~ 180/200 [2] ~ 70/100 [3] ~ 40/60

[4] Temperature at 1,3 kPa·s [5] Reproducibility in Table 1.

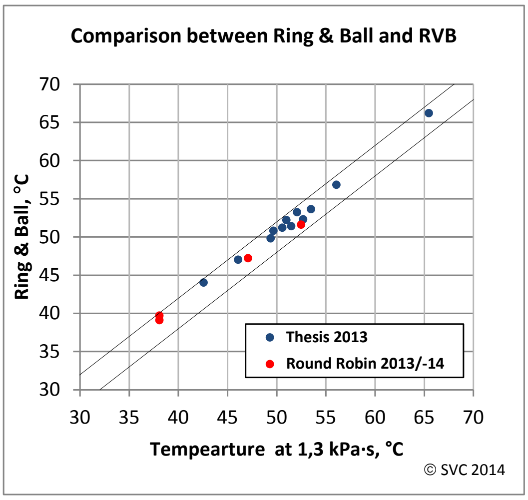

Figure 2: Comparison of Ring & Ball and RVB at 1,3 kPa·s. The red dots come from Table 3.

The *kinematic viscosities* in Table 3 correspond well. The same goes for the *Softening Point*, which is remarkable, since the comparison is based on the assumption that the Ring & Ball viscosity is 1,3 kPa·s. The difference between *dynamic viscosities* at 60 °C, however, exceeds well the accepted limit. There is no immediate explanation for this. It has to be repeated before being recognized. The major difference from EN 12595 for Kinematic Viscosities is the use of vacuum, which helps viscous bitumen to pass through the glass capillary. There is a chance that the method in EN 12596 is affected by a negative bias (see red figure in Table 3.), which should be investigated.
The Softening Point column in Table 3 is shown graphically in Figure 2 along with values from a Master Thesis in 2013. The limits in Figure 2 are drawn ± 2 °C from a diagonal, representing the reproducibility limits of the Ring & Ball test. All observations are within the limits, which leads to the conclusion that RVB measurements at 1,3 kPa·s could replace the Ring & Ball test.
Consequently, when the RVB is used no changes are necessary concerning limits or target values for Softening Points and Kinematic Viscosities. Values referring to EN 12596 about Dynamic Viscosities may need revisions if replaced with RVB measurements.

4. THE LTH MEASUREMENT PROCEDURE

A common way of running the rotational viscometer is by pushing buttons on the instrument and then reading the results from the instrument display. It is not very practical in the long run and it lacks traceability. Instead the instrument should be operated and monitored automatically by a computer, testing a range of temperatures in a single run. It streamlines execution compared to the measuring of one temperature at a time as described in American and European standards. Another improvement compared to the existing use is the possibility to calculate the temperature for any given viscosity.
The software has several options, such as statistical analyses and assessment of Newtonian behavior, but no one tailor-made for bitumen, more for general use. The most useful feature is the programming of a test. It is not very user friendly but it works. The operator is freed from continuously reading results. Since a single reading can take up to an hour, it saves a lot of time. However, the real time saver is when a series of measurements are carried out on the same sample. After completion of the measurements the results are automatically saved in the computer, ready to be used or exported.

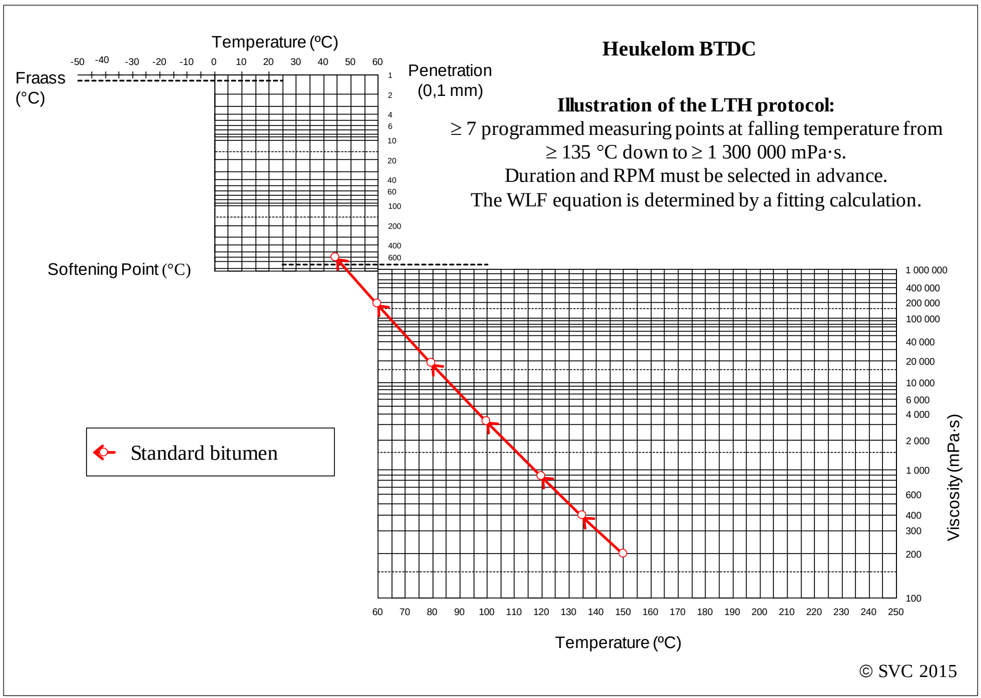

Figure 3: A correctly digitized Heukelom BTDC illustrating the LTH protocol.

Instead of repeating the measurements at the same temperature to ensure accuracy the sample is tested by single readings at seven or more pre-set temperatures from 135 °C or higher and down to and including the expected Softening Point as described in Figure 3. The relationship between temperature and viscosity is found through fitting calculation using the WLF-equation (Equation (2)). Then the temperature for any given viscosity can be computed, especially the Softening Point, which according to Willem Heukelom occurs at the viscosity of 1,3 kPa·s [1]. Any outlier is easily detected, visually or by calculation.
The accuracy of a reading is assessed by comparison with the calculated WLF value. Values within repeatability range (see Table 2; 3 % of current viscosity) are accepted. A single outlier may be replaced by the WLF value followed by a

note. If any value is closer to a regulated limit than twice the coefficient of variation (see Table 2; 2 % of current viscosity), the entire test series is repeated with a new sample. The mean values then form the accepted measurement series.
This description lacks the practical steps as sample preparation, programming, calculations etc. These are found in a Master Thesis at Lund University [5].

5. MEASUREMENT EXAMPLES

Three different sorts of bitumen were studied in a Master Thesis at LTH during 2014 [5]: one standard grade penetration 70/100, the same grade with 3 % Sasobit wax and a Polymer Modified Bitumen, PMB, of a regular type. All three were also aged with RTFOT (Rolling Thin Film Oven Test) and RTFOT+PAV (Pressure Aging Vessel). These results are presented in Figure 4 along with neat bitumen penetration 70/100 which is compared to bitumen-filler mastics containing granite filler. The last example comes from a Master Thesis in progress.

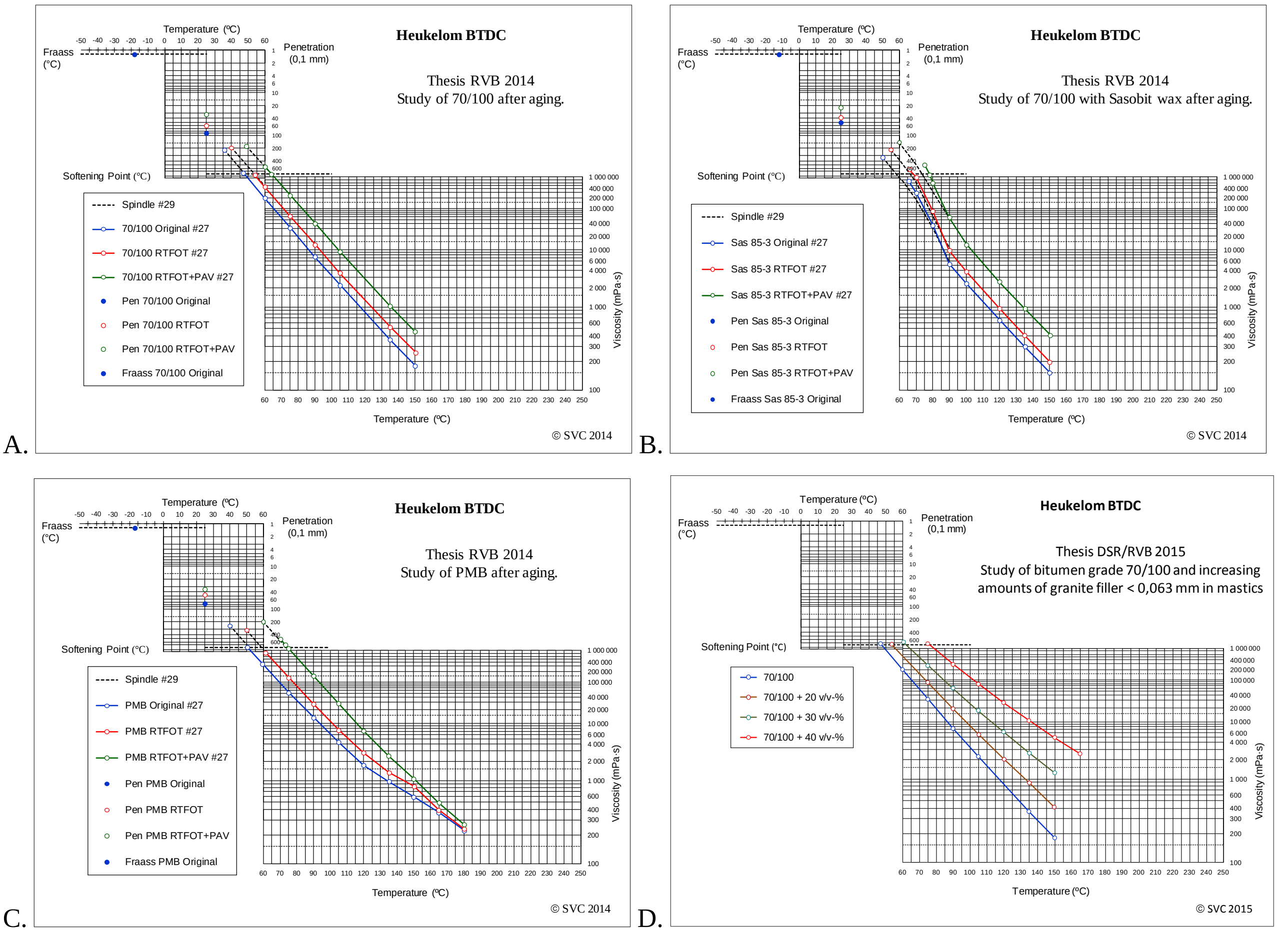

Figure 4: Measured values with the RVB for three different binders in original condition, aged with RTFOT and finally with RTFOT+PAV. D. compares neat bitumen with increasing amounts of filler in bitumen-filler mastics.

5.1 General comments

The purpose of these measurements was to illustrate the nature of various binders and the effects of artificial aging. The latter is often referred to in regulations as change of the Ring & Ball value or in the case of RVB the Softening Point. It is a known fact that the Ring & Ball test is not reliable when there are impurities or for modifications with Sasobit wax or PMB. In those cases the test does not produce results of rheological significance, since the test was meant for neat bitumen only (Heukelom [1]). The RVB, however, delivers credible results for the Softening Point, although these binders are very different in character.
The WLF equation is best suited for neat bitumen without impurities, especially from substances with a melting point, i.a. paraffin. Discontinuous relationships have to be divided into continuous parts around positions of interest, e.g. the Softening Point, before creating the WLF equation for that particular section.
Among these some 90 values only one was a suspected outlier: PMB/RTFOT/150 °C. PMBs are hard to measure in the liquid state.

The dots represent single readings and no one has been deleted or replaced. A few series were repeated regarding the Sasobit wax mixtures. Viscosities were difficult to predict, which the RVB program requires (see chapter 7.), and therefore some pretests were needed.

5. 2 Remarks concerning materials

Figure 4 A: Penetration 70/100

The RVB measurements match known values for the Softening Point and viscosities at 60 °C and 135 °C. The RTFOT aims to simulate the aging (hardening) effect from asphalt production. A maximum change of 6 °C for Softening Point is a normal requirement in a real case and that seems to coincide well with this artificial example. RTFOT combined with PAV intends to describe the final stage of bitumen in aged pavements. Normal values for extracted bitumen from reclaimed asphalt in Sweden are 63-66 °C, similar to the artificial aging with RTFOT+PAV. The matching results of two spindles, #27 and #29 causing different shear rates, indicate that neat bitumen, tested with the RVB, displays Newtonian behavior even at the Softening Point (1,3 kPa·s).

Table 4 gives the calculated constants for these neat bitumen varieties to be used in the WLF equation described in Equation (2).

Table 4: Constants in the WLF equation for bitumen 70/100, original and aged

Bitumen grade	Spindle #27		T_{ref} (Softening Point), °C	
	C1	C2	#27	#29
70/100 Original	7,83	105,2	47,6	48,0
70/100 RTFOT	7,79	106,3	53,2	53,6
70/100 RTFOT+PAV	8,23	119,0	63,6	64,0

Figure 4 B: Penetration 70/100 and 3 % Sasobit wax

The melting point of Sasobit wax in its pure form is about 115 °C. When it is blended with bitumen and during cooling Sasobit crystalizes not until at 90 °C, illustrated by a steep rise of the viscosity line. This is sometimes referred to as effects of “thermal history”. The LTH procedure is performed during temperature reduction to provide the samples the same thermal history and to copy the drop of temperature during asphalt production after mixing.
The elevated viscosity from crystallization is sensitive to rotation speed (share rate), which is displayed by higher viscosity for spindle #27, rotating slower than spindle #29. This characterizes non-Newtonian behavior. It indicates that the stiffening effect from Sasobit wax is likely to increase at lower loading frequencies. Plastic deformations are often caused by slow traffic, therefore this boosted effect could be beneficial in those cases. The stiffening effect of Sasobit wax seems to survive the artificial aging. Whether this is beneficial or damaging remains to be explored.

Table 5 presents the calculated Softening Points for various modes of the wax-modified bitumen.

Table 5: Softening Point for wax modified bitumen, original and aged

Bitumen grade	Softening Point, °C	
	#27	#29
Sas 85-3 Original	60,0	56,5
Sas 85-3 RTFOT	68,2	66,3
Sas 85-3 RTFOT+PAV	77,7	73,3

Figure 4 C: Polymer Modified Bitumen

When PMB is cooled from a liquid state at around 180 °C long, rubbery threads appear. It seems unlikely to expect any liquid behavior after seeing that but it works with the RVB. The sample was first heated to 195 °C to secure a liquid state at the first reading at 180 °C. The viscosity line flattens out for the original PMB and for the RTFOT version above 120 °C. This is well known at the mixing plants, since the temperature has to be increased considerably to around 175 °C for good mixing and workability from otherwise sufficient temperatures in Sweden at around 150 °C. This excessive heating may cause adverse hardening of the bitumen part, which actually composes 97 % of the PMB. The slope and shape of the viscosity line after RTFOT+PAV resemble that of neat bitumen, although very viscous. These hardening factors could explain the brittle and sudden disintegration that sometimes affect PMB pavements, even fairly new ones.

Table 6 shows the calculated Softening Points for the PMB versions.

Table 6: Softening Point for PMB, original and aged

Bitumen grade	Softening Point, °C	
	#27	**#29**
PMB Original	51,2	51,1
PMB RTFOT	58,4	59,0
PMB RTFOT+PAV	74,5	73,8

PMB has been claimed to display non-Newtonian behavior, but the Softening Point values in Table 6 indicate otherwise. Different shear stresses and shear rates with spindles #27 and #29 results in approximately the same Softening Points, which characterizes Newtonian behavior, unlike the figures in Table 5 for Sasobit wax.

Figure 4 D: Penetration 70/100 and bitumen-filler mastics

The chart compares neat bitumen with mastics containing increasing amounts of granite filler <0,063mm. The space between spindle and container, 3,6 mm for #27, would allow for larger grains but this size is chosen to avoid sedimentation during the test. The mastic display liquid properties resembling very viscous bitumen and show Newtonian behavior between 70 °C and 150 °C. These results come from a Master Thesis at Lund University in 2015.The viscosities of mastics were also the topic of a PhD Thesis presented at KTH, Sweden, in 2014 [6].

5.3 Final comments

This brief demonstration shows the benefits of using one accurate measuring technique for viscosities instead of several of different nature. It generates precise and comprehensive pictures to be assessed and evaluated both visually and by calculations. This is the main technical purpose of the LTH protocol for RVB measurements.

6. WORKING ENVIRONMENT AND COSTS

At laboratories specialized in bitumen, for example linked to bitumen producers, the personnel is familiar with all kinds of tests, sample preparations and handling with solvents. Residues are easily disposed of being close to a refinery. The situation is quite different at the contractor's or the asphalt producer's road laboratories. Besides analyzing asphalt mixes and pavements they also check the quality of several components besides bitumen. Measuring bitumen viscosity is therefore infrequent to most laboratory workers. The condition of the equipment and level of knowledge cannot always be on top. The capillary tests are time consuming and the equipment needs to be used frequently for accurate results. Sample preparation and cleaning up afterwards with solvents are not only laborious and environmentally and healthwise problematic. Glass capillaries are also fragile and expensive. Not surprisingly, this test is not particularly popular among road laboratory personnel, which is a poor prerequisite for important measurements.
The RVB represents the opposite of this and is much appreciated by the users. Sample preparation is simple and containers and spindles are easily cleaned with a disposable solvent. For those who seek a totally solvent free working environment disposable containers and spindles are available for reasonable costs.
The instrument needs no particular space to take care of emissions or noise. The procedure is so clean it could even be placed at the office (see Figure 1). The instrument can be operated by remote control, even over the internet. Different tasks might be shared, preparing the measurement on site and then running it from another location. Filled containers could be sent to the laboratory. This technique opens up for many modes of operation.
The RVB is less expensive than the capillary package and the Ring & Ball equipment. Moreover the LTH measuring model requires less hours per measured value. Without comparing sums the RVB is obviously cheaper, if used in accordance with the LTH practice.

7. NEED OF IMPROVEMENT

The most urgent problem concerns the software. Besides the not so user friendly programming routines, the rotation speed and duration must be decided in advance for every temperature. After several tests of standard bitumen it is not so hard to figure out but for unknown binders it is harder, especially for modified bitumen. When the actual viscosity falls outside the expected measuring range the reading will be stopped. If run by a program the test will move on to the next temperature, while leaving a gap in the results. The test can be restarted with adjusted settings but time is lost.
The need for predicting the viscosity and consequently the choice of rotation speed comes from the limited strength of the torque spring. It is rather weak to assure accuracy around expected results and to simplify the instrument design. A Dynamic Shear Rheometer, DSR, which also measures viscosities with rotating spindles, is more robust having a much stronger torque spring, which widens the measuring range. A DSR, primarily intended for advanced viscoelastic measurements, is however fairly expensive costing up to seven times more than a rotational viscometer.

This drawback concerning the RVB is eliminated if the software is added an interactive function, which automatically selects a proper rotation speed and reads the final value, once constant according to any requirement (equilibrium).

8. CONCLUSIONS

The rotational viscometer, although well known for many years, has yet to be recognized among bitumen specialists as a fully capable instrument for tests requiring high accuracy. Major steps towards this status were taken with the electrical heater and computer controlled operations. Prior to these improvements doubts were understandable. Now the instrument is not only equal to conventional methods but has surpassed them.
The RVB technique operated according to the LTH procedures:

- Replaces three existing viscosity methods: Kinematic Viscosity, Dynamic Viscosity and Ring & Ball
- Provides a precise and comprehensive picture of viscosities, visually in the BTDC and mathematically with the WLF equation
- Reduces costs
- Improves the working environment and makes the job more attractive.

The use of the WLF equation brings this RVB measuring technique at level with a Dynamic Shear Rheometer. The WLF equation is in fact the master curve of viscosities. Combined studies using both a DSR and the RVB seem only natural after these improvements.
The measurements were performed by students with only short and basic training in laboratory practices. Bearing that in mind, the results are not only a credit to them but also demonstrate the reliability and the accuracy of the instrument and the procedures composing the LTH protocol.

ACKNOWLEDGEMENTS

This presentation is based on Master Theses carried out at LTH (*Lunds Tekniska Högskola*), the Faculty of Engineering at Lund University, Sweden, from 2011 to 2014. The student's commitment was crucial to the final result. The company of Nynas AB prepared and supplied samples.
Finally, tributes are addressed to Williams, Landel and Ferry and Willem Heukelom. Their ground breaking work form the basis of the LTH protocol for viscosity measurements of asphalt binders.

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