Operator proficiency in standard methods and procedures becomes questionable. It also raises questions as to the use of a single set of specifications for both a single-laboratory and multi-laboratory testing scenario.

The above findings are based on a single project that was statistically analysed. In order to reach conclusive findings and to substantiate conclusions and recommendations, this study has to be expanded to include different projects, laboratories, personnel, production plants and bituminous materials.

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The views expressed in this paper are these of the author and do not necessarily reflect the standpoint of the University of Stellenbosch.

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APPLICATION OF THE BROOKFIELD VISCOMETER TEST METHOD IN SOUTH AFRICAN ROAD GRADE BITUMEN SPECIFICATIONS

MP Zacharias and SJ Emery

Bitumen Engineer
Shell South Africa (Pty) Ltd
Cape Town, South Africa

Professor of Asphalt Pavement Engineering
Department of Civil Engineering, University of Stellenbosch
Stellenbosch, South Africa

Abstract

The basis for the South African specification for normal road grade bitumens has recently changed from penetration to viscosity. The viscosity is determined using the Brookfield viscometer at 60°C and 135°C according to the ASTM test method D4402. This paper discusses the method, its repeatability and reproducibility and setting limits for the specification. The results of a major study covering a number of South African laboratories to investigate the reproducibility of the test are included. To enable the Brookfield results to be correlated with results from other test methods as are used in other countries, a comparison is made between the Brookfield and other viscosity test methods used in the 60°C and 135°C temperature range.

1 INTRODUCTION

An amended specification for road grade bitumens in South Africa was implemented in January 1994. It is a viscosity based specification, based on the ASTM D4402 test method using the Brookfield viscometer and Thermosel apparatus. The previous specification SABS 307-1972 (SABS,1), had been penetration based. A number of items in the new specification are given as 'report only' to allow for experience to be gained with the new test methods and likely test values for South African bitumens. Once sufficient confidence has been gained in the change from the old penetration grade specification, South African Bureau of Standards (SABS) will revise SABS-307 to reflect the new road grade bitumens. This is expected to be finalised in 1995.

This change to a viscosity specification enables the service performance of road grade bitumens in South Africa to be better modelled. It is based on the viscosity at 60°C, which is typical of the road surface temperature in South Africa in summer. South Africa is not alone in using a viscosity based specification; this is being done in at least 11 countries world-wide (Shell, 2). Accurate methods of measuring viscosity in-line are being developed which

means that products can be manufactured by blending directly into the tanker as demand requires with product quality being known immediately.

The Brookfield test method was adopted because it enables the testing of a wide range of bitumens over a wide range of temperatures, more so than most other viscosity measurement apparatus. The equipment was already proven in the American bitumen industry for waterproofing and roofing grade bitumens, and there was a Standard Method published (ASTM D4402). The cost of the equipment is low, relative to other equipment such as the sliding plate and Haake viscometers.

This paper discusses aspects of the viscosity of bitumen and the Brookfield test, its repeatability and reproducibility, and its incorporation into the South African specification. Comparisons are also made with some other viscosity based test methods to allow these methods to be used to approximate Brookfield measured viscosities.

2. VISCOSITY

2.1 Measurement of viscosity

Viscosity is the measure of the resistance to flow of a liquid. The ratio between the applied shear stress and the rate of shear is called the *coefficient of viscosity*, *dynamic viscosity* more often simply *viscosity*. The SI unit of dynamic viscosity is the Pascal-second (Pa.s) which is 1 N.sec/m^2 and the cgs unit is the gm/cm.s which is the poise (or more conveniently, centipoise cP), such that at a given temperature:

1 Pa.s = 1000 mPa.s = 10 poise = 1000 cP

Kinematic viscosity (υ) is the ratio of the dynamic viscosity (η) to the density (δ) of a liquid such that:

$\nu = (\eta \times 1000)/D$

$$\upsilon = \eta/\delta \quad \text{(Eq.1)}$$

Clause 170 175 Pa.s

where:

υ = ~~Dynamic~~ Kine viscosity in mm²/s

η = ~~Kinematic~~ Dynamic viscosity in Pa.s

δ = Density in kg/l at the temperature under consideration

1.73×10^5 mm²/s

The SI unit of kinematic viscosity is mm^2/sec, and the cgs unit is cm^2/s which is a Stoke or more conveniently, centistoke cSt, such that:

1 mm^2/s = 0,001 cm^2/s = 1 centistoke

Since densities of South African road grade bitumens vary between 0,970 and 1,040 kg/l at 60°C, a dynamic viscosity of 1 Pa.s would equate to a kinematic viscosity of between 962 and 1031 mm^2/s (at 60°C).

There are also empirical viscosity measures, such as the standard tar viscometer which measures the time of flow for a measured amount of bitumen through a standard orifice. These measures can be converted to fundamental units using conversion factors.

2.2 Newtonian and non-Newtonian fluids

The flow behaviour of bitumen can be divided into Newtonian and non-Newtonian. When it is behaving as a Newtonian fluid, the rate of shear is proportional to the shearing stress, and so the viscosity is independent of the shear rate.

For non-Newtonian conditions, the measured viscosity is not a unique material property, and depends on the bitumen, the test apparatus and conditions as well as the thermal history of the sample. Thus comparisons between non-Newtonian viscosity values can only be made for similar viscometers under similar conditions of shearing stress and shear and thermal history.

At about 90°C and above, bitumens are held to behave as true Newtonian fluids. At lower temperatures, they exhibit non-Newtonian behaviour to a greater or lesser extent, in which the rate of shear is not proportional to the shearing stress. The error of assuming Newtonian behaviour at temperatures below about 60°C, becomes greater than 10% of the theoretical *absolute* or *zero shear rate* viscosity (de Bats and van Gooswilligen, 3). However, the temperature which marks the transition between Newtonian and non-Newtonian behaviour in bitumen is not distinctive, and can vary between bitumens with different crude origins and refinery processing paths. The implications for the Brookfield viscometer test method are that the conditions of the 60°C test must be standardised to a much greater extent than previously thought necessary.

2.3 Viscosity test methods

There are a number of methods for measuring dynamic and the kinematic viscosity of bitumens.

Cup viscometers such as the Saybolt Furol, Redwood, Engler or Standard Tar viscometers (STV) are the simplest means of establishing viscosity. The time for the liquid to flow through a standard orifice is multiplied by a constant (particular to the instrument) to get the kinematic viscosity of the sample. Cup viscometers are capable of measuring viscosities in a range 10 to 100 000 mm^2/s (Heukelom and Wijga, 4).

The flow conditions are much better defined if the open orifice of the cup viscometer is replaced with a long narrow tube or capillary. Capillary viscometers, are used to measure kinematic viscosity, with the bitumen

flowing under its own weight, or dynamic viscosity, by 'pulling' bitumen through the viscometer under vacuum.

A commonly used kinematic capillary viscometer method is ASTM D2170 (5), which provides for several viscometer tubes including the Cannon-Fenske, Zeitfuchs Cross-Arm, Lantz-Zeitfuchs, and BS/IP/RF U-tube. These typically operate at 60°C and 135°C and measure viscosities in the range from 6 to 100 000 mm^2/s.

The dynamic viscosity method ASTM D2171 is the most commonly used throughout the world (Shell, 2). It provides for several vacuum viscometers: Cannon-Manning, Asphalt Institute, and modified Kopper. These operate at 60°C and measure viscosities in the range from 0,0036 to over 20 000 Pa.s (ASTM, 6).

The sliding plate viscometer is used to shear a thin (5 to 50 microns) bitumen film between two parallel flat plates at a known rate. This method allows for very high viscosities to be measured (i.e. at low temperatures). One such instrument is the Shell Sliding Plate Microviscometer which is capable of measuring viscosities in a range 10^5 to 10^9 Pa.s (Busby and Rader, 7).

Rotational viscometers such as concentric cylinder type, employ a theory similar to the sliding plates with one cylinder rotating coaxially inside a second (static) cylinder which contains the sample. To approximate a uniform stress distribution the space between the inner and outer cylinders (where the sample is sheared) must be as small as possible compared to the radius of the smaller cylinder. Another rotational viscometer is the cone and plate type. Uniform stress can be achieved when a sample is placed between the cone and a flat plate, the former just touching the plate. Companies such as Brookfield and Haake make viscometers that employ rotational principles.

In South Africa, test method ASTM D2170 is used to measure the kinematic viscosity of cutback bitumens in the reverse flow U-tube. Test method ASTM D244 incorporating the Saybolt Furol viscometer is used for emulsions, and the Standardisation of Tar Products Test Committee's method STPC RT3 measures the equiviscous temperature using the STV cup-type viscometer.

3. BROOKFIELD VISCOSITY TEST METHOD ASTM D4402.

3.1 Background

According to the test method ASTM D4402-91 (ASTM, 8), a Brookfield Synchro-Lectric rotational viscometer with Thermosel system is used to measure the viscosity of bitumen at elevated temperatures. The torque on a spindle rotating in a thermostatically controlled sample holder containing a small sample of bitumen is used to measure the relative resistance to rotation. A factor is applied (manually or electronically) to the torque dial reading to yield the viscosity of the bitumen in millipascal seconds (mPa.s).

For a given viscosity, the viscous drag is related to the spindle's speed of rotation and geometry (spindle shape and diameter, thus circumferential speed). Various spindle shapes exist: disc, cylindrical, coaxial, cone/plate, and t-bar. The larger the spindle or the higher the speed, the greater the drag will be.

ASTM D4402 requires that the *spindles for the Brookfield Thermosel Viscometer* are to be used in the test. Brookfield lists these as their SC-4 range of spindles (Brookfield, 9).

Another spindle that may have application to research on bitumen, although it is not specified by the ASTM D4402, is the T-bar spindle (Marsh, 10). The T-bar permits consistency testing of non flowing materials (such as pastes, gels or creams) that are rheoplexic or thixotropic, in that their relative viscosity/consistency changes with time (Brookfield, 11). To overcome this problem the T-Bar spindles are slowly lowered or raised so that the rotating shearing element will describe a helical path through the test sample and will always be cutting fresh material.

The ASTM D4402 test method provides that the selected spindle (SC-4 type) is equilibrated in the clean Thermosel chamber for 90 minutes. An 8-10 ml sample of heated bitumen is added to the chamber (with the spindle temporarily removed). The spindle is inserted into the bitumen, and allowed to equilibrate for a further 15 minutes. The viscometer is started, and provided the torque is between 2 and 98% of full scale (but refer to the manufacturers recommendation above), three readings are taken (60 seconds apart) and averaged. The viscosity in mPa.s is found by multiplying the reading by the factor for that combination of gearbox speed and spindle (dial type instruments) or read directly off the digital display (digital instruments).

3.2. Repeatability and reproducibility

The repeatability (r) and reproducibility (R) of a method is estimated from the analysis of variance of results of subsequent tests, carried out on the same apparatus by the same operator (repeatability), or tests performed by different laboratories (reproducibility). It is noted that the latter is usually greater than the former. Values for r and R should be included for each published test method. These parameters can be used in setting specified limits and this was done for SABS 307 and is discussed in section 4.1.

ASTM D4402 specifies that at the 95% confidence level, duplicate values by the same operator shall not be considered suspect unless they differ by more than 3,5% (repeatability). The values reported by each of two laboratories representing the arithmetic average of duplicate determinations, shall not be considered suspect unless they differ by more than 14,5% (reproducibility).

4. VISCOSITY LIMITS AND RECOMMENDED EQUIPMENT FOR SABS 307.

4.1 Viscosity limits

In order to change to the Brookfield method as the basis of the new SABS 307 specification, viscosity limits had to be established for each grade. During 1990 and 1991, a number of viscosity measurements were carried out on a number ofstandard production bitumens from all four South African refineries (Sapref, Natref, Genref and Calref). This data was used to establish the initial draft amendment to SABS 307 which was published in January 1993. Four grades: B5, B10, B20 and B35 were recommended.

As experience was gained with the initial draft specification, inter-laboratory test results indicated a wide scatter in measurements supposedly for like samples. It appeared that these differences arose from variations in the interpretation of the ASTM D4402 method. The variety of spindles which are supplied by Brookfield gave a wide range of viscosity results.

The South African Bureau of Standards (SABS) conducted a measurement assurance testing programme (MAP) on road grade bitumens with two main objectives:- Firstly, viscosity limits had to be set for bitumens conforming to the penetration based specification applicable at the time in order to arrive at a basis for the new specification. Secondly the various laboratories involved with manufacture and acceptance testing of the bitumens, had to assess their ability to conform to the specified reproducibility of the method.

In the MAP programme, samples of the standard production batches of each grade from each of the four South African refineries (Sapref, Calref, Natref and Genref) were submitted to the SABS. Each sample was split and distributed to the 6 laboratories, such that each tested all samples. Viscosity was measured at 60°C and 135°C according to the ASTM D4402 method. Details of the spindle sizes and rotational speeds used during the tests were reported together with viscosities.

Six sets of tests (referred to as 'MAP5, MAP6,....MAP10) were conducted between August 1992 and November 1993 (SABS, 13), during which time variations between laboratories interpretation of the method and use of equipment (non-specified spindles) were eliminated.

Figure 1 illustrates the performance of the 6 participating laboratories ability to test within the reproducibility of the method. Only results from MAPs 7 to 10 are included, as prior to that, the scatter was too wide for the results to be of any statistical value.

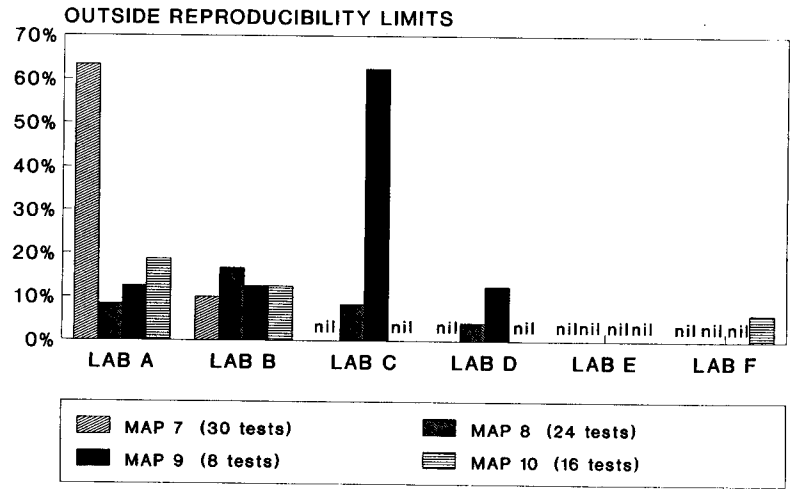

Figure 1: Reproducibility study showing percentage outliers for participating laboratories.

It is clear that whilst some laboratories showed improvement with gain in experience, others' performances were in some instances variable, with these experienced laboratories having difficulty in meeting reproducibility limits. This indicates the need for tight control in carrying out the specified method and a uniform approach industry-wide to the choice of equipment used.

When setting limits for specifications IP 367 (12) states that the value or values chosen shall take into account the reproducibility such that, for a double limit the specified range (upper - lower) shall not be less than four times R. By applying the IP 367 standard to setting specifications incorporating the ASTM method D4402, the specified range had to be at least as wide as 58% (4 times the reproducibility of 14,5%) of the intended midpoint of the range. This was applied in setting specified limits of viscosity of South African road grade bitumens. However some slight narrowing of ranges was necessary to avoid overlapping.

In November 1993 the final draft proposed ranges for road grade bitumens B4, B8, B12 and B24 (Table 2), were established by consensus from the results of MAPs 7 to 10, as well as from extensive in-house testing by the four bitumen-producing refineries. The MAP and in-house results are represented by the histogram plots shown in Figure 2.

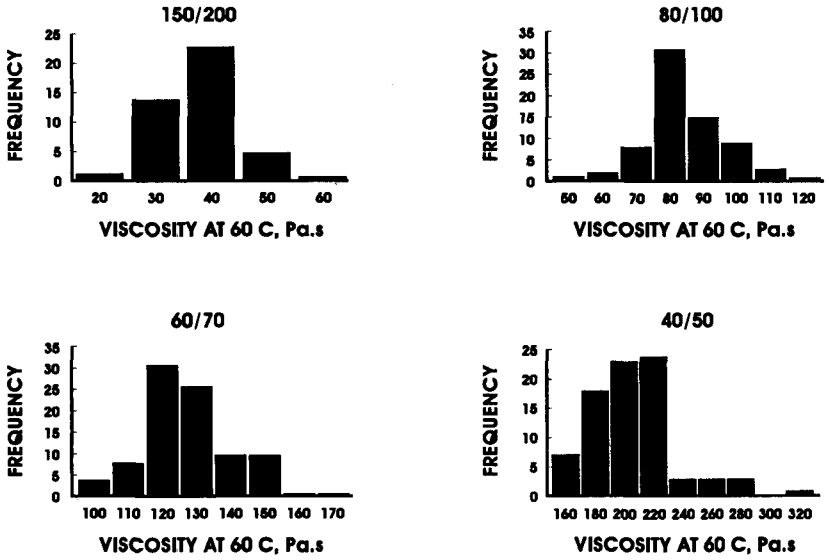

Figure 2: Frequencies of measured viscosity at 60°C for penetration grade bitumens in South Africa prior to January 1994.

4.2 Equipment for the Brookfield test

ASTM D4402 allows for any of the Brookfield viscometer models LV, RV, HA, or HB series to be used in the test. Allowable spindles include the SC-4 range as discussed in section 3.1.

Table 1: Viscosity measurement range for RV model Brookfield viscometers with recommended spindles

Spindle	Viscosity range (mPa.s)		
	RVT	RVF	RVF-100
SC4-21	50 - 100 000	250 - 25 000	250 - 25 000
SC4-27	250 - 500 000	1 250 - 125 000	250 - 25 000
SC4-29	1 000 - 2 000 000	5 000 - 500 000	1 000 - 100 000

The final recommendations from the MAP programme were that the Brookfield viscometer model; RVT with Thermosel System should be used for

specification purposes. The suggested spindle numbers; SC4-21, SC4-27, and SC4-29 and their ranges are given in Table 1.

4.3 SABS 307 road grade specification

The amended South African specification, derived from SABS 307 (1972) (1), with viscosity at 60°C replacing the penetration at 25°C as the main specifying parameter, was implemented in January 1994. Four grades are specified: B24, B12, B8, and B4. These names are based on the midpoint of the acceptable viscosity range.

The current viscosity ranges for the amended specification SABS 307 are given in Table 2.

Table 2: The amended specification for South African road grade bitumens

Property	Road grade (equivalent penetration grade)				Method
	B24 (40/50)	B12 (60/70)	B8 (80/100)	B4 (150/200)	
Viscosity,60°C, Pa.s	170 - 300	105 - 165	55 - 100	25 - 50	ASTM D4402[a]
Viscosity, 135°C, Pa.s. min	0,27	0,22	0,15	0,12	ASTM D4402[a]
Ductility, 10°C. mm	-	-	Report	Report	DIN 52013[b]
Ductility, 15°C. mm	Report	Report	-	-	DIN 52013[b]
Penetration, 25°C. 0,1mm	Report	Report	Report	Report	ASTM D5
Softening point (R&B). °C	Report	Report	Report	Report	ASTM D36
Spot test[c], % Xylene. max	30	30	30	30	ASSHTO T102
Performance in RTFOT					ASTM D2872
a. mass change, %m/m	0,5	0,5	0,5	0,5	ASTM D2872
b. visc ,60°C, % orig. max	300	300	300	300	ASTM D4402[a]
c. ductility, 10°C, mm	-	-	Report	Report	DIN 52013[b]
d. ductility,15°C, mm	Report	Report	-	-	DIN 52013[b]
e. softening pt, °C	Report	Report	Report	Report	ASTM D36
f. retained pen, % orig.	60	55	50	50	ASTM D5

Notes:

a. Recommended apparatus is the Brookfield model RV viscometer with Thermosel system, using SC-4 spindles.
b. Reliability of test to be confirmed before requirements are finalised.
c. Actuakl values to be reported in five-unit intervals.

5. COMPARISON BETWEEN BROOKFIELD AND OTHER VISCOSITY TEST METHODS

A number of comparisons were made between the Brookfield model RVT viscometers and other more commonly used methods.

5.1 Haake rotational viscometer.

To compare the results of Brookfield viscosity testing and a Haake rotational viscometer, the viscosity of a standardised control sample (a soft bitumen) was measured daily for 41 days, using a Haake model VT500 and two Brookfield model RVT viscometers with Thermosel systems (Raijmakers, 14). The testing was performed at 100°C because the Haake is not able to measure such viscous materials at 60°C. Then the control sample was changed and a further 6 days testing was performed. The operators varied, but the instruments were unchanged.

Table 3: Comparison of Haake VT500 and Brookfield RVT viscometers

Sample	Mean viscosity (cSt)[a]		
	Haake VT 500	Brookfield RVT (A)	Brookfield RVT(B)
1	461,2	451,4	447,7
2	444,3	450,0	447,2

Notes: a Converted from mPa.s to cSt by applying Eq. 1

Statistical analysis of the results showed differences between the Haake and Brookfield machines of the order of only 1-2% (Table 3), although they were statistically significant at the 99,99%. This is within the repeatability limits of the Brookfield standard and so results from the two test types are considered comparable at 100°C. It is considered that the two test types would also be comparable at 135°C due to the Newtonian characteristics of bitumen at both these temperatures.

5.2 Cannon Manning Vacuum Capillary viscometer (ASTM D2171).

To compare the results of Brookfield viscosity testing and the Cannon Manning vacuum capillary viscometer, a set of the 15 MAP7 samples, (one of each of 4 road grades from the Natref, Sapref and Genref and 3 from Calref), were tested for dynamic viscosity at 60°C according to ASTM method D2171. These were compared with the Brookfield results at 60°C from the MAP 7 run

It was found that the results from the ASTM D2171 method were consistently higher than the equivalent Brookfield measurements (Table 4). However, a good correlation was found between the two as shown graphically in Figure 3:

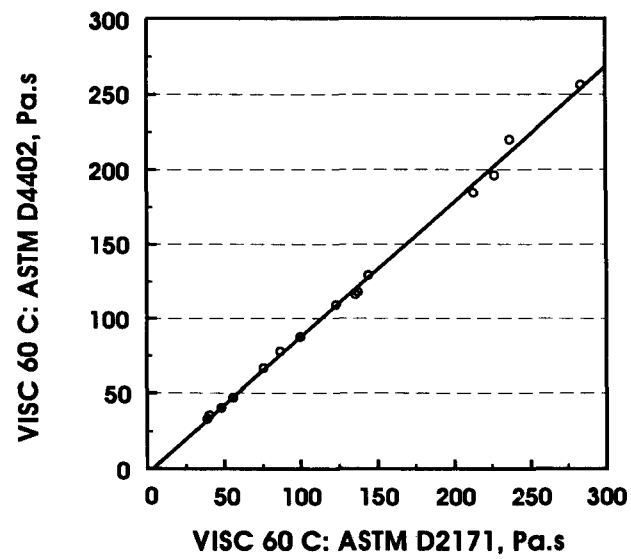

Figure 3: Viscosity at 60°C by ASTM D4402 vs. ASTM D2171

The two test methods are seen to be related by the equation:

$$V_{D4402} = 0{,}912(V_{D2171}) - 4{,}3 \qquad \text{(Eq. 2)}$$

where;

V_{D4402} = Dynamic viscosity, Pa.s at 60°C. ASTM D4402.

V_{D2171} = Dynamic viscosity, Pa.s at 60°C. ASTM D2171.

Coefficient of determination, $R^2 = 0{,}998$

5.3 U-tube Reverse Flow viscometer (ASTM D2170).

To compare the results of Brookfield viscosity testing and the U-tube Reverse Flow viscometer, MAP 7 samples were also tested at 135°C to ASTM method D2170 which measures the kinematic viscosity.

The density of each sample was measured at 25°C and converted to a density at 135°C The coefficient of cubical expansion used in this conversion was 0,00061, which is effectively independent of grade and is virtually constant over a temperature range of 15°C to 300°C (Pfeiffer,15). The density at 135°C was used in converting from kinematic to dynamic viscosity at this temperature

As with method D2171, these results were consistently higher than the MAP7 Brookfield results, but with good correlation between the two as depicted in Figure 4:

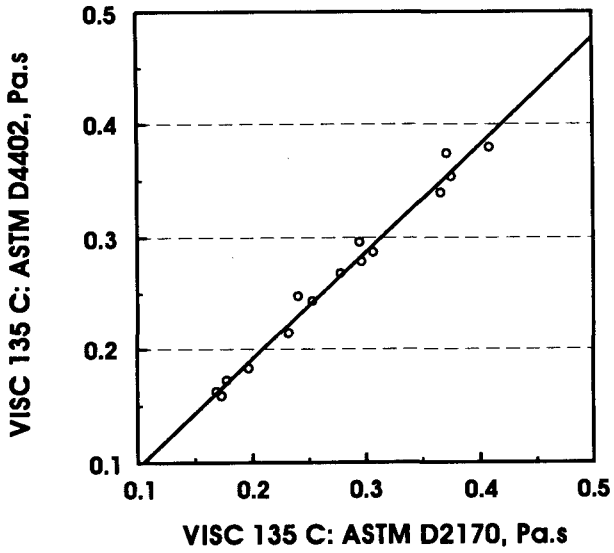

Figure 4: Viscosity at 135°C by ASTM D4402 vs. ASTM D2170

The two sets of results are related by the equation:

$$V_{D4402} = 0{,}955(V_{D2170})(DEN_{135}) - 0.002 \qquad \text{(Eq. 3)}$$

where;

V_{D4402} = Dynamic viscosity, Pa.s at 135°C. ASTM D4402
V_{D2170} = Kinematic viscosity, mm^2/s at 135°C. ASTM D2170
DEN_{135} = density of the bitumen at 135°C in kg/l.

Coefficient of determination, $R^2 = 0{,}986$

Table 5: Viscosities at 60°C and 135°C, measured by various methods.

Sample number BIT 7A/ 9305/...	Pen IP49	R&B IP58	Brook-field Dynamic Visc 60°C D4402 Pa.s	Brook-field Dynamic Visc 135°C D4402 Pa.s	Density 25°C D70 kg/l	Density 135°C Calculated (D70) kg/l	Cannon Manning Dynamic Visc 60°C D2171 Pa.s	U-tube rev. flow Kinematic Visc 135°C D2170 mm^2/s	Dynamic Visc 135°C Calculated (D70&D2170) Pa.s
B5A	160	40.00	46.60	0.183	1.0209	0.9540	56.41	205.41	0.196
B5B	151	43.00	39.90	0.170	1.0178	0.9511	47.67	188.50	0.179
B5C	181	41.00	33.70	0.158	1.0174	0.9507	39.14	182.21	0.173
B5D	169	42.00	34.00	0.160	1.0167	0.9501	41.01	178.76	0.170
B10A	70	49.00	109.60	0.266	1.0262	0.9589	124.13	291.62	0.280
B10B	86	46.00	86.40	0.240	1.0267	0.9594	100.19	265.65	0.255
B10C	98	45.00	76.20	0.245	1.0286	0.9612	87.65	250.85	0.241
B10D	104	44.00	65.70	0.214	1.0217	0.9547	76.00	243.40	0.232
B20A	64	49.00	118.30	0.278	1.0285	0.9611	137.96	309.32	0.297
B20B	66	48.00	117.10	0.295	1.0273	0.9600	136.94	308.99	0.297
B20C	68	51.00	127.90	0.286	1.0310	0.9634	144.50	317.41	0.306
B35A	43	52.00	219.10	0.354	1.0335	0.9658	236.20	387.90	0.375
B35B	38	50.00	256.10	0.379	1.0335	0.9658	282.80	423.38	0.409
B35C	52	50.00	183.80	0.339	1.0280	0.9606	212.90	382.16	0.367
B35D	55	50.00	198.50	0.373	1.0351	0.9700	226.20	384.00	0.372

6. CONCLUSIONS.

The bitumen specification SABS 307 (1) has been revised from a penetration based specification to a viscosity based specification, incorporating the ASTM D4402 test method with the Brookfield Thermosel Apparatus. The method and equipment used is capable of measuring the viscosity of bitumens over a wide range of temperatures and is relatively inexpensive.

A study of the repeatability and reproducibility of this test method has found that, whilst these limits are being met, even experienced laboratories sometimes have difficulties. It is suggested that this may be due to the non-Newtonian behaviour of some South African bitumens at 60°C, which means that the measured viscosity will be affected by shear rate, spindle geometry and probably thermal history.

To overcome the effects of thermal history it has been suggested that either the sample could be preheated to a much higher temperature, say 150°C and then cooled quickly to 60°C. This would minimise the dispersion of results, but then the sample would not represent what is being applied in the field. Alternatively, the thermal history of the test sample could be carefully controlled by specifying in detail, how the sample is to be brought to the test temperature and how the shear action of the viscometer is applied. This is

likely to be less successful in reducing scatter, but the results will more closely resemble the properties of the bitumen in the field.

The Brookfield test method must therefore be specified more tightly than is done in the ASTM D4402 test method. Recommendations are that the Brookfield model RVT viscometer and Thermosel system, with SC4-: 21, 27, and 29 spindles, is used for testing for conformance to the specification. To ensure quality control of viscosity measurements, an ongoing measurement assurance programme is required between laboratories using the Brookfield test method.

The Brookfield RVT and Haake VT500 rotational viscometers produce similar results at 100°C and it is considered that this would be the case at 135°C.

ASTM methods D2171 and D2170 produce results that correlate well with the Brookfield viscosity at 60°C and 135°C respectively. These methods can be used to approximate the Brookfield result by applying equations 2 and 3 described herein.

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