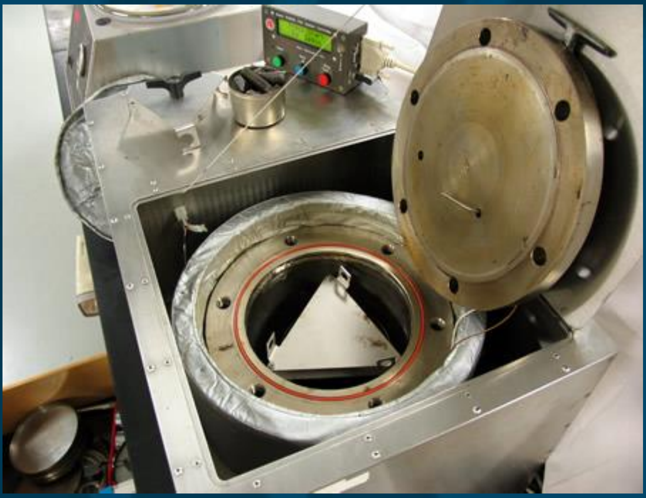

Laboratory Testing Protocols for Binders and Asphalt

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excellence in bituminous products

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LIST OF COMPANION SABITA MANUALS AND DVDs

Manual 1	*Technical guidelines: Construction of bitumen rubber seals*
Manual 2	*Bituminous binders for road construction and maintenance*
Manual 3 & 4	*(Withdrawn)*
Manual 5	*Guidelines for the manufacture and construction of hot mix asphalt*
Manual 6	*(Withdrawn)*
Manual 7	*SuperSurf – Economic warrants for surfacing roads*
Manual 8	*Guidelines for the safe and responsible handling of bituminous products*
Manual 9	*(Withdrawn)*
Manual 10	*Bituminous surfacing for low volume roads and temporary deviations*
Manual 11	*Labour enhanced construction for bituminous surfacings (This manual has been withdrawn and consolidated with Manual 12)*
Manual 12	*Labour Absorptive Methods in Road Construction using Bituminous Materials*
Manual 13	*LAMBs – The design and use of large aggregate mixes for bases*
Manual 14	*(Superseded by TG2)*
Manual 15 & 16	*(Withdrawn)*
Manual 17	*Porous asphalt mixes: Design and use*
Manual 18	*Appropriate standards for the use of sand asphalt*
Manual 19	*Guidelines for the design, manufacture and construction of bitumen rubber asphalt wearing courses* (CD)
Manual 20	*Sealing of active cracks in road pavements*
Manual 21	*(Superseded by TG2)*
Manual 22	*Hot mix paving in adverse weather*
Manual 23	*Code of practice: Loading bitumen at refineries*
Manual 24	*User Guide for the Design of Hot Mix Asphalt*
Manual 25	*Code of practice: Transportation, off-loading and storage of bitumen and bituminous products* (CD)
Manual 26	*Interim guidelines for primes and stone pre-coating fluids*
Manual 27	*Guidelines for thin hot mix asphalt wearing courses on residential streets*
Manual 28	*Best practice for the design and construction of slurry seals*
Manual 29	*Guide to the safe use of solvents in a bituminous products laboratory*
Manual 30	*A guide to the selection of bituminous binders for road construction*
Manual 31	*Guidelines for calibrating a binder distributor to ensure satisfactory performance*
Manual 32	*Best practice guideline and specification for warm mix asphalt*
Manual 33	*Design procedure for high modulus asphalt (EME)*
Manual 34	(A) *Guidelines to the transportation of bitumen and* (B) *Bitumen spill protocol*
Manual 35	*Design and use of Asphalt in Road Pavements*
Manual 36	*Use of Reclaimed Asphalt in the Production of Asphalt*
Manual 37	*Sampling Methods for Road Construction Materials – to be released in 2020*
Manual 38	*A Health & Safety Guide for Material Testing Laboratories in the Road Construction Industry*
Manual 39	*Laboratory Testing Protocols for Binders and Asphalt*
Manual 40	*Design and Construction of Surfacing Seals*

Technical guidelines

TG1	*The use of modified binders in road construction*
TG2	*Bitumen stabilised materials*
TG3	*Asphalt reinforcement for road condition*

DVDs

DVD100	*Test methods for bituminous products*
DVD200	*Training guide for the construction and repair of bituminous surfacings by hand*
DVD300	*Manufacture, paving and compaction of hot mix asphalt*
DVD410	*The safe handling of bitumen*
DVD420	*Treatment of bitumen burns*
DVD430	*Working safely with bitumen*
DVD440	*Firefighting in the bituminous products industry*
DVD450	*Safe loading and off-loading of bitumen*

PROTOCOLS FOR THE SOUTH AFRICAN BITUMEN PERFORMANCE GRADE SPECIFICATION

(PGP)

***Note**: All binder samples must be clearly labelled in order to identify its contents as testing an unidentified sample could represent a safety hazard and is thus not recommended. Sampling methods are detailed in Sabita Manual 37 / TMH 5 – Sampling Methods for Road Construction Materials*

PGP1: DETERMINING THE FLEXURAL CREEP PROPERTIES OF BITUMINOUS BINDER USING THE BENDING BEAM RHEOMETER (BBR)

1. INTRODUCTION

1.1 As part of the proposed SA bitumen PG grading testing, the aim is to develop detailed test protocols to ensure consistent and reliable results throughout the industry. This protocol covers the performance related test procedures for determing the flexural creep properties of bituminous binder using the bending beam rheometer. This protocol is for the determination of the flexural creep (S), m-value (m) and $\Delta T_{c.}$

1.2 This BBR protocol is in accordance with test methods ASTM D6648 and ASTM D7643. This protocol was developed based on the test method valid at the time of publication of this manual.

2. TESTING EQUIPMENT

2.1 ***Bending Beam Rheometer (BBR):*** Any BBR equipment that fulfills the requirements of ASTM D6648

2.2 ***Gauge block:*** A stepped gauge block with thickness measured to ±5 µm for calibrating and for verifying the calibration of the displacement transducer.

2.3 ***Test Specimen Moulds:*** stainless steel moulds with dimensions below. All dimensions should not vary more than ± 0.05 mm.

- Base bar: 6.35 mm (width) x 19.1 mm (depth) x 165 mm (length)
- Side bar: 6.35 mm (width) x 12.7 (depth) x 165 mm (length)
- End pieces: 6.35 mm (width) x 12.7 mm (depth) x 10.01 mm (length)

2.4 ***Plastic sheeting:*** Plastic sheeting used to line the mould should be 0.12 ± 0.04 mm thick and should not deform when hot binder is poured to the moulds.

2.5 ***Petroleum-Based Grease:*** Grease to hold the plastic strips to the moulds

***NOTE 1**: Silicone grease should not be used because silicone grease leaves a residue that is extremely difficult to remove even though the residue may not be visible*

2.6 ***Calibration and verification items:***

- Stainless steel (thick) beam: 6.4 ± 0.3 mm (thick) x 12.7 ± 0.3 mm (wide) x 127 ± 5 mm (long).

***NOTE 2**: Given the beam is used to calculate the thickness of the test specimens; it should be measured to the nearest 0.01mm.*

- Stainless steel (thin) beam: 1.0 to 1.6 mm (thick) x 12.7 ± 0.1 mm (wide) x 127 ± 5 mm (long).

***NOTE 3**: Given the beam is used to calculate the modulus of the test specimens; it should be measured to the nearest 0.01mm.*

- Mass pieces: four (4) 100.0 ± 0.2 g mass pieces for load cell calibration.
- Mass pieces: two (2) 2.0 ± 0.2 g mass pieces for load cell verification.

2.7 **Bath fluid:** A bath fluid that is not absorbed by or does not affect the properties of the binder being tested i.e. Methanol AR, at least 95% purity.

2.8 **Temperature probe:** a temperature probe that has been calibrated to two decimal places.

3. PREPARATION OF APPARATUS

3.1 Make sure the instrument is level

3.2 Make sure solvent level is at the recommended limit

3.3 *Switching on the intrument*

(i) Turn on the instrument

(ii) Make sure the anti-freeze is circulating

(iii) Make sure the magnetic stirrer is circulating

(iv) Make sure the instrument is set at LOCAL/ZERO

3.4 Adjust the external, line and bearing pressure

3.5 Check there is no beam below the load cell

3.6 Select the test temperature and allow the bath to equilibrate at the test temperature ±0.1°C before conducting a test.

Recommendation: It should not be less than 15 minutes

3.5 Turn on the data acquisition system and start the software as per manufacturer's manual.

3.6 *Verification of displacement transducer* – on each day before conducting any tests, verify by using a stepped-gauge block. Remove any beams from the bath and place the gauge block on the reference platform according to manufacturer's instructions. Apply a 100 g mass piece to the loading shaft and measure the rise in steps with the displacement transducer.

NOTE 4: Failure to pass this criteria implies an external calibration is required.

3.7 *Verification of freely operating air bearing* – on each day before conducting any tests, verify that the air bearing is operating freely and free of friction. Place the thin beam on the sample support and apply a 35 ± 10 mN load on the beam using the zero load regulator. Gently lift the loading shaft to ± 5 mm, and on release the shaft should immediately float downward and make contact with the beam.

Remove all beams from the supports. Use the zero load regulator to adjust the loading shaft so that it floats freely. Gently add the 2 g mass piece to the loading shelf and check to see if the shaft slowly drops downward under the weight.

3.8 *Verification of load cell* – the below verifications of the load cell should be carried out on each day before conducting any tests:

(i) *Contact Load* –verify the load cell in the range of the contact load. Place the thick beam onto the support. Using the zero load pressure regulator apply a load of 20 ± 10 mN. Add a 2.0 g mass piece to the loading platform and an increase of load increase displayed should be 20 ± 5 mN. Add another 2.0 g to the loading platform and the load reading should increase by 20 ± 5 mN. If this is not the case, then a calibration must be carried and verification repeated. Otherwise, please consult with the manufacturer.

(ii) *Test Load* –verify the calibration of the load cell. Place 6.35 mm thick beam on the supports and using the zero load regulator, apply 20 ± 10 mN load to the beam. Add 100 g mass piece to the loading platform and observe for an increase of 981 ± 5 mN. If out of range, calibrate and repeat. If requirements are still not met, please consult with the manufacturer.

(iii) Ensure that the loading shaft is at the vertical position that it will occupy at the start of the test.

(a) Determine the vertical position of the shaft by placing the thick beam on the supports and applying a 100 g mass piece on the loading platform. The reading displayed indicates, approximately, the position of the shaft during testing of a 6.35 mm beam,

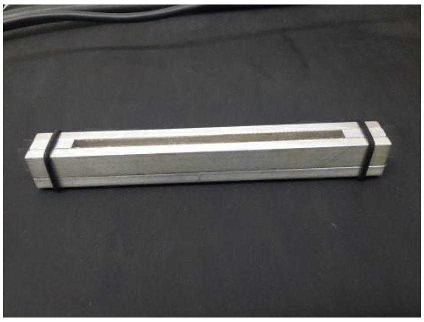
(b) The BBR shall display 0 ± 5 mN while free floating, otherwise repeat. If requirements are still not met, please consult with the manufacturer.

3.9 *Verification of Thermometric Device* – before carrying out a test at the desired temperature using a temperature probe that has been calibrated to two decimal places.

3.10 *Daily Overall System Check (confidence test)* – before any testing begins, perform an overall system's check. Place the thin stainless steel beam on the sample support and following the manufacturers' instructions place a 100 g mass piece to the beam. Apply another 100 to 300 g mass piece to the beam. The manufacturers' software shall use the change in load to calculate the modulus of the beam and report it to three significant figures.

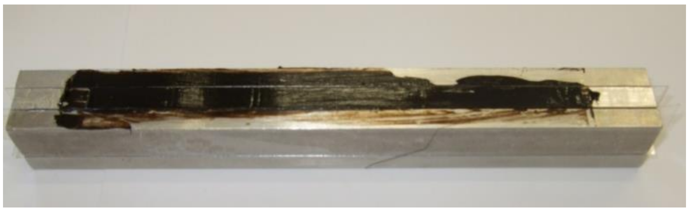
4. SAMPLE PREPARATION

4.1 Assemble a test beam mould that will yield a demolded test beam sample of 6.35 ± 0.05 mm thick by 12.70 ± 0.05 mm wide by 127 ± 2.00 mm long.



4.2 Pour the sample from the degassing oven. If left for storage, heat the stored PAV aged binder until it is sufficiently fluid to pour.

4.3 Pour the sample in the mould by beginning from one end to the other in one single pass. Slightly overfill the mould.



4.4 Leave the filled mould at room temperature to cool for 45 to 60 min.

4.5 Trim the excess sample with a hot spatula or knife.

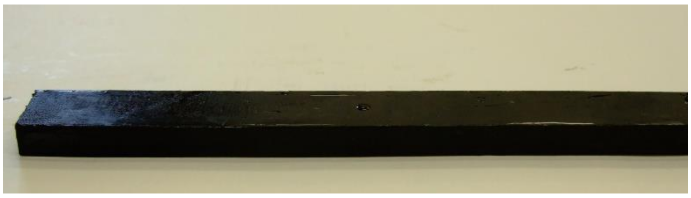
4.6 Leave the mould at room temperature prior to testing.

***NOTE 5**: Time-dependent increases in stiffness can occur when an asphalt binder is stored at room temperature for even short periods of time.*

Recommendation: Leave the sample at room temperature for an additional 45 to 60 min.

4.7 Just before testing, cool the sample in the freezer or ice bath for no longer than 5 min then quickly demould the specimen.

4.8 Place the test specimen in the BBR bath at the testing temperature for 60 ± 5min before testing.



***NOTE 6**: Measure the thickness of the sample. Thickness can be measured using either the thickness gauge or using the auto-thickness option if the instrument has it.*

***NOTE 7**: Testing shall be completed within four (4) hours after specimens are poured.*

5. TEST PROCEDURE

5.1 Place the thick stainless steel beam in position and adjust:

a. Zero force to 35 ± 10 mN using the zero regulator valve
b. Load force to 980 ± 50 mN using the load regulator valve

***NOTE 8**: Sample will break at the start of the test if the zero force is outside the specification*

5.2 Enter the sample identification details. Place the sample in the test support and lower the shaft to make contact with the sample. When the contact is made the load must be 35 ± 10 mN.

5.3 Activate the automatic test system and run the test.

5.4 The data acquisition will generate results of measured stiffness (*S*) and *m*-value (*m*) at loading times of 8.0, 15.0, 30.0, 60.0, 120.0 and 240.0 seconds.

5.5 Test at more than one temperature until *S* (60) ≤ 300MPa and *m* (60) ≥ 0.3 using a fresh sample for each temperature. Use the below recommendations:

Table 1: Bituminous Binder Grades and their BBR Testing Temperatures

PG Grade	BBR testing temperatures (°C) PAV-aged binder only
58-22	-6, -12, -18
64-16	0, -6, -12, -18
70-10	0, -6, -12, -18

NOTE 9: BBR test is done at low temperature grade + 10°C.

5.6 Print the results for stiffness (*S*) and *m*-value (*m*).

6. SHUTTING DOWN

6.1 Remove any beams from the support and heat the bath to ± 5°C.

6.2 Raise the load shaft and let it rest on the gauge block.

6.3 Turn off the ZERO and LOAD pressure.

6.4 Remove approximately 500 ml of the solvent (levels tend to rise).

6.5 Turn OFF the instrument.

7. CALCULATIONS

7.1 Calculations will be done in accordance with ASTM D6648 and ASTM D7643.

7.2 a. Calculate $T_{c,S}$:

The interpolation for each of the properties, except for *m*-value, shall be done using a linear relationship between the test results (log_{10} scale) and the test temperature (arithmetic scale). The interpolation takes the following form:

$$T_c = T_1 + ((\log_{10}(P_S) - \log_{10}(P_1))) / (\log_{10}(P_2) - \log_{10}(P_1)) \times (T_2 - T_1)$$

Where:

T_c = *Continuous grading temperature, °C,*

T_1 = *Lower of the two test temperatures, °C,*

P_s = *Specification requirement for property in question; determined at the respective PG grading temperature for the respective property,*

P_1 = *Test result for the specification property in question at* T_1*,*

P_2 = *Test result for the specification property in question at* T_2*, and*

T_2 = *Higher of two test temperatures, °C*

b. Calculate $T_{c,m}$*:*

The interpolation for the m-value shall be done using an arithmetic scale. The interpolation takes the following form:

$$T_c = T_1 + ((P_s - P_1) / (P_2 - P_1)) \times (T_2 - T_1)$$

c. Calculate ΔT_c:

1. *ΔT_c is the difference between $T_{c,S}$ [S(60sec)=300MPa] and $T_{c,m}$ [m(60sec)=0.3]*
2. *$\Delta T_c = T_{c,S\,(300MPa)} - T_{c,m\,(0.3)}$.*

***NOTE 10**: When performing the interpolation for stiffness or m-value ensure that the two results chosen for this calculation should be 6-degrees Celsius apart. Different temperatures may be required for the stiffness and m-value interpolations. If the temperatures given in section 5.5 do not allow for interpolation, then additional temperatures shall be added in 6-degree increments until interpolation is possible.*

8. TEST REPORT

8.1 Report the following:

1. a) measured flexural creep stiffness, *S* (8s, 15s, 60s, 120s , 240s) MPa reported to three significant figures at the test temperatures.
   b) measured *m*-value, *m* (60s) reported to the nearest 0.001 at the test temperatures.

2. Pass/Fail at the low temperature grade. Pass criteria: S (60) ≤ 300MPa and m (60) ≥ 0.3.

3. Calculated ΔT_c in 7.2 above.

4. Beam dimensions.

9. CALIBRATIONS & VERIFICATIONS

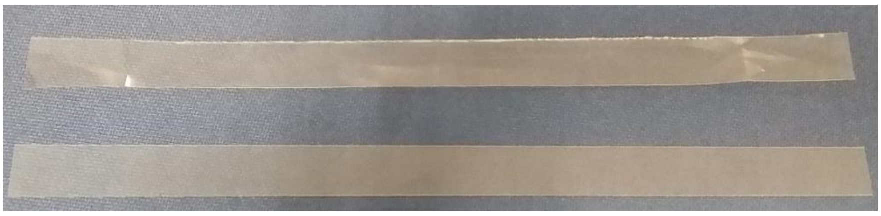
9.1 *Verification of Front-to-Back Alignment of Loading Shaft* – After installation or major disturbance through moving and handling.

9.2 Every three months, verify the force calibration following the manufacturer's instruction.

9.3 Change the solvent in the BBR regularly. A monthly change is recommended depending on testing frequency.

9.4 Change the anti- freeze in the air/water heat exchanger yearly.

9.5 Make sure that the plastic sheeting is changed once damaged/distorted as this affects the thickness of the sample.



BAD

GOOD

9.6 **Recommendation:** Instrument to be coupled to a UPS for power back-up in case of power cuts.

10 PRECISION AND BIAS

10.1 Table 2 contains single-operator precision data performed at the CSIR, showing: BBR measured *S* and *m*-value properties with coefficient of variation (COV) of ≤ 1.6%.

Table 2: Single-Operator Precision at -6°C

Randomly selected binders	Modification	Number of repeats	Measured COV (%)	
			S	***m***
1	Unmodified	4	0.4	1.3
2	Unmodified	4	0.3	1.0
3	Modified	4	0.8	1.6
4	Modified	4	1.2	0.0
5	Unmodified	4	0.8	0.9
6	Unmodified	4	1.6	0.1
7	Modified	4	1.0	0.3

11. REFERENCES

11.1 ASTM D6648: Determining the flexural creep stiffness of asphalt binder using the bending beam rheometer. American Society for Testing and Materials (ASTM) International.

11.2 ASTM D7643: Determining the continuous grading temperatures and continuous grades for PG graded asphalt binders. American Society for Testing and Materials (ASTM) International.

11.3 Technician's manual for specification of asphalt binders: MS-25 (3rd edition). Asphalt Institute. 2012.

11.4 Instruction and operation manual for TE-BBR models version 1.2g. CANNON® Instrument Company. May 2008.

PGP2: DETERMINING THE RHEOLOGICAL PROPERTIES OF BITUMINOUS BINDER USING A DYNAMIC SHEAR RHEOMETER: (G*, δ)

1. INTRODUCTION

1.1 As part of the proposed SA bitumen PG grading testing, the first step in this process is to develop detailed test protocols to ensure consistent and reliable results throughout the industry. This protocol covers the determination of rheological properties of asphalt binders using a Dynamic Shear Rheometer - (G*/sinδ).

1.2 The rheological properties will be determined in accordance with test method ASTM D7175. This protocol was developed based on the test method valid at the time of publication of this manual.

2. TESTING INSTRUMENT

2.1 ***Dynamic Shear Rheometer (DSR):*** Any DSR that fulfils the requirements of ASTM D7175.

2.2 ***Test Plates:*** Cylindrical metal lower and upper measuring plates of 25 ± 0.05 mm in diameter with a smooth ground surface.

2.3 ***Trimming tool:*** A tool suitable to trim excess binder from th*e* test plates.

2.4 ***Specimen mould:*** Silicon rubber mould for sample preparation.

2.5 ***Materials:***

- *Wiping material:* clean paper towel or clean cloth to wipe the test plates.
- *Cleaning solvent:* solvent to clean the test plate. After cleaning, the plate should have no residue.

NOTE 1: Acetone can be used to remove any residue left from the initial cleaning.

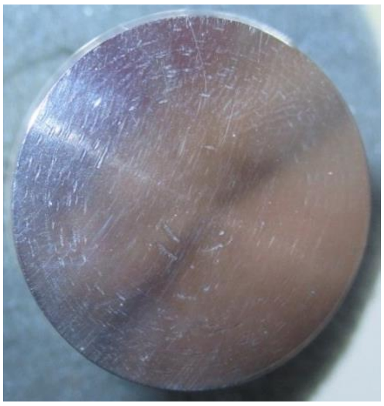
2.6 ***Oven:*** An oven that can maintain any temperature up to 260°C ± 3°C.

2.7 ***Hot plate/heating gun:*** A heating equipment to warm up the trimming tool.

3. PREPARATION OF INSTRUMENT

3.1 Follow manufacturer’s recommendations to prepare the instrument.

3.2 Inspect the test plates to ensure they have no scratches and/or dents.



BAD **GOOD**

3.3 Use a solvent to clean the test plates.

3.4 Follow manufacturers' instruction and select the correct workbook for testing the following:

- (*G*/sinδ*) - Unaged binder.

3.5 Mount the upper measuring plate to the instrument ensuring that it is aligned.

3.6 Attach the lower measuring plate to the instrument. Firmly tighten it.

3.7 Select the testing temperature to be used according to the SA specification. See Table 1 below:

Table 1: Maximum Pavement Design Temperature for South Africa

Maximum pavement design temperature (°C)		
58	64	70

3.8 Use the manufacturer's instruction to zero the gap at the selected temperature and correct gap size. If multiple temperatures are going to be used, zero gap at the middle temperature of the temperature range. Use the same sample for all temperatures.

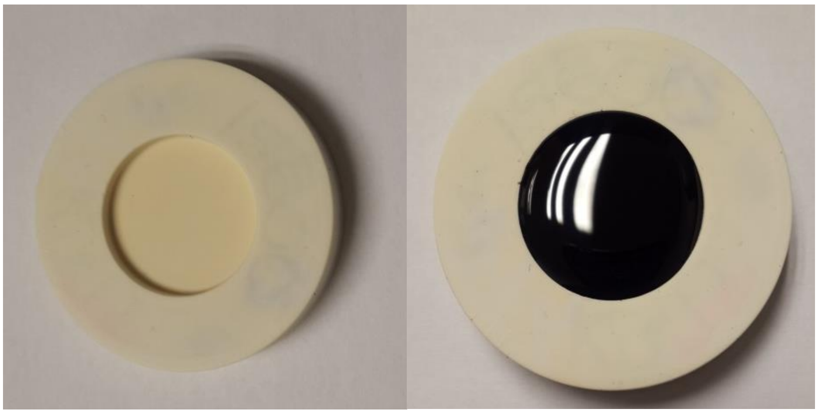
NOTE 2: Make sure that the correct gap size is selected for the 25-mm measuring plates.

- 25-mm (1-mm gap size).
- 25-mm (2-mm gap size for bitumen rubber).

4. SAMPLE PREPARATION METHOD

4.1 Prepare the unaged binder sample according to the sample preparation protocol.

4.2 Pour the hot binder prepared above into a silicon mould and leave to cool at room temperature.



Recommendation: A minimum mass of 0.6 g is required when testing with a 25-mm plate. Testing should be completed within 4 hours from the time the sample is poured on the mould.

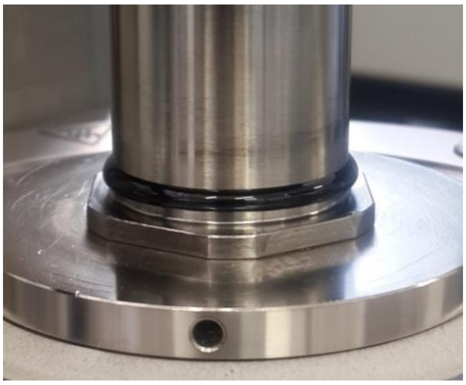
5. SAMPLE LOADING AND TRIMMING

5.1 Heat up the bottom measuring plate to loading temperature.

Recommendation: A loading temperature of 70°C is recommended.

5.2 **Take a wide and flat spatula and heat it up with a heating gun or hotplate. Use the heated spatula to warm up the upper measuring test plate by attaching the spatula to the upper measuring test plate while it is mounted to the instrument for approximately 10 sec.**

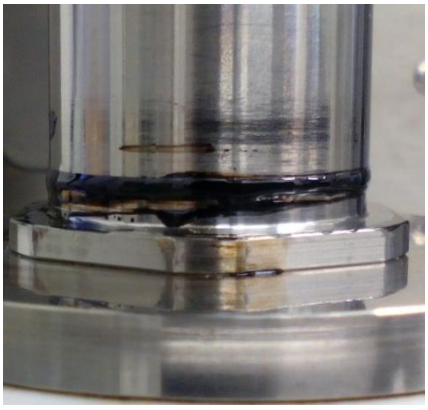
5.3 Demould the prepared sample and attach it **to the upper plate and lower the upper** measuring plate to trimming position (gap between the plates that equals the testing gap plus the gap closure required to create a bulge) – see picture below.



5.4 Heat up the trimming tool on the hot plate/ heating gun (it must be hot enough to be able to remove the sample) and use it to trim the excess binder by moving the tool around the edges of the plates so that the binder is even with the outer diameter of the plates.

NOTE 3: Use short movements when trimming and remove small amounts of the sample at a time.

5.5 After trimming, lower the upper measuring test plate to the measuring gap position to form a slight bulge – see picture below.



6. TESTING PROCEDURE

6.1 Test the G*/sinδ of the samples prepared in 5.4 above.

6.2 When testing at multiple temperatures, start testing from the lowest temperature.

6.3 Condition the sample at the selected starting temperature for ten (10) minutes before testing starts.

6.4 Test at a standard frequency of 10 rad/s and **at 12% LVE strain as per workbook.**

6.5 Obtain measurements of complex modulus (G*) and phase angle (δ) of between 8 to 16 loading cycles.

7. CALCULATIONS

7.1 Get an average of the complex modulus (G*) and the phase angle (δ) of the loading cycles.

7.2 Obtain the *G*/sinδ* of the averaged values using the software provided by the manufacturer.

8. REPORT

8.1 *G*, sinδ*, and *G*/sinδ* for unaged binder at $T_{max.}$ T_{max} is the design maximum pavement temperature.

9. CALIBRATIONS AND VERIFICATIONS

9.1 *Temperature verification:* follow manufacturer's recommendations and verify the temperatures (include 58°C, 64°C and 70°C) by using a rubber wafer or a dummy test specimen every ninety (90) days.

9.2 *Motor adjustment:* follow manufacturer's recommendations and do a motor adjustment with the measuring test plates at the correct gap sizes every ninety (90) days.

9.3 *External calibration:* done by the manufacturer yearly for properties such as temperature and torque.

9.4 *Instrument verification*: Use a reference fluid to check the overall performance of the instrument using the test temperature of 64°C. It is recommended that this verification check is performed on a weekly basis.

10. PRECISION AND BIAS

10.1 Data for the determination of single- and multiple-operator precision for the complex modulus (G*) performed at the CSIR is presented in Table 2.

Table 2: Single and Multiple Operator Precision

Material Properties	**G* of penetration grade bitumen Model Smartpave Plus**	**G* of SBS - modified binder Model Smartpave Plus**
Operator 1		
Result #1	7.0	10.0
Result #2	6.8	9.8
Result #3	6.6	9.7
Result #4	6.8	9.6
Result #5	6.9	9.5
Average for Operator 1	6.8	9.7
Standard deviation for Operator 1	0.15	0.19
Coefficient of Variation for Operator 1	**2.3%**	**1.9%**
Operator 2		
Result #1	-	11.7
Result #2	7.2	11.2
Result #3	7.1	11.1
Result #4	7.0	10.7
Result #5	7.1	11.5
Average for Operator 2	7.1	11.2
Standard deviation for Operator 2	0.06	0.39
Coefficient of Variation for Operator 2	0.9%	3.4%
Multi-operator Average	**7.0**	**10.5**
Multi-operator Standard deviation	**0.20**	**0.87**
Multi-operator Coefficient of Variation	**2.8%**	**8.3%**

11. REFERENCES

11.1 ASTM D7175: Determining the rheological properties of asphalt binder using a dynamic shear rheometer. American Society for Testing and Materials (ASTM) International.

11.2 Technician’s manual for specification testing of asphalt binders: MS-25 (3rd edition). Asphalt Institute, 2012.

11.3 CSIR test protocol for determining the rheological properties using dynamic shear rheometry GWDMS: 180052 (2009).

PGP3: DETERMINING THE RHEOLOGICAL PROPERTIES OF BITUMINOUS BINDER USING A DYNAMIC SHEAR RHEOMETER: Frequency sweep (*and the ageing ratio*)

1. INTRODUCTION

1.1 As part of the proposed SA bitumen PG grading testing, the first step in this process is to develop detailed test protocols to ensure consistent and reliable results throughout the industry. This protocol covers the determination of rheological properties of asphalt binders using a Dynamic Shear Rheometer.

1.2 The rheological properties will be determined in accordance with ASTM standard test method ASTM D7175. This test is performed on unaged, RTFO aged and PAV aged samples at the intermediate temperature for the PG grade. It is used to calculate ageing ratios and develop frequency sweeps for master curves**. This protocol was developed based on the test method valid at the time of publication of this manual.**

2. TESTING INSTRUMENT

2.1 ***Dynamic Shear Rheometer (DSR):*** Any DSR that fulfils the requirements of ASTM D7175.

2.2 ***Test Plates:*** Cylindrical stainless steel metal lower and upper measuring plates of 8 ± 0.1 mm and 25 ± 0.05 mm in diameter with a smooth ground surface.

2.3 ***Trimming tool:*** A tool suitable to trim excess binder from the test plates

2.4 ***Specimen mould:*** Silicon rubber mould for sample preparation

2.5 ***Materials:***

- *Wiping material:* paper towel or clean cloth to wipe the test plates
- *Cleaning solvent:* solvent to clean the test plate. After cleaning, the plate should have no residue.

NOTE 1: Acetone can be used to remove any residue left from the initial cleaning.

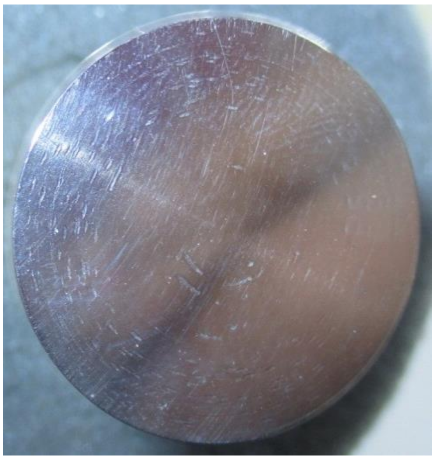
2.6 ***Oven:*** An oven that can maintain any temperature up to 260°C ± 3°C.

2.7 ***Hot plate / heating gun:*** A heating equipment to warm up the trimming tool

3. PREPARATION OF INSTRUMENT

3.1 Follow manufacturer's recommendations to prepare the instrument.

3.2 Inspect the test plates to ensure they have no scratches and/or dents.



BAD GOOD

3.3 Use a solvent to clean the test plates.

3.4 Follow manufacturer's instruction and select the correct workbook for testing the frequency sweeps

3.5 Mount the upper test plate to the instrument ensuring that it is aligned.

3.6 Attach the bottom plate to the instrument. Firmly tighten it.

3.7 Select the intermediate testing temperature to be used according to the SA specification. See Table 1 below:

Table 1: Intermediate Temperature (TIT) for SA Performance Grades

PG Grading	Intermediate Temperatures (°C)
58-22	22
64-16	28
70-10	34

3.8 Use the manufacturer's instruction to zero gap at the selected temperature and gap size. Zero gap at the middle temperature of the temperature range.

***NOTE 2**: Make sure that the correct gap size is selected for the different measuring plates.*

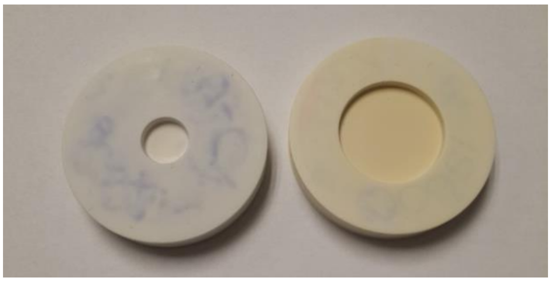
- 8-mm (2-mm gap size)
- 25-mm (1-mm gap size)
- 25-mm (2-mm gap size for bitumen rubber)

***NOTE 3**: 8-mm plates will be used unless the G* < 100 kPa. If G* < 100kPa, then a 25-mm plates will be used.*

4. SAMPLE PREPARATION METHOD

4.1 Prepare the binder sample according to the sample preparation protocol.

4.2 Pour the hot binder prepared above into the relevant silicon mould and leave to cool at room temperature – see picture below.



Recommendation: A minimum mass of 0.6 g if testing will be conducted using a 25-mm plate and a minimum mass of 0.2 g if testing will be conducted using an 8-mm plate. Testing should be completed within 4 hours from the time the sample is poured on the mould.

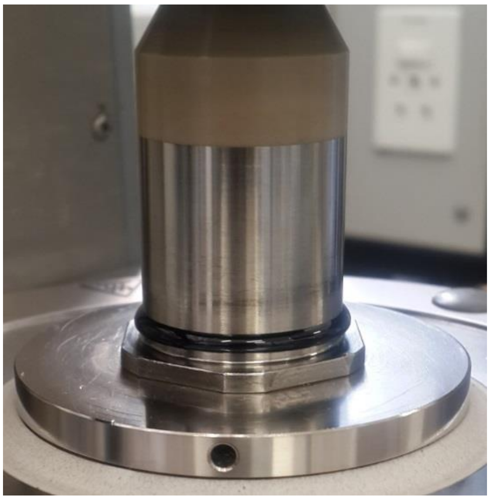
5. SAMPLE LOADING AND TRIMMING

5.1 Heat up the bottom measuring plate to loading temperature.

Recommendation: A loading temperature of 70°C is recommended.

5.2 Take a wide and flat spatula and heat it up with a heating gun or hotplate. Use the heated spatula to warm up the upper measuring test plate by attaching the spatula to the upper measuring test plate while it is mounted to the instrument for approximately 10 sec.

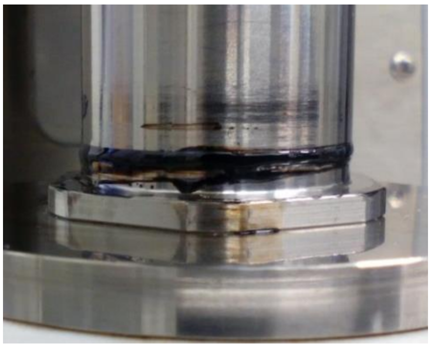
5.3 Demould the prepared sample and attach it to the upper plate and lower the upper measuring plate to trimming position (*gap between the plates that equals the testing gap plus the gap closure required to create a bulge*) – see picture below.



5.4 Heat up the trimming tool on the hot plate/heating gun (*it must be hot enough to be able to remove the sample*) and use it to trim the excess binder by moving the tool around the edges of the plates so that the binder is even with the outer diameter of the plates.

***NOTE 4*: Use short movements when trimming and remove small amounts of the sample at a time.**

5.5 After trimming, lower the upper measuring test plate to the measuring gap position to form a slight bulge – see picture below.



6. TESTING PROCEDURE

6.1 Test the frequency sweep of the samples prepared in 5.4 above.

6.2 The G* and δ will be measured at the intermediate temperatures given in Table 2 below. 8-mm plates will be used unless the G* < 100 kPa. If G* < 100 kPa, then 25-mm plates will be used. Strain levels and frequency range to be used are given in Table 3 and Table 4 respectively:

Table 2: Intermediate Temperature per PG Grading

PG Grading	Intermediate Temperatures (°C)
58-22	22
64-16	28
70-10	34

Table 3: Strain Level to use on Different Plate Sizes

Plate size (mm)	Strain level (%)
8	1
25	2

Table 4: Frequency Range

Log basis (rad/s)	Linear basis (rad/s)	Linear basis (Hz)
-0.6	0.251	0.0400
-0.4	0.398	0.0634
-0.2	0.631	0.100
-0.0	1.00	0.159
+0.2	1.58	0.252
+0.4	2.51	0.400
+0.6	3.98	0.634
+0.8	6.31	1.00
+1.0	10.0	1.59
+1.2	15.8	2.52
+1.4	25.1	4.00

6.3 Condition the sample at the selected starting temperature for ten (10) minutes before testing starts (unless thermal equilibrium analysis shows otherwise).

6.4 The frequency sweep will be conducted on unaged, RTFO and PAV aged samples.

7. CALCULATIONS

7.1 Calculate the aging ratio: ageing ratio is the ratio of the RTFO and PAV aged binders versus the unaged binder calculated at 10.0 rad/s.

- $G^*_{RTFO}/G^*_{Original}$
- $G^*_{PAV}/G^*_{Original}$

8. REPORT

8.1 Ageing ratios calculated in 7.1 above to one decimal place.

8.2 Complex modulus and phase angle values at the frequency range given in Table 4 above.

9. CALIBRATIONS AND VERIFICATIONS

9.1 *Temperature verification:* follow manufacturer's recommendations and verify the temperatures (include 22°C, 28°C and 34°C) by using a rubber wafer or a dummy test specimen every ninety (90) days.

9.2 *Motor adjustment:* follow manufacturers' recommendations and do a motor adjustment with the measuring spindles at the correct gap sizes every ninety (90) days.

9.3 *External calibration:* done by the manufacturer yearly for properties such as temperature and torque.

10. PRECISION AND BIAS

10.1 Data for the determination of single-operator precision performed at the CSIR is presented in Table 5.

Table 5: Single - Operator Precision for Guidance Only (Stiffness Range is Not Applicable)

Binder Type	Gap / mm	Frequency / Hz									
		0.0126		0.126		1.26		12.6		50.2	
		δ	G*	δ	G*	δ	G*	δ	G*	δ	G*
60/70pen	1.0	89.6	40.0	88.5	397	85.8	3 710	82.2	31 900	80.3	110 000
60/70pen	1.0	89.6	41.0	88.4	402	85.6	3 750	82.0	32 100	80.0	111 000
60/70pen	1.0	89.6	41.0	88.5	403	85.7	3 750	81.8	32 200	78.6	111 000
60/70pen	1.0	89.5	40.4	88.4	396	85.6	3 690	82.0	31 700	79.4	109 000
60/70pen	1.0	89.6	39.0	88.5	387	85.8	3 620	82.0	31 200	79.0	109 000
60/70pen	1.0	89.6	39.0	88.6	382	85.9	3 570	82.4	30 800	80.7	107 000
Average	**1.0**	**89.6**	**40.1**	**88.5**	**395**	**85.7**	**3682**	**82.1**	**31650**	**79.7**	**109500**
σ (60/70pen)	**1.0**	**0.04**	**0.95**	**0.08**	**8**	**0.12**	**72.8**	**0.21**	**546.8**	**0.80**	**1516.6**
Coeff. of Variation	**1.0**	**0.0**	**2.3**	**0.1**	**2**	**0.1**	**2**	**0.3**	**2**	**1.0**	**1**

11. REFERENCES

11.1 ASTM D7175: Determining the rheological properties of asphalt binder using a dynamic shear rheometer. American Society for Testing and Materials (ASTM) International.

11.2 Technician's manual for specification testing of asphalt binders: MS-25 (3rd edition). Asphalt Institute, 2012.

11.3 CSIR DSR work instruction: WI-CSIR-BE-LAB-20.36 (2013).

PGP4: MULTIPLE STRESS CREEP AND RECOVERY (MSCR) OF BITUMINOUS BINDERS USING A DYNAMIC SHEAR RHEOMETER

1. INTRODUCTION

1.1 As part of the proposed SA Bitumen PG grading testing, the first step in this process is to develop detailed test protocols to ensure consistent and reliable results throughout the industry. This protocol covers the determination of non-recoverable creep compliance of asphalt binders by means of Multiple Stress Creep and Recovery (MSCR) testing.

1.2 The MSCR will be performed in accordance with test method ASTM D7405. This protocol was developed based on the test method valid at the time of publication of this manual.

2. TESTING INSTRUMENT

2.1 ***Dynamic Shear Rheometer (DSR):*** Any DSR that fulfils the requirements of ASTM D7175.

2.2 ***Test Plates:*** Cylindrical metal lower and upper measuring plates of 25 ± 0.05 mm in diameter with a smooth ground surface.

2.3 ***Trimming tool:*** A tool suitable to trim excess binder from th*e* test plates.

2.4 ***Specimen mould:*** Silicon rubber mould for sample preparation.

2.5 ***Materials:***

- *Wiping material:* paper towel or clean cloth to wipe the test plates.
- *Cleaning solvent:* solvent to clean the measuring test plates. After cleaning, the plates should have no residue.

NOTE 1: Acetone can be used to remove any residue left from the initial cleaning.

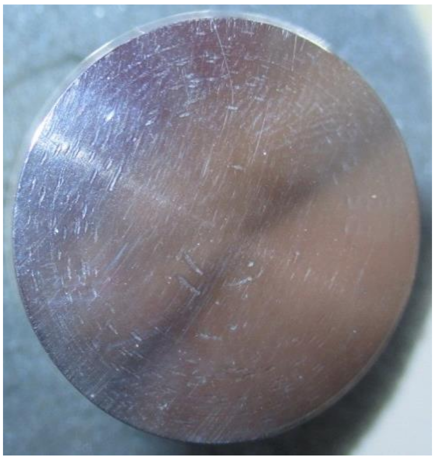
2.6 ***Oven:*** An oven that can maintain any temperature up to 260°C ± 3°C.

2.7 ***Hot plate / heating gun:*** A heating equipment to warm up the trimming tool.

3. PREPARATION OF INSTRUMENT

3.1 Follow manufacturer's recommendations to prepare the instrument.

3.2 Inspect the test plates to ensure they have no scratches and/or dents.



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GOOD

3.3 Use a solvent to clean the measuring test plates.

3.4 Follow manufacturers' instruction and select the correct workbook for testing the following:

- Multiple Stress Creep and Recovery (MSCR) after RTFO ageing.

3.5 Mount the upper measuring plate to the instrument ensuring that it is aligned.

3.6 Attach the lower measuring plate to the instrument. Firmly tighten it.

3.7 Select the T_{max} i.e. the design maximum pavement temperature. See Table 1 below:

Table 1: Maximum pavement design temperature for South Africa

Maximum pavement design temperature (°C)		
58	64	70

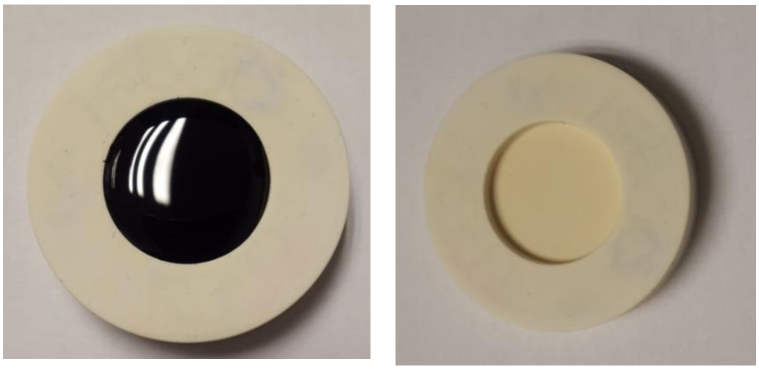
3.8 Use the manufacturer's instruction to zero gap at the selected temperature and gap size. Use a different sample for each temperature.

***NOTE 2**: Make sure that the correct gap size is selected for the 25-mm measuring plates.*

- 25-mm (1-mm gap size).
- 25-mm (2-mm gap size for bitumen rubber).

4. SAMPLE PREPARATION METHOD

4.1 Pour the hot RTFO aged binder into a silicon mould and leave to cool at room temperature.



Recommendation: A minimum mass of 0.6 g if testing will be conducted using a 25-mm plate. Testing should be completed within 4 hours from the time the sample is poured on the mould.

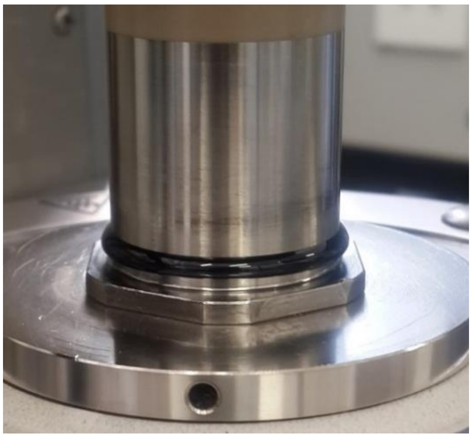
5. SAMPLE LOADING AND TRIMMING

5.1 Heat up the bottom plate to loading temperature.

Recommendation: A loading temperature of 70°C is recommended.

5.2 Take a wide and flat spatula and heat it up with a heating gun or hotplate. Use the heated spatula to warm up the upper measuring test plate by attaching the spatula to the upper measuring test plate while it is mounted to the instrument for approximately 10 sec.

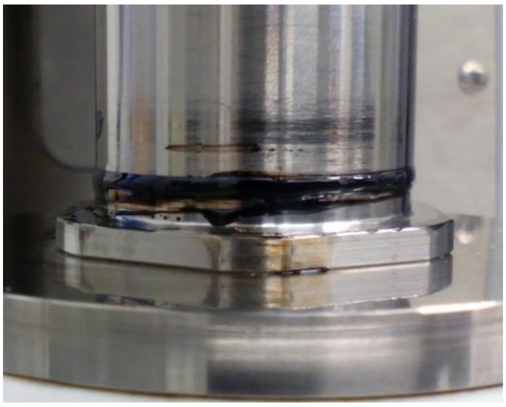
5.3 Demould the prepared sample and attach it to the upper plate and lower the upper measuring plate to trimming position (*gap between the plates that equals the testing gap plus the gap closure required to create a bulge*) – see picture below.



5.4 Heat up the trimming tool on the hot plate/heating gun (*it must be hot enough to be able to remove the sample*) and use it to trim the excess binder by moving the tool around the edges of the plates so that the binder is even with the outer diameter of the plates.

<u>NOTE 3</u>: Use short movements when trimming and remove small amounts of the sample at a time.

5.5 After trimming, lower the upper measuring test plate to the measuring gap position to form a slight bulge – see picture below.



6. SAMPLE LOADING AND TRIMMING

6.1 Condition the sample at the selected starting temperature for ten (10) minutes before testing starts (*unless thermal equilibrium analysis shows otherwise*).

6.2 Load the sample at a constant creep stress for duration of 1.0 s followed by a 9.0 s zero stress recovery. Record the stress and strain cycle at least every 0.1 s for the creep phase and at least every 0.45 s for the recovery cycle.

6.3 With no rest period in between cycles, perform creep and recovery cycles at 0.100 kPa. The first 10 cycles are for conditioning the sample and the second 10 cycles are for data collection

6.4 Allowing no rest period from task 6.3 and in between cycles, perform creep and recovery cycles at 3.200 kPa. Total time required to complete tasks 6.3 and 6.4 is 300 s.

6.5 For the last 10 cycles at 0.100 kPa and the 10 cycles at 3.200 kPa, record the following:

6.5.1 Initial strain value at the beginning of the creep portion of each cycle. This will be denoted as ε_o

6.5.2 The strain value at the end of the creep portion of each cycle. This will be denoted as ε_c.

6.5.3 The adjusted strain value at the end of the creep portion. This will be denoted as ε_1. ε_1 is calculated as: $\varepsilon_1 = \varepsilon_c - \varepsilon_o$

6.5.4 The strain value at the end of the recovery portion of each cycle. This will be denoted as $\varepsilon_{r.}$

6.5.5 The adjusted strain value at the end of the recovery portion of each cycle. This will be denoted $\varepsilon_{10.}$ ε_{10} is calculated as: $\varepsilon_{10} = \varepsilon_{r\,-}\varepsilon_o$

7. CALCULATIONS

7.1 Using the results obtained in 6.5. Determine the non-recoverable creep compliance (J_{nr}) for the binder at creep stress levels of 0.100 kPa and 3.200 kPa as follows:

7.1.1 For each of the last 10 cycles at creep stress of 0.100 kPa calculate the non-recoverable creep compliance, $J_{nr}(0.1, N)$, for $N = 1$ to 10:

$$J_{nr}(0.1, N) = \varepsilon_{10}/0.1$$

7.1.2 For each of the last 10 cycles at creep stress of 3.200 kPa calculate the non-recoverable creep compliance, $J_{nr}(3.2, N)$, for $N = 1$ to 10:

$$J_{nr}(3.2, N) = \varepsilon_{10}/3.2$$

7.1.3 Calculate the average non-recoverable creep compliance at 0.100 kPa:

$$J_{nr}\,0.1 = \text{SUM}\, J_{nr}(0.1, N)/10 \text{ for } N = 1 \text{ to } 10$$

7.1.4 Calculate the average non-recoverable creep compliance at 3.200 kPa:

$$J_{nr}\,3.2 = \text{SUM}\, J_{nr}(3.2, N)/10 \text{ for } N = 1 \text{ to } 10$$

W*here*: N represents the number of cycles

8. REPORT

8.1 Test temperature to the nearest 0.1°C.

8.2 Non-recoverable creep compliance (J_{nr}) at 0.1 kPa, to three significant figures.

8.3 Non-recoverable creep compliance (J_{nr}) at 3.2 kPa, to three significant figures.

9. CALIBRATIONS AND VERIFICATIONS

9.1 *Temperature verification:* follow manufacturer's recommendations and verify the temperatures (include 58°C, 64°C and 70°C) by using a rubber wafer or a dummy test specimen every ninety (90) days.

9.2 *Motor adjustment:* follow manufacturer's recommendations and do a motor adjustment with the measuring test plates at the correct gap sizes every ninety (90) days.

9.3 *External calibration:* done by the manufacturer yearly for properties such as temperature and torque.

10. PRECISION AND BIAS

10.1 Data for the determination of multi-laboratory precision for J_{nr} is presented in Table 2.

Table 2: Multi-Laboratory Precision

Condition	10-20pen		35-50pen		50-70pen		70-100pen	
	Coefficient of Variation (1s%)	Maximum Acceptable Range of Two Test Results (d2s%)	Coefficient of Variation (1s%)	Maximum Acceptable Range of Two Test Results (d2s%)	Coefficient of Variation (1s%)	Maximum Acceptable Range of Two Test Results (d2s%)	Coefficient of Variation (1s%)	Maximum Acceptable Range of Two Test Results (d2s%)
	Multi-laboratory Precision @58°C							
J_{nr} at 0.1 kPa	13.2	37.2	5.6-9.3	26.1	3.8-9.9	27.9	5.7-9.3	26.1
J_{nr} at 3.2 kPa	13.5	38.2	4.9-8.0	22.6	3.6-8.6	24.3	3.8-7.6	21.4
	Multi-laboratory Precision @64°C							
J_{nr} at 0.1 kPa	12.2	34.5	4.7-6.8	19.1	1.7-7.5	21.2	4.3-7.9	22.2
J_{nr} at 3.2 kPa	5.6	15.9	4.4-5.7	16.0	2.3-8.1	22.8	3.4-7.3	20.7

11. REFERENCES

11.1 ASTM D7405: Standard test method for multiple stress creep and recovery (MSCR) of asphalt binder using a dynamic shear rheometer. American Society for Testing and Materials (ASTM) International.

11.2 ASTM D7175: Standard test method for determining the rheological properties of asphalt binder using a dynamic shear rheometer. American Society for Testing and Materials (ASTM) International.

11.3 Technician's manual for specification of asphalt binders: MS-25 (3rd edition). Asphalt Institute, 2012.

11.4 O'Connell J, Mturi GA and Zoorob SE. A review of the development of the non-recoverable compliance, *Jnr*, for use in South Africa. CAPSA. 2015.

PGP5: ACCELERATED AGEING OF BITUMINOUS BINDER USING A PRESSURIZED AGING VESSEL (PAV)

1. INTRODUCTION

1.1 As part of the proposed SA bitumen PG grading testing, the first step in this process is to develop detailed test protocols to ensure consistent and reliable results throughout the industry. This protocol covers the accelerated aging of bituminous binder using a pressurized aging vessel (PAV).

1.2 The PAV will be performed in accordance with ASTM standard test method ASTM D6521. This protocol was developed based on the test method valid at the time of publication of this manual.

2. TESTING EQUIPMENT

2.1 ***PAV test apparatus:*** Any PAV equipment that fulfils the requirements of ASTM D6521. The PAV vessel should be designed to operate at 2.1 ± 0.1 MPa and at a temperature of 100 ± 10°C.

2.2 ***Pressure controller:*** A pressure regulating system that can control and maintain the pressure inside the vessel to ± 0.02 MPa

2.3 ***Temperature controller:*** A temperature regulating system that can control and maintain the temperature inside the vessel to ± 0.5°C.

2.4 ***Vacuum degassing oven:*** Any degassing system that fulfils the requirement of ASTM D6521 and is capable of maintaining temperature up to 180°C ± 5°C and absolute pressure of 15 ± 1.0 kPa.

2.5 ***Balance:*** A balance to determine the mass of the binder readable to 0.1 g.

2.6 ***Oven:*** An oven that can maintain any temperature up to 168°C to ± 5°C.

2.7 ***Stainless steel pans:*** Cylindrical pans, 140 ± 1 mm in inside diameter, 9.5 ± 1.5 mm deep and 0.6 mm metal thickness.

2.8 ***Scraping tool:*** A spatula or similar tool to scrape the binder out of the pans.

2.9 ***Container for PAV residue:*** A container of sufficient size such that when filled with PAV residue the depth of the binder is between 15 and 40 mm.

3. PREPARATION OF EQUIPMENT

3.1 Use a spirit level to check the levelness of the sample rack and the PAV instrument.

3.2 Open the pressure vessel lid by removing all bolts except one using an appropriate tool. The last bolt should be loosened enough, so it acts as a pivot, allowing the lid to swing open and rest on the lid support block if the equipment has it.

3.3 Insert the empty pan holder into the pressure vessel and close with the lid by finger-tightening the bolts in a staggered sequence.

3.4 Switch the instrument ON.

3.5 Follow manufacturer's instructions and select test temperature of 100°C.

3.6 Follow manufacturer's instructions for pre-heating the pressure vessel. Check that the temperature reaches 100°C ± 0.5°C.

3.7 Check the air pressure supply before testing to make sure you have sufficient air to pressurize the vessel to 2.1 ± 0.1 MPa for the remainder of the test period. Low air pressure will result in the test being declared invalid.

<u>NOTE 1</u>: Do not pressurize the vessel as yet at this stage.

4. SAMPLE PREPARATION

4.1 Testing should be done on RTFO aged binders. After RTFO ageing samples should immediately be prepared for PAV aging.

4.2 Pour the hot binder from the RTFO bottles (that will be used for PAV ageing) into one single preheated container and stir the binder well to ensure homogeneity.

4.3 PAV ageing should be done straight after the RTFO test without allowing samples to cool down.

5. TEST PROCEDURE

5.1 Place the stainless steel pan on a balance and weigh 50 ± 0.5 g mass of RTFO binder to the pans. Multiple pans can be used for one sample. Spread it out as evenly as possible (*this will yield approximately a 3.2 mm-thick film of binder*).

5.2 Remove the pan holder out of the pressure vessel, and insert the filled stainless steel pan in the pan holder. Fill the unused slots on the pan holder with empty pans.

CAUTION: The pressure vessel and the specimen holder may be very hot. Use protective clothing, heat resistant gloves and protective eyewear to prevent burns.

5.3 Reinsert the pan holder into the pressure vessel and close the vessel lid and cover.

5.4 Follow the manufacturer's instructions to start the test. Apply a pressure of 2.1 ± 0.1 MPa and time the aging process.

<u>NOTE 2</u>: If the temperature or pressure has not reached the desired targets within two hours of loading the pan holder, discontinue the procedure and discard the binder samples.

5.5 Maintain the temperature and air pressure inside the pressure vessel for 20h ± 10 min.

***NOTE 3**: If the temperature recording device shows that the temperature changed by more than ± 0.5°C for more than a total of one (1) hour during the ageing process (cumulative), declare the ageing process invalid and discard the sample.*

***NOTE 4:** If the pressure at the end of the PAV test is outside the 2.10 ± 0.1 MPa range, declare the ageing process invalid and discard the sample.*

5.6 Once the test is complete, slowly release the internal pressure of the PAV. This should be done within 15 minutes.

WARNING: Do not open the pressure vessel lid until the pressure is at least 0.15 MPa or lower. Allow adequate time to release all the pressure from the vessel before opening the oven lid and the pressure vessel lid. Any unreleased pressure can cause equipment damage and possible personal injury.

5.7 Open the vessel cover and lid and remove the pan holder from the pressure vessel. Use the putty-knife to remove the sample filled pan(s).

5.8 Place the sample filled pan(s) in an oven set at 168 ± 5°C for 15 ± 1 minute. Stir the sample gently to assist in the removal of air bubbles. **CAUTION: The pressure vessel and the specimen holder may be very hot. Use protective gloves to prevent burns.**

5.9 If the binder is aged in multiple pans, decant and combine the multiple pans into a single container and stir to homogenise the binder.

5.10 **Vacuum Degassing oven –** Follow the manufacturer's instructions to preheat the degassing oven until it is stable at 170 ± 5°C. Follow instruction 5.9 above. Select a container that will allow the sample to be between 15 to 40 mm in depth.

5.11 Put the container in the degassing oven and start the degassing process.

5.12 Maintain the pressure at 15 ± 2.5 kPa absolute for 30 ± 1 min. Release the vacuum and remove the container

5.13 If tests to determine the properties of the de-gassed, PAV aged specimen cannot be carried out immediately, cover and store the sample appropriately at a temperature between 5°C and 15°C for future testing. **CAUTION: The degassing oven and the sample container will be very hot. Use protective gloves to prevent burns.**

6. CALIBRATIONS

6.1 Temperature sensors should be verified every six months to within ± 0.5°C of a calibrated thermometer.

6.2 Pressure gauge should be verified every six months to within ± 0.02 MPa of a calibrated pressure gauge.

7. REFERENCES

7.1 ASTM D6521: Accelerated aging of asphalt binder using a pressurized aging vessel (PAV). American Society for Testing and Materials (ASTM) International.

7.2 Technician's manual for specification of asphalt binders: MS-25 (3rd edition). Asphalt Institute, 2012.

PGP6: STORAGE STABILITY

1. INTRODUCTION

1.1 As part of the proposed SA Bitumen PG grading testing, the first step in this process is to develop detailed test protocols to ensure consistent and reliable results throughout the industry. This protocol covers the storage stability method.

1.2 The storage stability will be performed in accordance with TG1: Method MB-6 and ASTM D7175. This protocol was developed based on the test method valid at the time of publication of this manual.

2. TESTING EQUIPMENT

2.1 ***Dynamic Shear Rheometer (DSR):*** Any DSR that fulfils the requirements of ASTM D7175.

2.2 ***Test Plates:*** Cylindrical stainless steel metal lower and upper measuring plates of 25 ± 0.05 mm in diameter with a smooth ground surface.

2.3 ***Trimming tool:*** A tool suitable to trim excess binder from the measuring test plates.

2.4 ***Specimen mould:*** Silicon rubber mould for sample preparation of the DSR specimens.

2.5 ***Materials:***

- *Wiping material:* paper towel or clean cloth to wipe the measuring test plates.
- *Cleaning solvent:* solvent to clean the measuring test plate. After cleaning, the plate should have no residue.

NOTE 1: Acetone can be used to remove any residue left from the initial cleaning.

2.6 ***Oven:*** An oven that can maintain any temperature up to 260°C ±3°C and can also maintain 180°C ±2°C.

2.7 ***Hot plate/heating gun:*** A heating equipment to warm up the trimming tool.

2.8 ***Glass tubes:*** An open ended 300 mm long glass tube with an internal diameter of 12.5 mm.

2.9 **Cork:** A cork with a v-notch cut along its length (*capable of withstanding temperatures of 185°C*) to seal a 12.5 mm diameter tube.

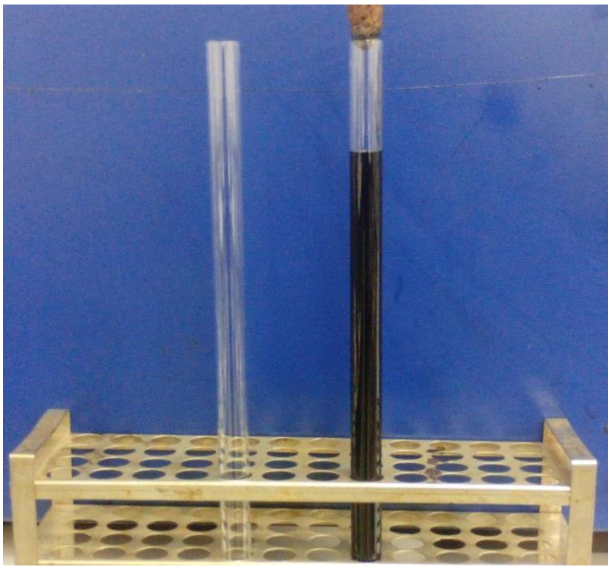
2.10 ***Stand:*** A stand that will hold the glass tubes vertically in the oven.

2.11 ***Glass cutter:*** To cut the top and bottom of the glass tube.

3. SAMPLE PREPARATION METHOD (STORAGE STABILITY)

3.1 Prepare the sample according to the sample preparation protocol.

3.2 Pour the heated sample into the pre-heated glass tube (pre-heated in the oven at 180°C for 30 minutes) to about 10 mm from the top and cover with a v-notch cut cork (*see picture below*). The v-notch cork acts as a breather.



3.3 Place the glass tube in the oven at 180 ± 2°C in a vertical position and leave for three days.

3.4 After three days, remove from oven and leave the glass tube at room temperature to cool.

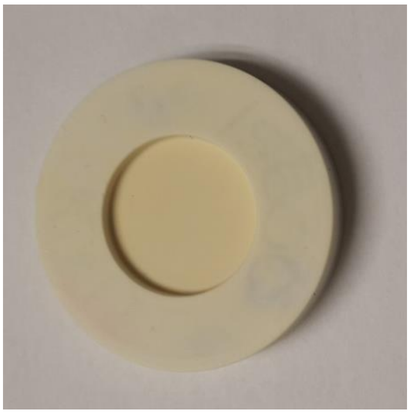
3.5 Cool the glass tube to about 0°C to be able to cut the tube transversely into three equal sections.

3.6 Prepare the top and bottom section as per sample preparation in 4 below.

4. SAMPLE PREPARATION METHOD (DSR)

4.1 Heat the top and bottom sample from the storage stability separately until they are sufficiently fluid to pour. Stir it well.

4.2 Pour the hot binder prepared above into a silicon mould and leave to cool at room temperature.

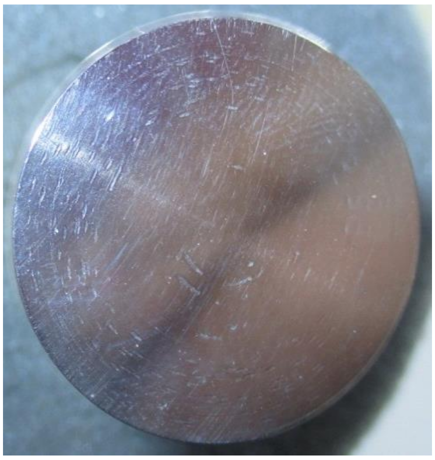


Recommendation: A minimum mass of 0.6 g if testing will be conducted using a 25-mm plate Testing should be completed within 4 hours from the time the sample is poured on the mould.

5. PREPARATION OF INSTRUMENT (DSR)

5.1 Follow manufacturer's recommendations to prepare the instrument.

5.2 Inspect the measuring test plates to ensure they have no scratches and/or dents.



BAD

GOOD

5.3 Use a solvent to clean the measuring test plates.

5.4 Follow manufacturer's instruction and select the correct workbook for testing the following parameters:

- *G*/sinδ*

5.5 Mount the upper measuring test plate to the instrument ensuring that it is aligned to the instrument.

5.6 Attach the lower measuring test plate to the instrument. Firmly tighten it.

5.7 Select the testing temperature to be used according to the SA specification. See Table 1 below:

Table 1: Maximum Pavement Design Temperatures for South Africa

Maximum pavement design temperature (°C)		
58	64	70

5.8 Use the manufacturer's instruction to zero the gap at the selected temperature and correct gap size. If multiple temperatures are going to be used, zero gap at the middle temperature of the temperature range. Use the sample for all temperatures.

NOTE 2: Make sure that the correct gap size is selected for the 25-mm measuring plates:

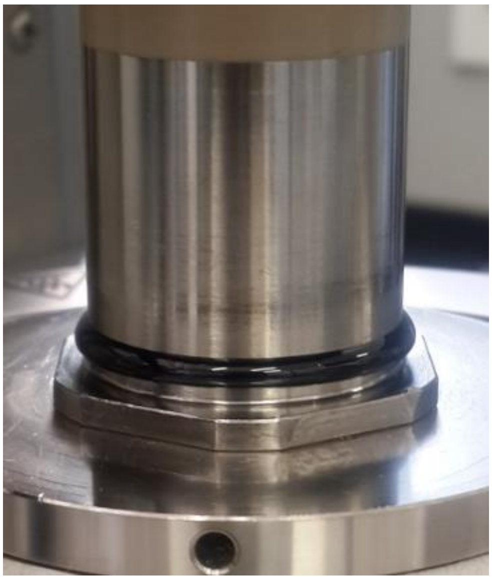
- 25-mm (1-mm gap size).
- 25-mm (2-mm gap size for bitumen rubber).

6. SAMPLE LOADING AND TRIMMING

6.1 Heat up the bottom measuring plate to loading temperature.
Recommendation: A loading temperature of 70°C is recommended.

6.2 Take a wide and flat spatula and heat it up with a heating gun or hotplate. Use the heated spatula to warm up the upper measuring test plate by attaching the spatula to the upper measuring test plate while it is mounted to the instrument.

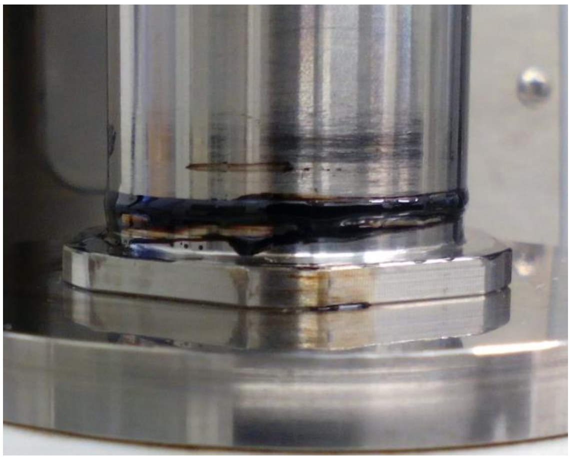
6.3 Demould the prepared sample and attach it to the upper measuring test plate and lower the upper measuring test plate to trimming position (*gap between the plates that equals the testing gap plus the gap closure required to create a bulge*) –see picture below.



6.4 Heat up the trimming tool on the hot plate/heating gun (*it must be hot enough to be able to remove the sample*) and use it to trim the excess binder by moving the tool around the edges of the plates so that the binder is even with the outer diameter of the plates.

NOTE 3: ***Use short movements when trimming and remove small amounts of sample at a time.***

6.5 After trimming, lower the upper measuring test plate to the measuring gap position to form a slight bulge – see picture below.



7. TESTING PROCEDURE

7.1 Test the G*/sinδ of the samples prepared in 6.5 above.

7.2 When testing at multiple temperatures, start testing from the lowest temperature, using the same sample.

7.3 Condition the sample at the selected starting temperature for ten (10) minutes before testing starts (*unless thermal equilibrium analysis shows otherwise*).

7.4 Test at a standard frequency of 10 rad/s and target LVE strain value.

7.5 Obtain measurements of complex modulus (G*).

8. CALCULATIONS

8.1 Storage stability: G* will be measured at T_{max} of both top and bottom sample. T_{max} is the design maximum pavement temperature – see Table 1.

- $[G^*_{HIGH} - G^*_{LOW}] / G^*_{HIGH}$

NOTE 4: ***The storage stability is determined from the top and bottom of the glass tube. G* (measured at T_{max}) from either may not always be the highest, therefore the storage stability is calculated as the difference in G* (highest minus lowest) divided by the highest G* value.***

9. REPORT

9.1 Storage stability: % difference in G* at T_{max} to one decimal place.

10. DSR CALIBRATIONS AND VERIFICATIONS

10.1 *Temperature verification:* follow manufacturers' recommendations and verify the temperatures (include 58°C, 64°C and 70°C) by using a rubber wafer or a dummy test specimen every ninety (90) days.

10.2 *Motor adjustment:* follow manufacturers' recommendations and do a motor adjustment with the measuring spindles at the correct gap sizes every ninety (90) days.

10.3 *External calibration:* done by the manufacturer yearly for properties such as temperature and torque

11. REFERENCES

11.1 ASTM D7175: Determining the rheological properties of asphalt binder using a dynamic shear rheometer.

11.2 Technical guideline (TG1) July 2015: The use of modified bituminous binder in road construction (3rd edition).

11.3 Technician's manual for specification testing of asphalt binders: MS-25 (3rd edition), 2012.

PGP7: VISCOSITY DETERMINATION OF A BITUMINOUS BINDER AT ELEVATED TEMPERATURES USING A ROTATIONAL VISCOMETER

1. INTRODUCTION

1.1 As part of the proposed SA bitumen PG grading testing, the first step in this process is to develop detailed test protocols to ensure consistent and reliable results throughout the industry. This protocol covers the viscosity determination of bituminous binders at elevated temperatures using a rotational viscometer. This excludes filled bituminous materials.

1.2 The viscosity will be performed in accordance with ASTM standard test method ASTM D4402. This protocol was developed based on the test method valid at the time of publication of this manual.

2. TESTING EQUIPMENT

2.1 ***Rotational viscometer:*** Any rotational viscosity equipment that fulfils the requirements of ASTM D4402

2.2 ***Temperature controller***: A temperature controller that can maintain sample temperatures to ± 2°C for temperatures between 150 and 260°C. This forms part of the equipment.

2.3 ***Sample chamber heater:*** The chamber with a cover for maintaining the binder sample at test temperature.

2.4 ***Oven:*** An oven that can maintain any temperature up to 260°C ± 3°C.

2.5 ***Balance:*** A balance to determine the mass of the binder readable to 0.1 g.

2.6 ***Measuring geometry:*** A measuring geometry that will attain the required equipment torque specification. For Brookfield Viscometers, use spindle S-21 or spindle S-27.

2.7 ***Sample chambers:*** Reusable or disposable chambers.

3. PREPARATION OF APPARATUS

3.1 Make sure that the viscometer and chamber heater are levelled as recommended by the instrument manufacturer.

3.2 Follow manufacturer's instructions and zero the viscometer before use or as needed.

4. TEST PROCEDURE

4.1 Switch the instrument ON by following the manufacturer's operating instructions.

4.2 Warm the instrument for at least five minutes before you start.

4.3 Set the temperature controller to desired test temperature (*taking into account the relevant correction factors*).

4.4 Select the appropriate measuring geometry that will give you a torque between 10 and 98 % while testing.

***NOTE 1**: In the event that the binder type is unknown, select the measuring geometry by trial and error.*

***NOTE 2**: Use the speed (RPM) or shear rate (sec^{-1}) that will give you the highest torque. The higher the torque, the more accurate the measurements will be.*

4.5 Warm up the sample holder and the measuring geometry at the testing temperature of 165°C for at least 15 min in the viscometer.

4.6 Add the volume of sample appropriate for the selected measuring geometry and sample chamber according to the manufacturer's manual.

***NOTE 3**: A convenient way of measuring the volume is by weighing out the amount calculated from appropriate density data for the sample.*

4.7 Insert the preheated measuring geometry into the sample holder and attach it to the viscometer

4.8 Leave the sample to reach desired temperature within 30 min. Once it reaches temperature, leave it to equilibrate for a minimum of 10 min before starting the rotation.

4.9 Start the rotation of the viscometer at a speed (RPM) that will give you a torque between 10 and 98 %. Maintain this speed for an additional 5 min. Temperature should not change by more than ± 1.0°C during this conditioning period. For Brookfield Viscometers:

- Spindle S-21 – speed has to exceed 32 RPM (bottom left of screen in figure below), while remaining within the torque requirements of 10 – 98 % of maximum torque (*bottom right of screen in the figure below*).
- Spindle S-27 – speed has to exceed 88 RPM, while remaining within the torque requirements of 10 – 98 % of maximum torque.

4.10 After conditioning, measure the torque and the viscosity at 1 min interval for a minimum of three minutes or until the reading is stable.

***NOTE 4**: Use a fresh sample for each new test.*

***NOTE 5**: Decrease the speed (RPM) if the torque reading is above 98 %.*

5. CALCULATIONS

5.1 If the viscometer displays the viscosity readings in centipoise (cP), multiply the viscosity reading by 0.001 to convert to Pascal seconds (Pa.s).

5.2 Calculate the average of the three readings.

6. REPORT

6.1 Report the test temperature, °C.

6.2 Report the viscosity of the highest torque in pascal seconds (Pa.s) to the nearest 0.001.

6.3 In the case where a stable reading could not be achieved, report the viscosity range of the three readings.

6.4 Report the speed (RPM) or shear rate (sec^{-1}), torque (%) and state the measuring geometry.

7. CALIBRATIONS AND VERIFICATIONS

7.1 The unit shall be externally calibrated at least once a year for torque, speed and temperature (*including 165°C*).

7.2 Use a reference sample of known viscosity every three (3) months to verify the accuracy of the viscometer at various temperatures. The measured viscosity should be within the ±2% of the certified value. Make sure there is an overlap between verification temperatures and calibrated temperatures.

NOTE 6: The frequency of verification can be reduced with enough data.

8. PRECISION AND BIAS

8.1 Data for the determination of single- and multiple-operator precision performed at the CSIR is presented in Table 1.

Table 1: Single and Multiple Operator Precision

Material Properties	**Modified Bitumen (SBS) (165ºC)**
Operator 1	
Result #1	0.391
Result #2	0.387
Result #3	0.382
Result #4	0.385
Result #5	0.383
Average for Operator 1	**0.386**
Standard deviation for Operator 1	**0.004**
Coefficient of Variation for Operator 1	**0.93%**
Operator 2	
Result #1	0.374
Result #2	0.377
Result #3	0.374

Result #4	0.382
Result #5	0.372
Average for Operator 2	**0.376**
Standard deviation for Operator 2	**0.004**
Coefficient of Variation for Operator 2	**1.0%**
Multi-operator Average	**0.381**
Multi-operator Standard deviation	**0.006**
Multi-operator Coefficient of Variation	**1.6%**

9. REFERENCES

9.1 ASTM D4402: Viscosity determination of asphalt at elevated temperatures using a rotational viscometer. American Society for Testing and Materials (ASTM) International.

9.2 Technician’s manual for specification of asphalt binders: MS-25 (3rd edition). Asphalt Institute, 2012.

PGP8: THE EFFECT OF HEAT AND AIR ON A MOVING FILM OF ASPHALT: ROLLING THIN-FILM OVEN TEST (RTFOT)

1. INTRODUCTION

1.1 As part of the proposed SA bitumen PG grading testing, the first step in this process is to develop detailed test protocols to ensure consistent and reliable results throughout the industry. This protocol covers the effect of heat and air on a moving film of asphalt: Rolling Thin-Film Oven Test (RTFOT).

1.2 The RTFOT will be performed in accordance with ASTM D2872 and Sabita manual TG1: Method MB-3.

2. TESTING EQUIPMENT

2.1 ***RTFO test apparatus:*** Any RTFO equipment that fulfils the requirements of ASTM D2872.

2.2 ***Flowmeter***: Any flowmeter type capable of accurately measuring the airflow at a rate of 4000 ± 200 mℓ/min.

2.3 ***Thermometer***: A thermometer that fulfil the requirements of ASTM D2872.

2.4 ***Sample containers***:

1. A heat-resistant glass container that is clear, transparent and has the following dimensions:

   - Height: 139.7 ± 1.5 mm
   - Thickness: 2.4 ± 0.3 mm
   - Width: 64 ± 1.2 mm
   - Opening size: 31.75 ± 1.5 mm

2. Brass metal bottles and rollers that have the following dimensions:
   - Height: 140 ± 1 mm
   - Thickness: 1.6 ± mm
   - Width: 63 ± 1 mm
   - Opening size 12 ± 0.5 mm
     - Roller length including ends: 138 ± 1 mm
     - Roller must have a 90° left hand twist from end of roller

2.5 ***Balance***: An analytical balance to determine the mass of the binder readable to 0.001 g.

2.6 ***Oven***: An oven that can maintain any temperature up to 180 ± 5°C.

2.7 ***Cooling rack***: A wire or metal rack that will allow the sample containers to cool in a horizontal position with each container in the same horizontal plane.

2.8 ***Spirit level***: To check the levelness of the RTFO instrument.

3. PREPARATION OF APPARATUS

3.1 Use a spirit level to check the levelness of the RTFO instrument.

3.2 Preheat the oven at 163 ± 0.5°C for a minimum of 4 hours prior to testing

***NOTE 1**: The previous edition of the ASTM D2872 test method required that the oven be preheated for 16 hours.*

4. TEST PROCEDURE A (if the sample is an unmodified binder)

4.1 Heat the sample for the minimum necessary time to ensure that the sample is completely fluid. Manually stir the sample to ensure homogeneity but avoid incorporating air bubbles.

4.2 Individually weigh the empty glass containers to the nearest 0.001 g.

4.3 Pour 35 ± 0.5 g of the sample into the required glass containers.

4.4 Immediately after pouring the sample, turn the container to a horizontal position. Rotate the container slowly for at least one full rotation to pre-coat the surface. Take care that the sample does not flow out during this step.

4.5 Place the container horizontally in a clean cooling rack at room temperature away from any source of heat.

4.6 Leave the sample to cool in the cooling rack for a minimum of 60 minutes and a maximum of 180 minutes.

4.7 Use 2 marked separate containers for mass change determination. After cooling, determine the mass of these containers separately to the nearest 0.00 g.

4.8 Place the filled containers in the pre-heated rolling thin film oven. Arrange the containers and ensure that the rotating carousel is balanced. Fill the unused spaces with empty containers.

4.9 With re oven set at the operating temperature of 163 ± 0.5°C and the airflow set at 4000 ± 200 mℓ/min, close the door and rotate the carriage at a rate of 15 ± 0.2 r/min for 85 minutes.

4.10 If the oven does not reach the test temperature of 163 ± 0.5°C within 10 min, discontinue the test.

4.11 At the end of the test, use clean heat resistant gloves to remove the containers for the mass change determination and place them horizontally in the cooling rack. Allow the containers to cool at room temperature for a minimum of 60 minutes and a maximum of 180 minutes.

4.12 Remove the remaining sample containers one at a time and transfer the binder into one container and stir to homogenize the sample. Test the aged binder within 72 hours of performing the RTFO test.

4.13 Individually weigh the mass change containers to the nearest 0.001 g.

5. TEST PROCEDURE B (IF THE SAMPLE IS A MODIFIED BINDER)

5.1 If the sample is known to be a modified binder, follow Sabita manual TG1: MB-1 and MB-2 sample preparation methods.

5.2 Individually weigh the empty metal containers with the rollers inside to the nearest 0.001 g.

5.3 Pour 40 ± 0.5 g of the sample into the required metal containers with rollers inside – see below.

5.4 Immediately after pouring the sample, close the container and turn the container to a horizontal position. Rotate the container slowly for a least one full rotation to pre-coat the surface. Take care that the sample does not flow out during this step.

5.5 Place the container horizontally in a clean cooling rack at room temperature away from any source of heat.

5.6 Leave the sample to cool in the cooling rack for a minimum of 60 minutes and a maximum of 180 minutes.

5.7 Use two marked separate containers for mass change determination. After cooling, determine the mass of these containers separately to the nearest 0.001 g.

5.8 Place the filled containers in the pre-heated RTFO. Arrange the containers and ensure that the rotating carousel is balanced. Fill the unused spaces with empty containers.

5.9 With the oven set at the operating temperature of 163 ± 0.5°C and **NO** air flowing, close the door and leave the samples for 30 minutes in the oven.

5.10 If the oven does not reach the test temperature of 163 ± 0.5°C within 10 minutes, discontinue the test.

5.11 After 30 minutes, with the airflow set at 4000 ± 200 mℓ/min, rotate the carriage at a rate of 15 ± 0.2 r/min for 60 minutes ± 10 seconds.

5.12 At the end of the test, use clean heat resistant gloves to remove the containers for the mass change determination and place them horizontally in the cooling rack. Allow the containers to cool at room temperature for a minimum of 60 minutes and a maximum of 180 minutes.

5.13 Remove the remaining sample containers one at a time and transfer the binder into one collection container and stir to homogenize the sample. Test the aged binder within 72 hours of performing the RTFO test.

5.14 Individually weigh the mass change containers to the nearest 0.001 g.

6. REPORT

6.1 Report the average mass change of the binder in the two containers as a mass percentage of the original to the nearest 0.0001%.

6.2 Comment on the sample behaviour during the test in terms of its flow characteristics: i.e. did the sample leak out of the container? Does the sample have a distinct smell? Or any other relevant comment.

7. CALIBRATIONS AND VERIFICATIONS

7.1 Calibrate the flow meter at least once a year.

7.2 Verify the oven thermometer with a calibrated thermometer at least once a year.

7.3 Verify the carriage rate with a calibrated stop watch at least once a year.

8. PRECISION AND BIAS

8.1 Data for the determination of single and multiple operator precision for the percentage mass change is given in Table 1 below.

Table 1: Single and Multiple Operator Precision

Material Properties	**% Mass Change**
Operator 1	
Result #1	0.083
Result #2	0.074
Average for Operator 1	**0.079**
Standard Deviation for Operator 1	**0.006**
Coefficient of Variation for Operator 1	**8.1**
Operator 2	
Result #1	0.074
Result #2	0.080
Average for Operator 2	**0.077**
Standard Deviation for Operator 2	**0.004**
Coefficient of Variation for Operator 2	**5.5**
Multi-operator Average	**0.078**
Multi-operator Standard Deviation	**0.005**
Multi-operator Coefficient of Variation	**5.8**

9. REFERENCES

9.1 ASTM D2872 (2012): Effect of heat and air on a moving film of asphalt (Rolling Thin-Film Oven Test). American Society for Testing and Materials (ASTM) International.

9.2 Technician's manual for specification of asphalt binders: MS-25 (3rd edition). Asphalt Institute, 2012.

9.3 Sabita Technical Guideline 1: The use of modified bituminous binder in road construction – 5th edition, November 2020.

PGP9: THE CHEMICAL ANALYSIS OF BITUMINOUS BINDER USING THIN LAYER CHROMATOGRAPHY (TLC)

1. INTRODUCTION

1.1 This protocol covers the Saturates, Aromatics, Resins and Asphaltenes (SARA) analysis of bituminous binders.

1.2 The chemical analysis will be performed in accordance with IP standard test method IP 469/01.

2. TESTING EQUIPMENT

2.1 ***TLC/FID analyser***: Thin Layer Chromatography/Flame Ionization Detection analyser equipment that fulfils the requirements of IP 469/01 test method.

2.2 ***Coated quartz chromarod***: A thin layer quartz rod 0.9 mm in diameter and 152 mm in length specifically designed for TLC/FID analysis.

2.3 ***Chromarod frame/holder***: A specifically designed numbered and graduated chromarod frame to hold up to 10 think layer chromarods.

2.4 ***Developing tanks***: A minimum of three tanks made of plate glass of 180 mm height x 155 mm width x 35 mm depth with a removable glass cover.

2.5 ***Drying chamber***: A thermostatically-controlled drying chamber or oven capable of maintaining a temperature of 60 ± 10°C and can accommodate the chromarod frame.

2.6 ***Balance***: An analytical balance to determine the mass of the binder readable to 0.001 g.

2.7 ***Oven***: An oven that can be controlled over the temperature range of 40°C to 150°C.

2.8 ***Filter paper***: General purpose to cover the bottom and sides of the developing tanks.

2.9 ***Sample applicator***: A syringe or disposable micropipette capable of applying 1 µl test sample manually to each chromarod, or a semi-automatic autospotter.

2.10 ***Storage chamber:*** A covered glass or plastic tank for the storage of the chromarods.

2.11 ***Volumetric glassware:*** Vials and measuring cylinders of 10 mℓ capacity.

2.12 ***Integrator/data system:*** The data system that is compatible with the analyser and is capable of peak area integration and has a minimum sampling rate of 5 Hz.

2.13 ***Ultrasonic bath:*** Optional.

2.14 ***Rinsing solvent:*** i.e. Acetone.

3. REAGENTS AND MATERIALS

3.1 All liquid reagents shall be of analytical reagent grade or higher purity. The following solvents are required: toluene, heptane, dichloromethane and methanol.

3.2 Gasses shall be of purity suitable for use with FID's. The following gasses are required: hydrogen and air.

Recommended: Air zero – instrument grade (IG) to be used.

4. DEVELOPING TANKS PREPARATION

4.1 Fill Tank A with heptane to a depth of 12.5 ± 2.5 mm. Make sure that the entire filter paper is saturated with solvent. Replace the glass cover and allow the tank to stand for 30 ± 1 minute before use.

4.2 Fill Tank B with 80:20 toluene:heptane by volume to a depth of 12.5 ± 2.5 mm. Make sure that the entire filter paper is saturated with solvent. Replace the glass cover and allow the tank to stand for 30 ± 1 minute before use.

4.3 Fill Tank C with 95:05 dichloromethane:methanol by volume to a depth of 12.5 ± 2.5 mm. Make sure that the entire filter paper is saturated with solvent. Replace the glass cover and allow the tank to stand 30 ± 1 minute before use.

***NOTE 1**: The development solvent shall be newly prepared every day since the composition of prepared development solvent will be gradually varied by its evaporation as time goes on.*

5. PREPARATION OF EQUIPMENT

5.1 Set up the TLC/FID analyser as per manufacture’s instruction and adjust the hydrogen supply pressure to the recommended values.

5.2 Switch on the TLC/FID analyser instrument and let it stand for a minimum of 10 minutes.

5.3 Turn on the hydrogen valve at the back of the instrument.

5.4 At the front of the instrument, increase airflow to 2.0 mℓ/min and increase hydrogen flow to 160 mℓ/minute.

5.5 Switch on the flame but DO NOT touch any part of the instrument with the igniter.

5.6 Connect the analog box.

5.7 Turn on the computer and analysis software.

5.8 Load a suitable method to identify and integrate the four peaks due to saturates, aromatic, resins and asphaltenes.

6. SAMPLE PREPARATION (TO BE PERFORMED IN A TEMPERATURE CONTROLLED ROOM)

6.1 Make a TLC sample of 200 ± 10 mg bitumen in a 10 ± 0.1 mℓ volumetric flask, filled up to the mark with dichloromethane.

6.2 Activate chromarods by blank scanning at least twice through the FID flame before use each day. This should be done immediately before sample injection.

Blank scanning process:

- Select “BLANK SCAN” on the instrument.
- Select the number of “Blank Scan” by keying in the following manner:

BLANK SCAN – NUMERIC KEY(S) 1 TO 99 – ENTRY KEY

- For example, if the display indicates: “30 BLK 2 1 – 10”, it means the following procedure can be performed: The scanning speed is 30 second per scan, rod numbers 1 through 10 will be scanned for 2 times repeatedly.

***NOTE 2:** New chromarods should be taken through the solvent development procedure (refer to 6.8 to 6.19) before FID scans. For new chromarods, leave the chromarods to travel to the 100 mm graduated mark in all three tanks.*

6.3 Rinse the sample applicator with TLC sample.

6.4 Carefully inject 1 µl of sample onto a chromarod on the origin point of the chromarod (i.e. zero point graduation of the road holder). Make sure that the tip of the sample applicator is in contact with the chromarod. Do not exert any pressure on the chromarod because it is fragile and could break. See the figures below.

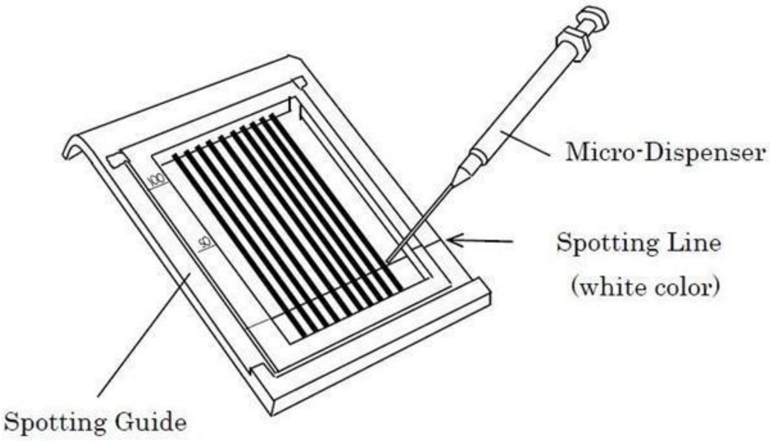


Figure 1: Sample spotting by hand

Figure 2: Sample spotting by Auto spotter

***NOTE 3:** Each sample should be analysed on at least 3 chromarods.*

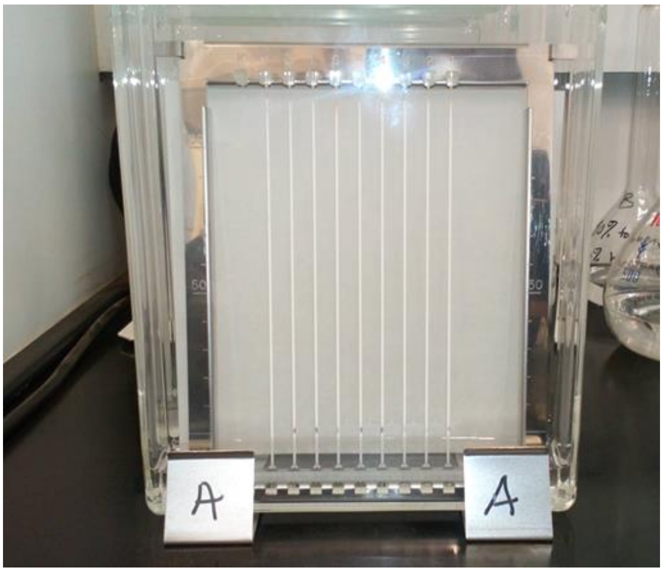
6.5 Dry the chromarods in a drying chamber set at 70°C for 5 ± 1 minute and rinse the needle in a solvent (i.e. acetone).

6.6 Place the chromarods in a clean, covered storage chamber and allow to cool to room temperature for exactly 10 ± 1 minute.

6.7 Make sure the solvent in tank A, B and C is between 12.5 ± 2.5 mm.

6.8 Place the chromarods in tank A – see below.

6.8 Place the chromarods in tank A – see below.



6.9 Leave the chromarods **undisturbed** until the solvent has travelled to 100 ± 5 mm graduation of the rod holder.

6.10 Dry the chromarods in a drying chamber for 5 ± 1 minute.

6.11 Place the chromarods in a clean, covered storage chamber and allow cooling to room temperature for 10 ± 1 minute.

6.12 Place the chromarods in tank B.

6.13 Leave the chromarods **undisturbed** until the solvent has travelled to 50 ± 5 mm graduation of the rod holder.

6.14 Dry the chromarods in a drying chamber for exactly 5 ± 1 minute.

6.15 Place the chromarods in a clean, covered storage chamber and allow cooling to room temperature for 10 ± 1 minute.

6.16 Place the chromarods in tank C.

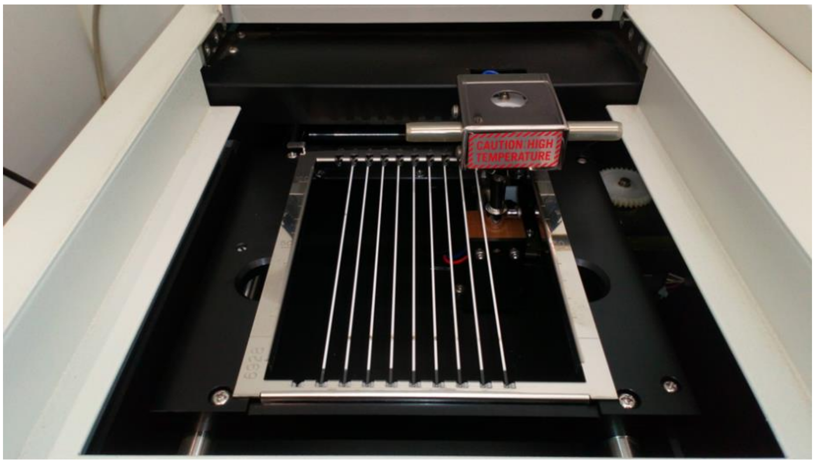
6.17 Leave the chromarods **undisturbed** until the solvent has travelled to 20 ± 2 mm graduation of the rod holder.

6.18 Dry the chromarods in a drying chamber for 5 ± 1 minute.

6.19 Place the chromarods in a clean, covered storage chamber and allow to cool to room temperature for 10 ± 1 minute.

7. TEST PROCEDURE

7.1 Place the frame with the chromarods in the TLC/FID analyser for scanning preparation – see picture below.



NOTE 4: Following measurement conditions shall be applied for FID single measurement.

· Hydrogen 160mL/min， Air 2.0L/min， Scan Speed 30sec/scan.

7.2 Choose 'Normal Scan' at 30 sec scan speed.

7.3 Choose the chromarods number to be tested (this is the slot number on the frame of the chromarod to be analysed).

7.4 Press AUX SGN (this allows the TLC/FID analyser to synchronise with the computer).

7.5 Press AUTO ZERO (this baseline corrects the signal).

7.6 Press START.

7.7 Run 9 "Blank Scans" at the end of the test to ensure the baseline is flat (repeat scanning if necessary, i.e. when the line is not flat).

7.8 Store the clean chromarods in a clean, covered storage chamber for future use.

NOTE 5: Never store chromarods in a vessel containing drying agent.

8. WASHING OF CHROMARODS

8.1 The expected number of repeated use of chromarod is approx. 20 times. However, in case of selective measurement of P by FPD of Iatroscan MK-6, the pre-treatment and washing of chromarods are required. See washing procedure below:

8.2 Most organic compounds can be eliminated by the "Blank Scan". Execute the "Origin Scan" several times to remove the organic compound(s) remained on the origin point.

8.3 In case that the organic compounds are remaining on the whole of the chromarod, conduct the Blank Scan twice at the scanning speed of 60 seconds/scan.

8.4 If the organic residuals cannot be removed by the scanning, wash the chromarods with concentrated sulphuric acid in the manner described below.

NOTE: 6: ***The separation pattern to be obtained after the cleaning will sometimes be different from that of previous measurement as the development conditions may be, because the sulfuric acid used will possibly remain on the rod(s) even after finishing this cleaning.***

8.5 Wash the chromarods lightly with deionized water.

8.6 Soak them in the concentrated sulfuric acid overnight.

8.7 Upon taking out the chromarods from the concentrated sulfuric acid, thoroughly rinse them with deionized water.

8.8 Remove the water by drying the chromarods for 1 hour at 120°C, or by conducting "Blank Scan" twice after drying them for 3 minutes at 120°C.

8.9 Keep the chromarods in a clean glass vessel with a lid to avoid their contamination caused by adsorption of organic/inorganic matter coming from the dust in the air.

NOTE 7: Only continue using these chromarods if you get the same chromatogram as before with a known sample. If not, replace the chromarod(s) with new one(s).

9. SWITCHING OFF

9.1 Turn OFF only the main switch of the hydrogen.

9.2 Wait for the Hydrogen to read zero (0) at the front of the instrument.

9.3 Then turn OFF the hydrogen regulator, then the hydrogen valve at the back of the instrument and the hydrogen flow valve at the front of the instrument (completely - until knob does not turn anymore).

9.4 Turn OFF the air flow completely.

9.5 Turn instrument OFF.

9.6 Exit the data system and turn computer OFF.

9.7 Wait until the computer is completely OFF then disconnect the Analog box.

10. CALCULATION

10.1 For each chromarod, normalize the four peak areas to give percent concentrations to the nearest 0.1% for the SARA components.

11. REPORT

11.1 Report the mean percent concentrations calculated in 10.1 of the SARA components to the nearest 0.1%.

12. PRECISION AND BIAS

12.1 Data for the determination of single operator precision for the SARA components is given in the Table below.

No of repeats	% Saturates	% Aromatics	% Resins	% Asphaltenes
1	2.78	41.39	31.87	23.95
2	2.92	39.74	34.26	23.07
3	3.01	37.80	34.46	24.73
Average	**2.90**	**39.64**	**33.53**	**23.92**
STDev	**0.12**	**1.80**	**1.44**	**0.83**
COV	**3.99**	**4.53**	**4.30**	**3.47**

13. REFERENCE

13.1 IP 469/01 (2006): Determination of saturated, aromatics and polar compounds in petroleum products by thin layer chromatography and flame ionization detection.

13.2 CSIR TLC work instruction: WI-CSIR-BE-LAB-20.70 (2014).

PROTOCOLS FOR ADVANCED TESTING OF ASPHALT IN SOUTH AFRICA

(ASP)

ASP1: THE COMPACTION OF ASPHALT SPECIMENS IN THE GYRATORY COMPACTOR

1. INTRODUCTION

This test protocol describes a procedure for the preparation and compaction of asphalt specimens for asphalt mix design including moisture sensitivity test, and performance property tests such as dynamic modulus, Hamburg wheel tracking, moisture damage, and permeability. This protocol was developed based on the test method valid at the time of publication of this manual.

2. NORMATIVE REFERENCES

- Sabita Manual 35/TRH8, Design and use of asphalt in road pavements.
- SANS 3001-AS10, Determination of bulk density and void content of compacted asphalt.
- SANS 3001-AS11, Determination of the maximum void-less density of asphalt mixes and the quantity of binder absorbed by the aggregate.
- ASTM D6752 / D6752M, Standard test method for bulk specific gravity and density of compacted asphalt mixtures using automatic vacuum sealing method.
- SANS 1649, Non-automatic self-indicating, semi-self-indicating and non-self-indicating weighing instruments with denominated verification scale intervals.
- TMH 5, Sampling methods for roads construction materials.
- TRH 21, Recycled Asphalt.
- AASHTO R 83, Standard practice for preparation of cylindrical performance test specimens using the Superpave gyratory compactor.
- AASHTO T 312, Preparing and determining the density of hot mix asphalt specimens by means of the Superpave gyratory compactor.
- AASHTO R 30, Mixture conditioning of hot mix asphalt (HMA).

***NOTE 1*: The latest versions of the standard test methods are to be used in this protocol**.

3. DEFINITIONS AND ABBREVIATIONS

BD	bulk density of compacted mix
MVD	maximum void-less density of loose asphalt mix
RA	reclaimed asphalt
Asphalt	mixture of aggregate, mineral filler and bituminous binder
Constant mass	less than 0.1% change in mass after two successive (more than 1 h) periods of oven-drying
Ndesign	number of gyrations at which the air voids content equal to 4 percent
Angle of gyration	the compaction angle of the gyratory compactor

Rate of gyration	a constant speed of 30 revolutions per minute at which the base of the gyratory compactor is rotated during compaction
Riffle	splitting of a sample to an acceptable size in a manner that keeps the portions as representative as possible
Tare	to reset a balance reading to zero so that it excludes the mass of the container
RA	Recycled asphalt
TMH	Technical manual for highways
TRH	Technical Recommendation for Highways

4. APPARATUS

Test apparatus required in the preparation of asphalt mixes includes the following:

- Gyratory compactor, compaction moulds of steel with inside diameter of 150 mm and 100 mm, and base plate conforming to the requirements of AASHTO T 312 or AASHTO R 83.
- Thermostatically-controlled forced ventilation oven for heating binder, aggregates, and apparatus, and capable of attaining a temperature of 200°C.
- A riffler with suitably sized slots for the particular asphalt mixture.
- Flat-bottom metal pans for heating aggregates
- Metal dishes (mixing bowl), of capacity about 10 kg, for mixing binder and aggregate.
- Scoop for batching aggregates.
- Containers, i.e. tins, beakers, or pouring pots, for heating binder.
- Electronic scale complying with SANS 1649, reading to 0.1 g, capable of weighing at least 8 kg sample or of suitable capacity (binder, aggregates) and compacted specimens.
- Metal spike thermometer, 10°C to 250°C in two degree divisions, for determining temperature of binder, aggregates, and asphalt mixes.
- Mechanical mixer, of capacity at least 10 kg, equipped with metal mixing bowls.
- Large mixing spoon or small trowel.
- Large metal spatula.
- Wide-mouthed funnel.
- Welders gloves for handling hot equipment.
- Paper disks, 150 mm or 100 mm depending on mould size for compaction.
- Paint, markers, or crayons, for identifying test specimens.
- Table fan for cooling compacted specimens.

5. EQUIPMENT CALIBRATION AND VERIFICATION

Equipment calibration shall be done annually in accordance with manufacturer / supplier's instructions and requirements. Items requiring verification include:

- Stress of the loading ram – verify that the vertical stress is 600 ±18 kPa.
- Angle of gyration – verify that the external angle of gyration is 1.25 ± 0.02° or internal angle of gyration is 1.16 ± 0.02°.
- Gyration frequency – the rate of gyration should be 30 gyrations/min.
- Others – oven temperatures, and thermometer.

6. SAMPLE PREPARATION

6.1. AGGREGATE PREPARATION

6.1.1 Dry the aggregates including RA for approximately 16 h in an oven at a temperature of approximately 110°C to a constant mass.

6.1.2 Riffle out each aggregate fraction.

6.1.3 Determine the particle size distribution of each fraction of aggregate and any RA intended for use in the mix in accordance with SANS 3001-AG1.

6.1.4 Calculate the mass of each aggregate fraction required to constitute the mix, and the mass of each aggregate fraction required per specimen (section 9.1). Estimated mass required to produce a gyratory specimen of mass 4 500 g compacted to a standard height of 115 mm, and at the target voids of 4% (for mix design) can be expressed as follows:

$$\boldsymbol{M_T = 5055 - 355 \times \frac{VIM_t}{VIM_s}}$$

Similarly, the estimated mass required to produce a compacted specimen of mass 6 650 g to a standard height of 170 mm, and at the target voids of 7% (for dynamic modulus testing) can be expressed as follows:

$$\boldsymbol{M_T = 7175 - 525 \times \frac{VIM_t}{VIM_s}}$$

$\boldsymbol{M_T}$ = estimated mass of mixture required to produce a compacted specimen with a target air void content of $\boldsymbol{VIM_t}$, g

$\boldsymbol{VIM_t}$ = target voids in the mix, %

$\boldsymbol{VIM_s}$ = specimen voids produced with a compacted specimen mass of $\boldsymbol{M_s}$ %

6.1.5 Weigh each fraction of aggregate according to the mix design to produce a specimen. Mineral filler, if required is not added to the aggregate at this stage.

6.2. HEATING AND MIXING OF THE BINDER AND AGGREGATE

6.2.1 For unmodified binders, determine the mixing and compaction temperatures using a plot of viscosity versus temperature (mixing and compaction temperature corresponding with binder viscosity of 0.17 ± 0.02 Pa.S and 0.28 ± 0.03, respectively).

6.2.2 When modified binders are used the value of the mixing and compaction temperatures shall be obtained from the supplier.

6.2.3 Place dishes (mixing bowl) containing the aggregates in an oven set approximately 25°C above the required mixing temperature. Two to four hours are required for the aggregates to reach the mixing temperature, heat all mixing apparatus.

6.2.4 Heat the binder (unmodified) to the desired mixing temperature (Table 1) for 3 to 5 h. Do not use hotplate to heat the binder, and avoid reheating of the binder. Heat modified binder as per TG 1.

6.2.5 When the aggregates and binder have reached the mixing temperature, place the mixing bowl on the scale and zero the scale.

6.2.6 Form a "crater" in the blended aggregate and add the required amount of binder into the mixture to achieve the desired mass of a batch /specimen. Add the mineral filler, if it is required by the design, at this stage.

6.2.7 Mix the binder and aggregate using the mechanical mixer until the aggregate is thoroughly coated.

Table 1: Typical Mixing and Compaction Temperatures

Mix Type	Binder Type	Mixing Temperature	Compaction Temperature
Sand skeleton	Pen grade (50/70)	150 ± 2.5°C	135 ± 2.5°C
	A-P1	155 ± 2.5°C	145 ± 2.5°C
	A-E2	160 ± 2.5°C	145 ± 2.5°C
Stone skeleton	Pen grade (35/50)	150 ± 2.5°C	140 ± 2.5°C
	A-E2	160 ± 2.5°C	145 ± 2.5°C
	A-P1	165 ± 2.5°C	142 ± 2.5°C
	A-R1	170 ± 2.5°C	145 ± 2.5°C

NOTE 2: ***Temperatures presented in the table may not necessarily be the optimum values for all modified binders; the manufacturer's recommendation should be followed. Once a PG specification for bituminous binders is introduced, table 1 would require revision.***

NOTE 3: ***A wide range of WMA technologies that enable asphalt to be successfully manufactured and compacted at much reduced temperatures are covered in Sabita Manual 32. More work is however, required in terms of protocol for the compaction temperature determination to ensure that all laboratories follow the same method.***

NOTE 4: ***A number of recommended temperatures for SA mixes and binders are given, although the point is made that the user should ensure that the correct temperature is employed, especially in the cases of mixes***

with high proportions of RA where comingling of reclaimed and new binder is relied upon and for modified binders, in which case information should be obtained from the manufacturer.

***NOTE 5**: Loose samples of asphalt mixture should remain uncompacted for the maximum voidless density test.*

***NOTE 6**: Specimens used for mix design and workability determination require sufficient loose mixture to prepare a specimen of 115 ± 2 mm height (Level IB mix design, Sabita Manual 35 / TRH 8).*

***NOTE 7**: Specimens used for moisture damage testing (modified Lottman) require sufficient loose mixture to prepare a specimen of 100 mm diameter by 62.5 ±2 mm height. The sample dimensions should meet the requirements of Sabita Manual 35 / TRH 8.*

***NOTE 8**: Specimens used for Hamburg wheel tracking rutting test requires sufficient loose mixture to prepare a specimen of 60 ± 2 mm height (specimen height should be at least twice the nominal maximum aggregate size).*

***NOTE 9:** Specimens used for dynamic modulus performance testing requires sufficient loose mixture to prepare a specimen of 170 mm ± 5 mm high (Level II and Level III mix design, Sabita Manual 35 / TRH 8).*

6.3. SAMPLES CONTAINING RECLAIMED ASPHALT

6.3.1 Determine the design proportions of the virgin aggregates to RA before the procedure is started and calculate the relative masses required.

6.3.2 Prepare the virgin aggregate, RA and binder as described in 6.1 and 6.2.

6.3.3 Determine the combine penetration of RA and binder by estimating the value as described in TRH 21.

6.3.4 Establish from table 1 the target mixing and compaction temperatures based on the combined penetration value (unmodified binders). For modified binders, the value of the mixing and compaction temperatures should shall be obtained from the supplier.

6.3.5 Heat the combined virgin aggregate and RA to 25°C above the required mixing temperature.

6.3.6 Prepare and heat binder as described in 6.2.

6.3.7 Add the mineral filler if required by the design to the virgin aggregate, RA, mixing it rapidly.

6.3.8 Repeat 6.2.4 to 6.2.6.

Table 2: Percentage RA Versus Mixing Time (TRH 21)

RA in mix (%)	Mixing time (min)
< 15	5
15 – 30	7
> 30	8

6.4. MIXTURE CONDITIONING

6.4.1 Condition the asphalt mixture at the compaction temperature (table 1) before compaction takes place.

6.4.2 Place the mixture in a flat shallow pan at an even thickness, and place the pan in the forced draft oven at compaction temperature.

6.4.3 Condition mixture for volumetric mix design for 2 h ± 5 min in accordance with the short-term oven ageing procedure in AASHTO R 30. Mixture for performance property testing are to be conditioned for 4 h ± 5 min.

6.5. FIELD LOOSE ASPHALT MIX SAMPLES

6.5.1 Measure and record the temperature of the mixture on arrival at the Lab if sampled from the field.

6.5.2 Check whether the temperature of the mixture is within limits for compaction (table 1).

6.5.3 Proceed immediately with compaction (section 7).

NOTE 10: ***When the mixture temperature has fallen below the compaction temperature, it should be reheated.***

NOTE 11: ***Mixture containing unmodified binder are to be placed in an oven for 30 min to bring it up to the compaction temperature. On the other hand, mixture containing modified binder will need longer time (1 h) to be reheated. Reheat cold asphalt mixture that has been left to cool overnight to maximum of 5 h.***

NOTE 12: ***Reheated material should not be used for performance property testing (i.e. dynamic modulus, rutting, moisture damage, permeability, etc.).***

7. COMPACTING THE SPECIMEN

7.1. Turn on the power to the gyratory compactor for the warm up period recommended by the manufacturer.

7.2. Set up the gyratory compactor while the asphalt mixture is short term ageing. This includes setting the vertical stress to 600 kPa, the external or internal angle of gyration to 1.25° or 1.16°, respectively, and the total gyrations as per Table 3.

Table 3: Compaction Requirements (Sabita Manual 35 / TRH 8)

Design level	Design traffic range [E80]	Ndesign
Level IB	0.3 – 3 million	75
Level II	> 3 – 30 million	100
Level III	> 30 million	125

7.3. Before compaction of the first specimen place the compaction mould and base plate, wide-mouth funnel, and spatula in the oven set at the compaction temperature.

7.4. Remove the mould and base plate from the oven and wipe the inside lightly with a damp rag moistened with releasing agent.

7.5. Remove the mixture from the oven after short-term ageing.

7.6. Measure the temperature of the mixture to ensure that it is within the compaction temperature range. If not, sample is to be heated with heating time recorded and added to compaction data.

7.7. Place the base plate in the mould and place a paper disk on top of the base plate.

7.8. Mix the entire aged asphalt mixture to be compacted with a heated spoon, and then using a wide mouth funnel to avoid material loss, transfer mixture into the mould without adding any additional compactive effort.

7.9. Place the loose asphalt mixture (aged sample) in the mould in a single lift, the top of the sample should be slightly rounded. Special attention must be given to filling of the mould to prevent segregation – ensure coarse aggregates are not separated from the rest of the mass during transfer of the mixture into the mould.

7.10. Following 7.9, place a paper disk on top of the mixture.

7.11. Lubricate the loading ram head (apply lubricant to protect the top plate from the ram head).

7.12. Place the mould and content in the compactor and centred under the loading ram. The size of the ram head is 150 mm or 100 mm diameter depending on the size of mould selected for compaction.

7.13. Proceed to locking the mould in place as per manufacturer's instruction and ensure the door and/or other safety devices are activated.

7.14. "Start" the gyratory compactor to begin the compaction process.

7.15. When the specified number of gyrations (or height) has been reached, the gyratory compactor automatically stops, angle and stress are released and the loading ram will be raised.

7.16. Remove the mould containing compacted specimen.

7.17. After trial specimen compaction, the specimen deformation needs to be verified to ensure that the cooling time employed is sufficient. The cooling period may be more sensitive on certain mixtures or binder types and hence needs confirmation.

7.18. Extrude the specimen from the mould.

7.19. Remove the paper disk from top and bottom of the specimen and allow the specimen to cool undisturbed.

7.20. Identify specimen with a suitable marker.

7.21. Allow specimens to stand at the temperature of 20 ± 1°C for a minimum of 16 h before further testing.

***NOTE 13:** The inside of the gyratory mould should be cleaned with a solvent (releasing agent) at the end of each day to avoid asphalt materials build-up. This is easiest when the mould is hot, however care must be taken to prevent injury whilst doing so.*

***NOTE 14:** Avoid loss of material and segregation of coated aggregates while placing the mixture into the mould.*

***NOTE 15:** Compact all the specimens at the same temperature as per table 1 to improve consistency of the test results (high variations in the compaction temperature could introduce variations in the density and engineering property results).*

***NOTE 16:** While the compactor is running, monitor the number of gyrations, stress level and angle of gyration and record any variations.*

***NOTE 17:** When moving the mould, firmly grasp it on either side under the upper flange.*

8. DETERMINING AIR VOIDS CONTENT

8.1. Measure the maximum voidless density of loose asphalt mix samples in accordance with SANS 3001-AS11.

8.2. Determine the bulk density for each specimen following the provisions in Sabita Manual 35/TRH8, i.e.

8.2.1 For mixes other than those intentionally designed to be porous and/or having an open-textured finish, the automatic vacuum sealing method will be prescribed as detailed in AASHTO T 331

8.2.2 For porous mixes with VIM > 12 %, BD is determined by direct measurement of the volume of specimens, as detailed in section 6.6 of the current version of SANS 3001-AS10:2011, Edition 1.

8.3. Calculate the air void content of the specimen in accordance with Sabita Manual 35/TRH8. The voids content shall be 4% ± 0.5% for mix design and workability specimens. However, the voids content for specimens prepared for dynamic modulus and permanent deformation testing must be approximately 7 % ± 0.5%. For EME/high modulus mixes, the voids content must be 4.5% ± 0.5%.

9. CALCULATIONS

9.1. The following sections may be referenced for the calculation of amount of aggregate materials and binder for gyratory compacted specimens.

Assume the following aggregate fractions:

Material	**Blend percentage**
14 mm aggregate	50%
7.1 mm aggregate	20%
Crusher dust	25%
Mineral filler	5%

9.1.1 If the mass of blend of aggregates required for preparation of a mixture is 6 800 g. Then, the amount of material batched for one gyratory compacted specimen is shown below:

14 mm aggregate	50% x 6 800	= 3 400 g
7.1 mm aggregate	20% x 6 800	= 1 360 g
Crusher dust	25% x 6 800	= 1 700 g
Mineral filler	5% x 6 800	= 340 g
Total		= 6 800 g

9.1.2 The quantity of binder required for the specimen may also be calculated as follows:

$$B = 100 \times \left(\frac{M_B}{M_A + M_B}\right)$$

Where

B *is the percentage of binder, expressed as a percentage of total mix*

M_B *is the mass of binder required for one specimen, expressed in grams (g)*

M_A *is the mass of aggregate required for one specimen, expressed in grams (g)*

9.1.3 Calculating the adjustment of the specimen mass

If the gyratory specimen does not compact to the specified height, then use the following equation to correct the amount of material to put in the mould.

$$M_{ADJ} = \left(\frac{h_A \times M_1}{h_B}\right)$$

Where

M_{ADJ} *is the adjusted mass of material for the next specimen, expressed in grams (g)*

h_A *is the required height of the specimen, expressed in millimetres (mm)*

M_1 *is the mass of the compacted specimen, expressed in grams (g)*

h_B *Is the specimen height obtained, expressed in millimetres (mm)*

9.2. Changes in specimen density:

Results of height measurements taken during compaction are used to calculate changes in specimen density expressed as a percent of the maximum voidless density (MVD %). A plot is usually made of the percent MVD versus the log of number of gyrations.

9.3. At the completion of the bulk density, determine the percent compaction (% MVD) at any point in the compaction process as follows:

$$\% \, MVD = \left(\frac{BD \times h_m}{MVD \times h_x}\right) \times 100$$

Where

$\% \, MVD$ *is corrected relative density expressed as a percentage of the the maximum voidless density*

BD *is bulk density of the extruded specimen*

MVD *is the maximum voidless density of the mix*

h_m *is height in millimetres of the extruded specimen*

h_x *is height in millimetres of the specimen after "x" gyrations*

10. REPORTING

Report on the following for each test:

- type of bituminous material, source and content.
- percent binder in specimen, nearest 0.1 percent.
- average diameter of compacted specimens, nearest 1.0 mm.
- mass of specimen, nearest 0.1 g.
- percent maximum voidless density (% MVD).
- period of reheating of the sample if re-heating was necessary.
- temperature of each specimen before compaction.
- compacted bulk density of the spec.

ASP2: PROTOCOL FOR DETERMINING FATIGUE CHARACTERISTICS OF ASPHALT MIX IN THE FOUR-POINT BEAM DEVICE

1. INTRODUCTION

This protocol covers procedures for testing the fatigue life of asphalt mixes using the four-point beam testing device. Test results provide asphalt parameters that can be used to predict the number of load repetitions to fatigue cracking as a function of the tensile strain and mix stiffness. This protocol was developed based on the test method valid at the time of publication of this manual.

2. NORMATIVE REFERENCES

NOTE 1: _The latest versions of the standard test methods are to be used in this protocol._

- Sabita Manual 35/TRH8, Design and use of asphalt in road pavements.
- TMH5, Sampling methods for roads construction materials.
- SANS 3001-AS10, Determination of bulk density and void content of compacted asphalt
- SANS 3001-AS11, Determination of the maximum void-less density of asphalt mixes and the quantity of binder absorbed by the aggregate.
- ASTM D6752 / D6752M, Standard test method for bulk specific gravity and density of compacted asphalt mixtures using automatic vacuum sealing method.
- AASHTO T321, Standard method of test for determining the fatigue life of compacted hot-mix asphalt subjected to repeated flexural bending. For mixes designed in accordance with Sabita Manual 33 the AASHTO T321: 2014 version is to be applied.

3. DEFINITIONS AND ABBREVIATIONS

BD	bulk density of compacted mix
Constant mass	less than 0.1% change in mass after two successive (more than 1 h) periods of drying
Laboratory-produced specimen	rectangular prismatic specimen prepared from slab compacted samples
LVDT	linear variable displacement transducer
MVD	maximum void-less density of loose asphalt mix
Fatigue life	is defined by the lesser of (1) the load cycle at which the product of the specimen stiffness and the loading cycles is a maximum, and (2) the load cycle at which the specimen reaches a 70% reduction of the initial flexural stiffness. For EME/high modulus mixes, fatigue life is indicated by the number of load cycles to reduce the stiffness of the specimen to 50 percent of its initial value.
RA	Recycled asphalt
TMH	Technical manual for highways
TRH	Technical Recommendation for Highways

4. APPARATUS

- Four-point bending testing device, multi-axis data acquisition and control system, and a beam cradle system.
- A temperature-controlled chamber capable of maintaining temperatures from 0 to 20°C to an accuracy of ± 0.5°C.

- Measurement system that is fully computer-controlled and capable of measuring and recording the time history of the applied load (with an electronic load cell), and deflections measured with LVDT.
- Mechanical mixer, of capacity at least 20 kg.
- A slab compactor (e.g. Dyna-Comp roller asphalt compactor).
- A machine for cutting/sawing beam specimen size and ends to the appropriate dimensions. A diamond circular saw is preferred.
- Electronic scale complying with SANS 1649, reading to 0.1 g, capable of weighing 25 kg sample (binder, aggregates) and compacted beams.
- Thermostatically-controlled forced ventilation oven for heating binder, aggregates, and apparatus, and capable of attaining a temperature of 200°C.
- Metal spike thermometer, 10°C to 250°C in two degree divisions, for determining temperature of binder, aggregates, and asphalt mixes.
- Metal pans, of approximate capacity for mixing binder and aggregate.
- Large metal spatula.
- Welders gloves for handling hot equipment.
- Paint, markers, or crayons, for identifying beam specimens.
- Table fan for cooling compacted slabs.

5. EQUIPMENT CALIBRATION AND VERIFICATION

Equipment calibration shall be done annually in accordance with manufacturer / supplier's instructions and requirements. Items requiring verification include:

- Verify the capability of the environmental chamber to maintain the required temperature within the accuracy specified by manufacturer.
- Verify the calibration of load cell and LVDT device of the testing system within manufacturer's recommendations.
- If verification data do not comply with manufacturer's specification, re-calibrate prior to proceeding with testing.

6. SAMPLE PREPARATION

6.1. AGGREGATE PREPARATION

6.1.1 Dry the aggregates including RA for approximately 16 h in an oven at a temperature of approximately 110°C to a constant mass.

6.1.2 Riffle out each aggregate fraction.

6.1.3 Determine the particle size distribution of each fraction of aggregate and any RA intended for use in the mix in accordance with SANS 3001-AG1.

6.1.4 Calculate the mass of each aggregate fraction required to constitute the mix, and the mass of each aggregate fraction required for a slab (section 10.1).

Use the formula BD = M/V. Where, BD = bulk density determined using SANS 3001 AS10.

Assume a typical wearing course mix with A-E2 binder has BD of 2,382 kg/m^3, a volume of 0.0072 m^3 (based on slab length = 400 mm, width = 300 mm, thickness = 60 mm), and binder content of 5%:

The required mass of asphalt mixture would be equal to 2,382 x .0072 x = 17 150.4g. The mass of binder is thus 857.5 g, and total mass of aggregates including the mineral filler is 17 150.4g – 857.5g = 16 292.9g.

6.1.5 Weigh each fraction of aggregate according to the mix design to produce a slab. Mineral filler, if required is not added to the aggregate at this stage.

6.2. HEATING AND MIXING OF THE BINDER AND AGGREGATE

6.2.1 For unmodified binders, determine the mixing and compaction temperatures using a plot of viscosity versus temperature (mixing and compaction temperature corresponding with binder viscosity of 0.17 ± 0.02 Pa.s and 0.28 ± 0.03, respectively).

When modified binders are used the value of the mixing and compaction temperatures shall be obtained from the supplier.

6.2.2 Place dishes (mixing bowl) containing the aggregates in an oven set approximately 25°C above the required mixing temperature. Two to four hours are required for the aggregates to reach the mixing temperature, heat all mixing apparatus.

6.2.3 Heat the binder (unmodified) to the desired mixing temperature for 3 to 5 h (table 1). Do not use hotplate to heat the binder, and avoid reheating of the binder. Heat modified binder as per TG 1.

6.2.4 When the aggregates and binder have reached the mixing temperature, place the mixing bowl on the scale and zero the scale.

6.2.5 Form a “crater” in the blended aggregate and add the required amount of binder into the mixture to achieve the desired mass of a slab. Add the mineral filler, if it is required by the design, to the aggregate.

6.2.6 Mix the binder and aggregate using the mechanical mixer until the aggregate is thoroughly coated.

Table 1: Typical Mixing and Compaction Temperatures

Mix Type	Binder Type	Mixing Temperature	Compaction Temperature
Sand skeleton	Pen grade (50/70)	150 ± 2.5°C	135 ± 2.5°C
	A-P1	155 ± 2.5°C	145 ± 2.5°C
	A-E2	160 ± 2.5°C	145 ± 2.5°C
Stone skeleton	Pen grade (35/50)	150 ± 2.5°C	140 ± 2.5°C
	A-E2	160 ± 2.5°C	145 ± 2.5°C
	A-P1	165 ± 2.5°C	142 ± 2.5°C
	A-R1	170 ± 2.5°C	145 ± 2.5°C

***NOTE 2*: Temperatures presented in the table may not necessarily be the optimum values for all modified binders; the manufacturer's recommendation should be followed. Once a PG specification for bituminous binders is introduced, table 1 would require revision.**

***NOTE 3*: Wide range of WMA technologies that enable asphalt to be successfully manufactured and compacted at much reduced temperatures are covered in Sabita Manual 32. More work is however, required in terms of protocol for the compaction temperature determination to ensure that all laboratories follow the same method.**

***NOTE 4*: A number of recommended temperatures for SA mixes and binders are given, although the point is made that the user should ensure that the correct temperature is employed, especially in the cases of mixes with high proportions of RA where comingling of reclaimed and new binder is relied upon and for modified binders, in which case information should be obtained from the manufacturer.**

6.3. SAMPLES CONTAINING RECLAIMED ASPHALT

6.3.1 Determine the design proportions of the virgin aggregates to RA before the procedure is started and calculate the relative masses required.

6.3.2 Prepare the virgin aggregate, RA and binder as described in 6.1 and 6.2.

6.3.3 Determine the combine penetration of RA and binder by estimating the value as described in TRH 21.

6.3.4 Establish from table 1 the target mixing and compaction temperatures based on the combined penetration value. For modified binders, the value of the mixing and compaction temperatures should shall be obtained from the supplier.

6.3.5 Heat the combined virgin aggregate and RA to 25°C above the required mixing temperature.

6.3.6 Prepare and heat binder as described in 6.2.

6.3.7 Add the mineral filler if required by the design to the virgin aggregate, RA, mixing it rapidly.

6.3.8 Repeat 6.2.4 to 6.2.6.

Table 2: Percentage RA Versus Mixing Time (TRH 21)

RA in Mix (%)	**Mixing Time** (min)
< 15	5
15 – 30	7
> 30	8

6.4. MIXTURE CONDITIONING

6.4.1 Condition the asphalt mixture at the compaction temperature (table 1) before compaction takes place.

6.4.2 Place the mixture in a flat shallow pan at an even thickness, and place the pan in the forced draft oven at compaction temperature.

6.4.3 Condition mixture for 4 h ± 5 min in accordance with the short-term oven ageing procedure in AASHTO R 30.

6.4.4 Field loose asphalt mix samples.

6.4.5 Measure and record the temperature of the mixture on arrival at the Lab if sampled from the field.

6.4.6 Check whether the temperature of the mixture is within limits for compaction (table 1).

6.4.7 Proceed immediately with compaction (section 6.6).

<u>NOTE</u> 5: When the mixture temperature has fallen below the compaction temperature, it should be reheated.

<u>NOTE 6</u>: Mixture containing unmodified binder are to be placed in an oven for 30 min to bring it up to the compaction temperature. On the other hand, mixture containing modified binder will need longer time (1 h) to be reheated. Reheat cold asphalt mixture that has been left to cool down overnight to maximum of 5 h.

6.5. PRODUCTION OF THE SLAB COMPACTED SAMPLES:

6.5.1 Heat all components of the slab mould in the oven set at the compaction temperature.

6.5.2 Input the "mass" and final "thickness" of the sample (normally 60 mm) in the software of the slab compactor.

6.5.3 Load the mould with the asphalt mix into the slab compactor and fix it in position. Special attention must be given to filling of the mould to prevent segregation – pour the mix onto a tray and spread carefully to a uniform thickness of about 50 mm. Quarter the mix and fill half the opposite quarters in the moulds, starting from either ends.

6.5.4 Turn on the machine to start compaction (this is done automatically on some devices).

6.5.5 Remove the sample from the compactor when compaction is complete, and allow sample to cool at a temperature of 20±1°C for at least 16 h.

6.5.6 Cut beam specimens of dimensions 380 mm long by 63 mm wide by 50 mm high from the compacted slabs for the fatigue testing. Cut all four faces of beam specimens using a diamond circular saw. All dimensions should be within ± 2 mm.

6.6. AIR-DRY BEAM SPECIMENS TO A CONSTANT MASS AT A TEMPERATURE OF 20 ± 1°C

6.7. STORAGE – IF SPECIMEN WILL NOT BE TESTED WITHIN 24 H, STORE IN AN ENVIRONMENTALLY PROTECTED STORAGE AREA AT TEMPERATURES BETWEEN 0 AND 20°C.

6.7.1 Place specimens on flat and clean surfaces during the storage period.

6.7.2 Do not stack specimens on top of each other in storage. If samples will be stored for between 1-2 weeks, then a temperature of 20 ± 1°C is adequate.

<u>NOTE 7</u>: Size of compacted slab should be such that at least three beam specimens can be cut and trimmed from it. Record the length, width and thickness of the beam at three different points to an accuracy of 0.1 mm.

<u>NOTE 8</u>: Minimum of three duplicate beam specimens are prepared and tested at the design voids and design binder content for each strain level and temperature.

<u>NOTE 9</u>: For large aggregate mixes, it is suggested that bigger dimensions would be appropriate for the beam specimen. However, more data and discussions are needed to set the dimensions for such mixes.

NOTE 10: Though the testing of four point bending for fatigue life determination described within this protocol follows the prescriptions of the ASTM method, it has been observed that guidelines and suggestions made in the BS EN 12697-24 standard pertaining to assuring consistency and accuracy of results are paramount to consider. Amongst these, required curing time and maximum age of specimens prior to testing are pivotal toward achieving the desired consistency."

7. DETERMINING AIR VOID CONTENT

7.1. Measure the maximum voidless density of loose asphalt mix samples in accordance with SANS 3001-AS11.

7.2. Determine the bulk density for each specimen following the provisions in Sabita Manual 35/TRH8, i.e.

7.2.1 For mixes other than those intentionally designed to be porous and/or having an open-textured finish, the automatic vacuum sealing method will be prescribed as detailed in AASHTO T 331

7.2.2 For porous mixes with VIM > 12 %, BD is determined by direct measurement of the volume of specimens, as detailed in section 6.6 of the current version of SANS 3001-AS10:2011, Edition 1.

7.3. Calculate the air void content of the specimen in accordance with Sabita Manual 35/TRH8. The voids content for the specimen must be equal to design voids ± 0.5%, except for porous asphalt mixes. Beams cut from the road are tested at the air void content at which they are obtained.

8. SPECIMEN CONDITIONING

8.1. Condition test specimens in the environmental temperature chamber (Figure 1a) of the fatigue test set-up. For test temperatures of 5 and 10°C, condition the specimens for approximately 3 h prior to testing. For the test temperature of 20°C, condition the specimens for 2 h.

8.2. A dummy specimen (100 mm in diameter x 150 mm high briquette) of the same asphalt mix with a thermometer in its centre can be placed in the chamber to verify uniform mass temperature of the specimen.

8.3. Allow approximately 30 minutes for specimens to attain equilibrium testing temperature before the next specimen is tested. The equilibrate time may not be exactly the same for all Labs since environmental conditions are more likely to differ from one laboratory to another one. Also different mixes may have different times to achieve the equilibrium temperature.

8.4. For large projects, specimens may be conditioned in a separate environmental chamber that can contain all the specimens.

9. TESTING OF BEAM SPECIMEN

9.1. Set up the four-point beam testing device, the data acquisition and control system.

9.2. For Level II mix design (Sabita Manual 35/TRH8), conduct the fatigue test at one test temperature of 10°C (or specified temperature for a project) and a loading frequency of 10 Hz, at three selected strain levels to generate fatigue curve for the mix. For Level III mix design (Sabita Manual 35/TRH8), conduct the test at three temperatures of 5, 10 and 20°C at 10 Hz, and a minimum of three strain levels to generate fatigue curves for the mix.

9.3. Place the beam specimen in the environmental chamber (Figure 1a), allowing it to equilibrate to the specified testing temperature ± 0.5°C.

9.4. Lower the actuator and slide the beam into the test fixture allowing it to rest on the two outer clamps – fasten the outer clamps. For bitumen rubber mixes, a supporting plate is to be used when sliding specimen into fixture.

9.5. Slowly raise the actuator until the load cell readout registers a contact load – clamp the two inner clamps. When clamped slowly move the actuator until zero load is measured.

9.6. Adjust LVDT to zero. By hand feel each clamp is tight.

9.7. Input the desired strain in the software program. Set the loading frequency at 10 Hz and a sinusoidal load wave.

9.8. Set up appropriate testing conditions.

9.9. For mixes designed in accordance with Manual 35 /TRH8: Set the software to terminate the test at the point where the specimen has achieved a 15% reduction in the peak product of the specimen stiffness (S) and the load cycle (n) (hereinafter referred to as S x n) from the point at which S x n peak has been reached. The strain level selection needs to be of such a magnitude that the S x n peak is not reached prior to 10 000 load cycles being applied to the specimen. For mixtures designed in accordance with Manual 33: Set the software to terminate the test at the point where the a 50 percent reduction of the initial stiffness, determined at the first 50 load cycles /repetitions, is achieved or when a pre-set number of load cycles /repetitions is reached as required.

9.10. Close the environmental chamber, allowing approximately 30 minutes for the test temperature to equalize. During this time the on-specimen LVDT may need re-adjustment

9.11. Start the test in a controlled-strain mode, applying a continuous sinusoidal strain waveform to the specimen. A minimum of three repeat tests should be conducted at each strain level.

9.12. Once test replicates for the mixture have been completed at the required temperatures and strain levels, the point at which 1 million cycles is attained is to be determined by interpolation and reported accordingly.

(a) temperature controlled chamber

(b) four-point beam fatigue cradle

Figure 1: Test Specimens in a Four-Point Beam Fatigue Set-Up

10. CALCULATIONS

10.1. The following sections may be referenced for the calculation of amount of aggregate materials and binder for compacted slab.

Assume the following aggregate fractions:

Material	Blend percentage
14 mm aggregate	50%
7.1 mm aggregate	20%
Crusher dust	25%
Mineral filler	5%

10.1.1 If the mass of blend of aggregates required for preparation of a mixture is 16 000 g, then, the amount of material to be batched for one slab is shown below:

14 mm aggregate	50% x 16 000 g	= 8 000 g
7.1 mm aggregate	20% x 16 000 g	= 3 200 g
Crusher dust	25% x 16 000 g	= 4 000 g
Mineral filler	5% x 16 000 g	= 800 g
Total		= 16 000 g

10.1.2 The quantity of binder required for the slab may be calculated as follows:

$$B = 100 \times \left(\frac{M_B}{M_A + M_B}\right)$$

Where

B *is the percentage of binder, expressed as a percentage of total mix*

M_B *is the mass of binder required for one specimen, expressed in grams (g)*

M_A *is the mass of aggregate required for one specimen, expressed in grams (g)*

10.2. A typical fatigue test results is shown in Figure 2.

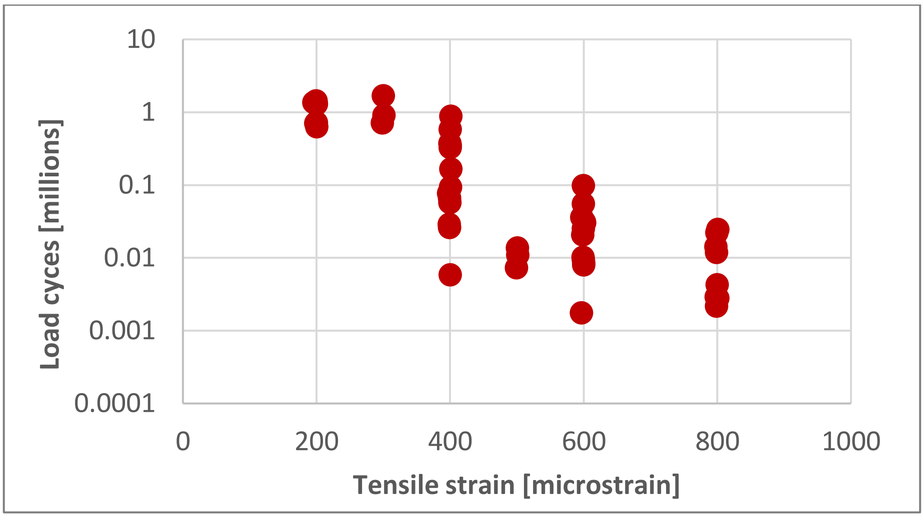


Figure 2: Fatigue Test Results

11. REPORTING

Report on the following for each test:

- Asphalt sample /materials information
- Sample preparation (Lab, plant or field)

- Mix design properties
- Voids content for beam specimen
- Final flexural stiffness
- Number of cycles to failure
- Test temperature
- Strain levels
- Fatigue life curve, if requested

ASP3: DETERMINING THE DYNAMIC MODULUS OF COMPACTED ASPHALT MIXES

1. INTRODUCTION

This protocol describes procedures for testing the dynamic modulus of asphalt mixes. The protocol provides a detailed experimental design to establish the dynamic modulus of compacted asphalt mixes. Dynamic modulus is used for the resilient response characterisation of asphalt in the South African Pavement Design System (SARDS). This protocol was developed based on the test method valid at the time of publication of this manual.

2. NORMATIVE REFERENCES

<u>NOTE 1</u>: The latest versions of the standard test methods are to be used in this protocol.

- Sabita Manual 35/TRH8, Design and use of asphalt in road pavements.
- Sabita ASP1, Protocol for the compaction of asphalt specimens in the gyratory compactor.
- SANS 3001-AS10. Determination of bulk density and void content of compacted asphalt
- SANS 3001-AS11, Determination of the maximum void-less density of asphalt mixes and the quantity of binder absorbed by the aggregate.
- ASTM D6752 / D6752M, Standard test method for bulk specific gravity and density of compacted asphalt mixtures using automatic vacuum sealing method.
- TMH 5, Sampling methods for roads construction materials.
- AASHTO T 378, Standard method of test for determining the dynamic modulus and flow number for asphalt mixtures using the asphalt mixture performance tester (AMPT).

3. DEFINITIONS AND ABBREVIATIONS

Constant mass	less than 0.1% change in mass after two successive (more than 1 h) periods of oven-drying
Dynamic Modulus	absolute value, defined as the maximum stress divided by the recoverable maximum axial strain
Field-produced specimen	field compacted and cored specimen
Laboratory-produced specimen	cylindrical specimen prepared from the gyratory compactor
LVDT	linear variable displacement transducer
Sitting time	the shortest possible time period between compaction and actual time of testing
TMH	Technical manual for highways
TRH	Technical Recommendation for Highways
UTM	Universal Testing Machine

4. APPARATUS

- Universal testing machine[1], integrated multi-axis data acquisition and control system capable of producing a haversine compressive loading.
- A temperature-controlled chamber capable of maintaining temperatures from -5 to 60°C to an accuracy of ± 0.5°C.
- Measurement system that is fully computer controlled and capable of measuring and recording the time history of the applied load (with an electronic load cell), and the axial deformations (measured with LVDT).
- Loading platens with a diameter equal to or greater than that of the test specimen.
- Air compressor capable of producing a pressure of 10 bar and an air flow rate of 300 litres per minute.
- A machine for cutting/sawing test specimen ends at right angle and to the appropriate length. A diamond circular saw is preferred.
- A coring machine with cooling system and a diamond bit for cutting 100 mm diameter test specimens.
- Teflon sheet.
- Apparatus required for gyratory compaction as listed in Sabita ASP1.

5. EQUIPMENT CALIBRATION AND VERIFICATION

Equipment calibration shall be done annually in accordance with manufacturer / supplier's instructions and requirements. Items requiring verification include:

- Verify the capability of the environmental chamber to maintain the required temperature within the accuracy specified by manufacturer.
- Verify the calibration of load cell and LVDT device of the testing system within manufacturer's recommendations.
- If verification data do not comply with manufacturer's specification, re-calibrate prior to proceeding with testing.

6. PREPARATION OF TEST SPECIMEN

6.1. Compact specimen to a diameter of 150 mm and thickness of 170 ± 5 mm in accordance with Sabita ASP1.

6.2. Core the test specimen (diameter of 100 mm) from the centre of the gyratory compacted specimen for each test. Coring is done after a minimum of 16 h from the time of compaction.

6.3. Prepare the ends of the cored specimen to achieve 150 ± 5 mm high specimens by cutting/sawing both ends of the 170 ± 5 mm high gyratory compacted specimen.

6.4. Air-dry cut and cored gyratory compacted specimens to constant mass at a temperature of 20 ± 1°C.

6.5. Storage – if specimen will not be tested within 24 h, store in an environmentally protected storage area at temperatures between 0 and 20°C.

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[1] Pneumatic devices do not provide reliable data above a loading frequency of 10Hz, hence hydraulic devices are recommended for frequency sweep tests that include a loading frequency above 10Hz.

6.5.1 Place specimens on flat and clean surfaces during the storage period. Do not stack specimens on top of each other in storage.

6.5.2 Do not stack specimens on top of each other in storage. If samples will be stored for two days or more, then a temperature of 20 ± 1°C is adequate.

***NOTE 2**: Ensure that the ends and sides of test specimen are at right angles to each other. Surfaces of specimens shall be smooth and free from steps, ridges, grooves, etc. during coring and cutting.*

***NOTE 3**: The number of test specimens required for one binder type is five (i.e. five repeat tests are conducted per mix and the average test results is used to represent the dynamic modulus of the mix).*

7. DETERMINING AIR VOID CONTENT

7.1. Measure the maximum voidless density of loose asphalt mix samples in accordance with SANS 3001-AS11.

7.2. Determine the bulk density for each specimen following the provisions in Sabita Manual 35/TRH8, i.e.

7.2.1 For mixes other than those intentionally designed to be porous and/or having an open-textured finish, the automatic vacuum sealing method will be prescribed as detailed in AASHTO T 331.

7.2.2 For porous mixes with VIM > 12 %, BD is determined by direct measurement of the volume of specimens, as detailed in section 6.6 of the current version of SANS 3001-AS10:2011, Edition 1.

7.3. Calculate the air void content of the specimen in accordance with Sabita Manual 35/TRH8. The voids content for the specimen must be approximately 7 % ± 0.5%. For EME/high modulus mixes, the voids content must be 4.5% % ± 0.5%. Cores cut from the road are tested at the air void content at which they are obtained.

8. SPECIMEN CONDITIONING

***NOTE 4**: Conditioning times may vary slightly depending on the type of environmental chamber in use, and the room temperature of individual laboratories. Conditioning of specimens for a single test temperature such as Level II design and EME/high modulus mixes shall be done using a dummy specimen with a thermocouple (see NOTE 5)*

8.1. Condition test specimens in the environmental temperature chamber (Figure 1) of the dynamic modulus test set-up as per Table 1.

Table 1: Time Required to Bring Specimen to Test Temperature

Test Temperature	Minimum Time	Maximum Time
Room temp. to -5°C	4 h	6 h
-5°C to +5°C	4 h 15	6 h
+5°C to 20°C	4 h	6 h
20°C to 40°C	2 h 30	3 h
40°C to 55°C	1 h 15	2 h

***NOTE 5**: A dummy specimen (100 mm in diameter x 150 mm high briquette) of the same asphalt mix with a thermocouple / thermometer in its centre can be placed in the chamber to verify uniform mass temperature of the specimen.*

***NOTE 6**: Conditioning time must be consistent for all test specimens and form part of reporting on each test.*

Figure 1: Test Specimens in a Temperature Chamber for Conditioning

9. TESTING OF SPECIMEN

9.1. Set up the universal testing machine, data acquisition and control system.

9.2. At Level II mix design (Sabita Manual 35 /TRH8), conduct dynamic modulus test at frequency sweeps of 0.1, 0.5, 1, 5, 10, and 25 Hz at one test temperature of 20°C. EME/high modulus mixes are to be tested at the temperature of 15°C. At Level III mix design (Sabita Manual 35 /TRH8), conduct a full factorial test of dynamic modulus at the five frequencies above and at five temperatures (-5, 5, 20, 40 and 55°C). Maintain temperatures at ± 0.5°C during testing.

9.3. Instrument the specimen with LVDT brackets mounted on the specimen at radial spacing at 120 degrees, and axial spacing at 70 mm around specimen centre (Figure 2). For some mixes such as bitumen rubber mixes, when these LVDT brackets are unstable, larger surface area mounting buttons (available in South Africa) are required for the LVDT brackets.

9.4. Place the specimen in the environmental chamber (Figure 1), allowing it to equilibrate to the specified testing temperature in table 1.

9.5. Place a loading platen (steel disk) at the bottom of the loading frame, and a Teflon paper on top of the plate. The diameter of the Teflon sheet should not be less than the diameter of test specimen.

9.6. Place the specimen on top of the Teflon sheet, and mount the axial LVDTs to the specimen.

9.7. Adjust the LVDT to near the end of its linear range to allow the full range to be available for the accumulation of compressive permanent deformation, if any.

9.8. Place another Teflon sheet followed by loading platen (a steel disk) on top of the specimen.

9.9. Centre the specimen and the loading platens with the hydraulic load actuator visually, to avoid eccentric loading.

9.10. At the extreme temperatures (i.e. -5°C and 55°C) allow 30 minutes for stabilising temperature.

9.11. Apply a contact load equal to five percent of the applied deviator (dynamic) load to the specimen. Manually adjusting the contact load is risky, rather bring the actuator down to within 2mm and allow the software to apply the contact load when starting the test

9.12. Adjust and balance all electronic measuring system as necessary before testing.

9.13. Apply a haversine load waveform with no rest period to the specimens at all test temperatures and loading frequencies.

9.14. To avoid damage to the specimen, ensure that the measured strain is always maintained within the linear range of 75 to 125 microstrain.

9.15. For Level III, test the specimens from lowest to highest temperature, i.e. from -5°C to 55°C. At each test temperature, programme the software to apply the loading from highest to lowest frequency, i.e. from 25 Hz to 0.1 Hz. The time period may vary between 10 and 30 minutes for changing and reconnecting the next test specimen.

***NOTE 7**: When opening and closing the environmental chamber for setting up of the specimen to be tested, allow the chamber temperature to stabilise before running the test.*

***NOTE 8**: The recommended cumulative axial permanent strain of the specimen should not exceed 1500 microstrain at any stage of testing. Specimens that exceed this strain level should be discarded.*

***NOTE 9**: The selected loading frequencies cover performance related vehicle speeds to adequately simulate field conditions.*

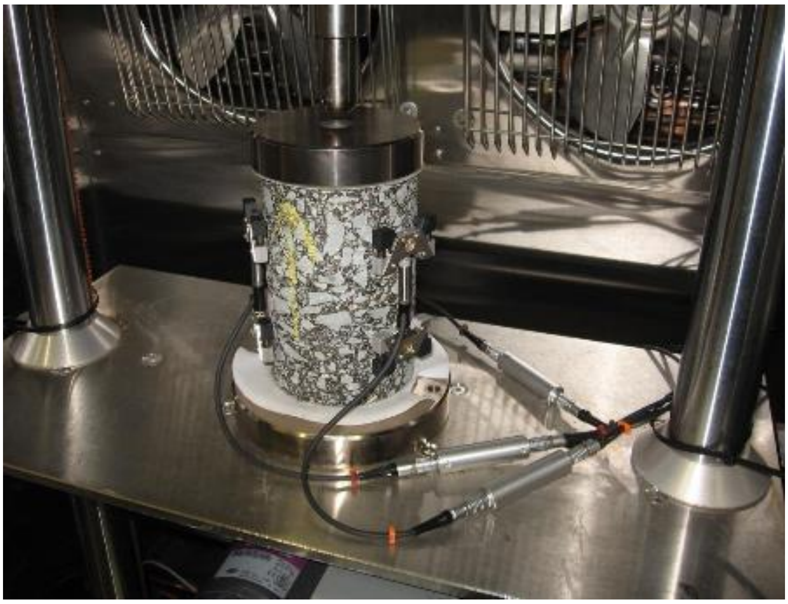

Figure 2: Instrumented Dynamic Modulus Specimen

10. CALCULATIONS

10.1. DYNAMIC MODULUS DEFINITION

The dynamic modulus is defined as the maximum stress (σ_0) divided by the recoverable maximum axial strain (ε_0)

$$|E*| = \frac{\sigma_0}{\varepsilon_0}$$

The dynamic modulus results are automatically computed by the software (table 2, typical results), and are usually presented in a master curve for pavement analysis and design.

Table 2: Dynamic Modulus Test Results

	20 Degrees					
	25Hz	10Hz	5Hz	1Hz	0.5Hz	0.1Hz
Deviator Stress (kPa)	2004	1924	1794	1475	1328	991
Contact	160	117	99	78	63	47
SUMMARY DATA						
Project name	Asphalt					
Sample ID	Sample 1					
Average Diameter (mm)	101.1	101.1	101.1	101.1	101.1	101.1
Average Height (mm)	150.4	150.4	150.4	150.4	150.4	150.4
Average Temperature °C	19	19	19	19	19	19
Dynamic modulus (MPa)	20332	18621	17246	13962	12517	9258
Standard Deviation (MPa)	1108	990	884	691	597	432
CoV	5.4	5.3	5.1	4.9	4.8	4.7
Phase angle (Degrees)	8.2	9.6	10.8	14	15.7	20.3
Standard Deviation (Degrees)	0.5	0.5	0.5	0.6	0.6	0.8
CoV	6.1	5.2	4.6	4.3	3.8	3.9
Average micro-strain (LVDT)	89.6	96.4	97.6	99	99.6	100.6

10.2. DYNAMIC MODULUS TEST RESULTS

Table 3 shows coefficient of variation for dynamic modulus test results on 10 duplicate specimens prepared from 60/70 medium continuously graded asphalt mix as an example. The work leading to the establishment of the coefficient of variation values was done prior to the grade change from 60/70 to 50/70.

Table 3: Coefficient of Variation (Percent) for Dynamic Modulus Test Results (*Anochie-Boateng et al. 2010. Hot-mix asphalt testing for the South African pavement design method (SAPDM)).*

Test Temperature	**Loading Frequency**					
	0.1 Hz	**0.5 Hz**	**1 Hz**	**5 Hz**	**10 Hz**	**25 Hz**
-5°C	4.8	4.6	4.5	4.4	4.3	5.7
5°C	7.4	6.8	6.7	6.4	6.5	6.9
20°C	8.7	6.9	6.4	4.6	4.1	6.1
40°C	14.4	16.2	16.8	16.1	15.4	13.5
55°C	8.8	11.1	12.2	14.5	14.1	16.9

NOTE 10: Coefficient of variation for dense asphalt mixes ranged from 5.5 to 25.8% at the test temperatures of 4, 20 and 40°C, and loading frequencies of 0.01, 0.1, 1 and 10Hz. For SMA mixes, the values were between 4.0 and 18.5% under the same testing conditions (NCHRP 702, 2011).

11. REPORTING

Report on the following for each test:

- Asphalt sample (Lab, plant or field).
- Specimen information (height, diameter).
- Specimen conditioning time at all test temperatures.
- Mix design properties.

- Air void content for gyratory compacted specimen, the average and standard deviations for replicate test specimens.
- Air voids content in the cored and cut cylindrical specimen.
- Conditions of the sample tested due to temperature, frequency effects.
- Dynamic modulus values for all replicate specimens tested in addition to average values, standard deviation and coefficient of variation.
- Master curve, when requested.

ASP4: DETERMINING RUTTING AND STRIPPING OF COMPACTED ASPHALT SAMPLES IN THE HAMBURG WHEEL TRACKING DEVICE

1. INTRODUCTION

This protocol describes procedures for testing the rutting and stripping performance of compacted asphalt samples using the Hamburg wheel tracking device. The protocol is based on the use of *Smartracker* double wheel tracking device, but any similar Hamburg rutting apparatus may be used and may present some changes to items such as specimen mounting and operational instructions, etc.

2. NORMATIVE REFERENCES[2]

- Sabita Manual 35/TRH8, Design and use of asphalt in road pavements.
- Sabita-ASP1, Protocol for the compaction of asphalt specimens in the gyratory compactor.
- SANS 3001-AS10. Determination of bulk density and void content of compacted asphalt
- SANS 3001-AS11, Determination of the maximum void-less density of asphalt mixes and the quantity of binder absorbed by the aggregate.
- ASTM D6752 / D6752M, Standard test method for bulk specific gravity and density of compacted asphalt mixtures using automatic vacuum sealing method.
- TMH 5, Sampling methods for roads construction materials.
- TG 1, The use of modified binders in road construction.
- AASHTO T 324, Standard method of test for Hamburg wheel-track testing of compacted hot mix asphalt (HMA).

3. DEFINITIONS AND ABBREVIATIONS

***NOTE 1**: The latest versions of the standard test methods are to be used in this protocol.*

Constant mass	less than 0.1% change in mass after two successive (more than 1 hour) periods of oven-drying
Creep slope	inverse of the deformation rate in the creep phase
Field-produced specimen	field compacted and cored specimen
Laboratory-produced specimen	cylindrical specimen prepared from the gyratory compactor
LVDT	linear variable displacement transducer
Maximum rut depth	rut depth obtained at the completion of the test
Number of passes at maximum rut depth	at a fixed rut depth value, an asphalt mix with a larger number of passes is more resistant to rutting
Sitting time	the shortest possible time period between compaction and actual time of testing

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Strip slope	the inverse of the deformation rate in the region where the rut depth increases suddenly
Stripping inflection point (SIP)	the point where the rutting curve has a sudden increase in rut depth. SIP reflects the phase where the asphalt binder starts to strip from the aggregate
TMH	Technical manual for highways
TRH	Technical Recommendation for Highways

4. APPARATUS

- Hamburg wheel tracking device.
- High-density polyethylene mould.
- Temperature control system (a water bath capable of maintaining a temperature of 50 ± 0.5°C).
- Rut depth measurement system- an LVDT device capable of measuring depth of impression of the wheel within 0.01 mm and **a** total travel of 25 mm.
- Wheel pass counter that records wheel pass over the specimen.
- Specimen mounting system (a stainless steel tray that can be mounted rigidly to the wheel tracking machine).
- Cutting template mould, a diamond circular saw.
- Apparatus required for gyratory compaction as listed in Sabita ASP1.

5. EQUIPMENT CALIBRATION AND VERIFICATION

Equipment calibration shall be done annually in accordance with manufacturer / supplier's instructions and requirements. Items requiring verification include:

- Measure the water bath temperature at four locations to ensure temperature is within ± 1.0°C of the testing temperature.
- Verify load from the wheel loading assembly at the initial sample contact level as per the manufacturer's recommendations.
- Verify the wheel is reciprocating back and forth on the test sample at 52± 2 passes per minute.

6. PREPARATION OF TEST SPECIMEN

6.1. Compact specimen to a diameter of 150 mm and thickness of 60 ± 2 mm in accordance with Sabita ASP1. Specimen diameter for field cores must be 150 mm (no larger).

6.2. Allow the specimen to cool down at a temperature of 20 ±1°C for at least 16 h.

6.3. Cut specimens using a wet saw after cooling along a secant line/ chord as shown in Figure 1. This is done by trimming off approximately 15 mm vertically from each core using a diamond circular saw.

6.4. Allow specimens to stand (air dry) at a temperature of 20 ± 1°C for a minimum of 16 h before testing.

6.5. Storage – if specimen will not be tested within 24 h, store in an environmentally protected storage area at temperatures between 0 and 20°C.

6.5.1 Place specimens on flat and clean surfaces during the storage period.

6.5.2 Do not stack specimens on top of each other in storage. If samples will be stored for two days or more, then a temperature of 20 ± 1°C is adequate.

***NOTE 2**: The number of test specimens required for one binder content is two, hence a duplicate test per binder content is conducted per mix and the average value is used to represent the rutting performance of the mix.*

***NOTE 3**: Plant produced samples shall be prepared to the same size and thickness as the Lab produced samples (i.e. diameter of 150 mm and thickness of 60 ± 2 mm).*

7. DETERMINING AIR VOID CONTENT

7.1. Measure the maximum voidless density of loose asphalt mix samples in accordance with SANS 3001-AS11.

7.2. Determine the bulk density for each specimen following the provisions in Sabita Manual 35/TRH8, i.e.

7.2.1 For mixes other than those intentionally designed to be porous and/or having an open-textured finish, the automatic vacuum sealing method will be prescribed as detailed in AASHTO T 331

7.2.2 For porous mixes with VIM > 12 %, BD is determined by direct measurement of the volume of specimens, as detailed in section 6.6 of the current version of SANS 3001-AS10:2011, Edition 1.

7.3. Calculate the air void content of the specimen in accordance with Sabita Manual 35/TRH8. The voids content for the specimen must be approximately 7 % ± 0.5%. For EME/high modulus mixes, the voids content must be 4.5% ± 0.5%. Cores cut from the road are tested at the air void content at which they are obtained.

8. SPECIMEN MOUNTING, CONDITIONING AND TESTING

***NOTE 4**:* See manufacturer's instruction for step wise process for the specific equipment you are working with. The primary steps may be in slightly different order but include the general steps provided under this section.

***NOTE 5**:* Some devices from other manufacturers have a pre-heat function for the water, which will change the order of the procedure here as some devices are struggling to get the water temperature to 50°C prior to the maximum 60 min. cut-off time.

8.1. Set up the wheel tracking device for testing.

8.2. Load the specimen such that they are level with the surface of the mould.

8.3. Place the moulds into the mounting tray and fit specimens into each one.

8.4. Fasten the mounting trays into the empty water bath.

8.5. Fill the water bath until the float device floats to the horizontal position, monitoring the temperature of the water on the computer screen. Adjust water temperature as necessary.

8.6. Once the water has reached the test temperature (i.e. 50 ±1°C, Sabita Manual 35 /TRH8), saturate (precondition) the combined test specimens in the water within a time range of 30 to 60 min.

8.7. Enter the specimen/core information into the computer software.

8.8. Select the maximum allowable rut depth /minimum number of passes base on Sabita Manual 35 /TRH8.

8.9. Note the LVDT readout at the beginning of test, this will be subtracted from the total displacement on the screen readout.

8.10. Lower the wheels onto the edge of the test specimen.

8.11. “Start” the wheel tracking testing device software to begin the process.

8.12. When the applied number of desired passes or when the maximum allowable rut depth has been reached, the testing device automatically stops.

8.13. Turn off machine and main power supply after testing is complete. Open the valves beneath the tanks to drain the baths.

8.14. Raise the wheels and remove the specimen mounting system and rutted specimens.

8.15. Clean the water bath, heating coils, wheels, and temperature probe with water and scouring pads (or as per manufacturer's recommendation).

8.16. Use a wet-dry vacuum to remove particles that have settled to the bottom.

8.17. Clean the filter elements and rutting mould.

8.18. Turn the wheels after each test so that the same section of the wheel is not in contact with the specimen from one test to another.

8.19. Open the report program in the software, and record the rut depth at specified number of wheel passes (as per Sabita Manual 35 /TRH8), and the number of passes at failure.

Figure 1: Cut Face Cores/Specimens and Specimen Mounting Mould /System

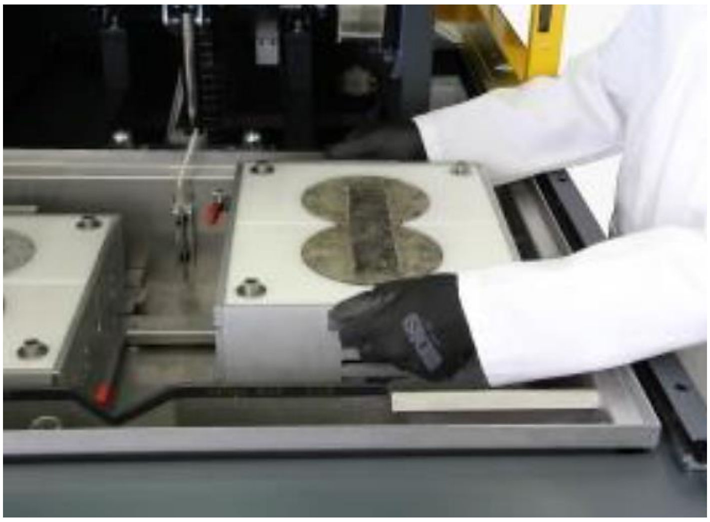


Figure 2: Specimens in Rutting Mould (After Testing)

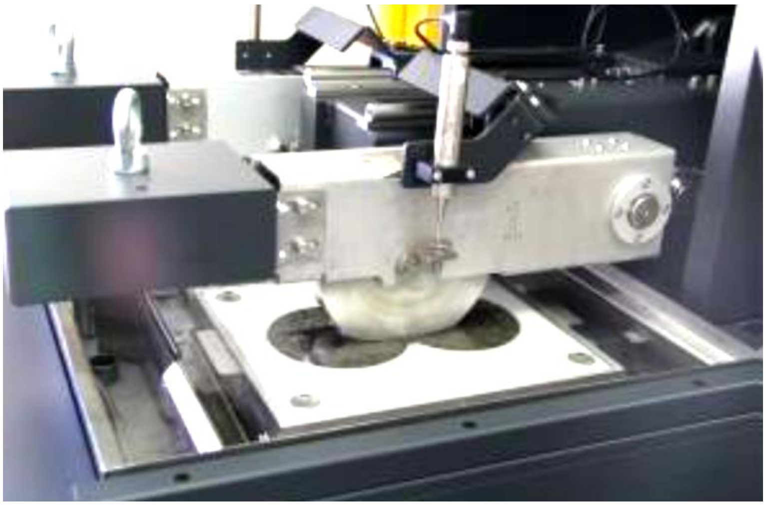


Figure 3: Hamburg Wheel Tracking Testing Set-Up

9. CALCULATIONS

The test outputs include post-compaction consolidation, usually assessed at 1 000 wheel passes, creep slope, stripping slope, and stripping inflection point, as illustrated in Figure 4.

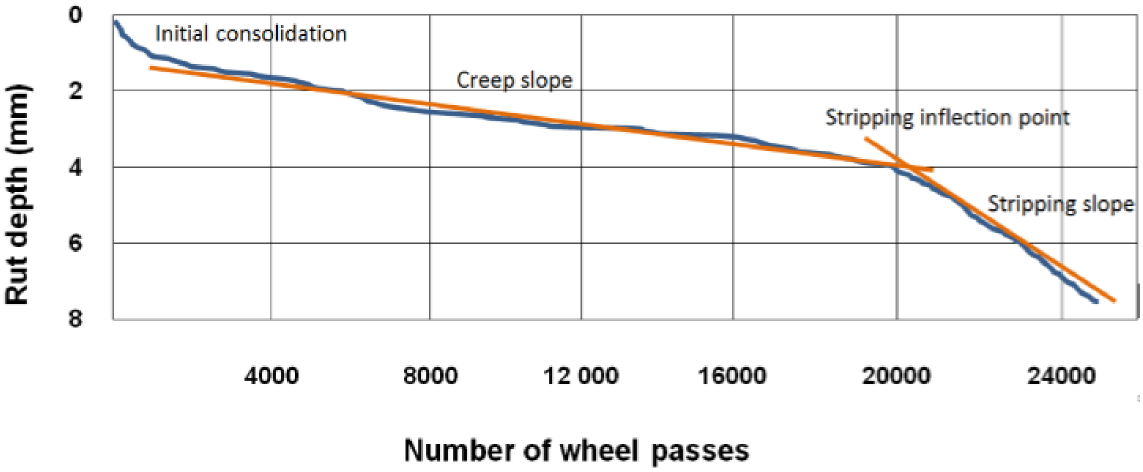


Figure 4: Definition of the Hamburg Wheel Tracking Device Results (Sabita Manual 35 /TRH 8)

10. REPORTING

Report on the following for each test:

- Asphalt sample (Lab, plant or field)
- Specimen information (e.g. air voids)
- Sampling rate (speed)
- Type and amount of anti-stripping additive in the mix
- Rut depth at 1 000, 10 000, 16 000, and 20 000 (Sabita Manual 35 /TRH 8 criteria)
- Minimum number of passes to 6 mm rut (failure criterion, Sabita Manual 35 /TRH 8)
- Minimum number of passes to stripping point
- Test temperature
- Creep slope; Strip slope
- Stripping inflection point

ASP5: PROTOCOL FOR DETERMINING THE FUNDAMENTAL /.INTRINSIC (AIR) PERMEABILITY OF ASPHALT MIXES

IN PREPARATION

ASP8: DETERMINING THE BULK DENSITY OF COMPACTED ASPHALT MIXTURES

1. INTRODUCTION

This test protocol describes the procedures for determining the bulk density of compacted asphalt specimens prepared for design volumetric and/or performance testing. Testing specimen preparation where the selection criteria and procedures are to be used, include but are not limited to N_{des} compaction, rutting, durability, stiffness, permeability and workability.

2. NORMATIVE REFERENCES

- SANS 1649, *Non-automatic self-indicating, semi-self-indicating and non-self-indicating weighing instruments with denominated verification scale intervals.*
- SANS 3001-AG20, *Civil engineering test methods – Part AG20: Determination of the dry bulk density, apparent density and water absorption of material retained on the 5 mm sieve.*
- SANS 3001-AG21, *Civil engineering test methods – Part AG21: Determination of the dry bulk density, apparent density and water absorption of aggregate particles passing the 5 mm sieve.*
- SANS 3001-AS1, *Civil engineering test methods – Part AS1: Making of asphalt briquettes for Marshall tests and other specialized tests.*
- SANS 3001-PR1, *Civil engineering test methods – Part PR1: Determination of uncertainty of measurement, repeatability, reproducibility and bias.*
- AASHTO T331, *Standard Method of Test for Bulk Specific Gravity (G_{mb}) and Density of Compacted Asphalt Mixtures Using Automatic Vacuum Sealing Method*
- SABITA Manual 39, *Laboratory testing Protocols for Binders and Asphalt – Part ASP9: Maximum Voidless Density of asphalt mixtures.*
- SABITA Manual 35, *Design and use of asphalt in road pavements*
- ASTM D 6925, *Standard Test Method for Preparation and Determination of the Relative Density of Hot Mix Asphalt (HMA) Specimens by Means of Superpave Gyratory Compactor*
- SANS 3001 AS10, Determination of the bulk density and void content of compacted asphalt
- SANS 3001 AS11, Determination of the maximum void-less density of asphalt mixes and the quantity of binder absorbed by the aggregate

***NOTE 1*: For dated references, only the cited edition is applicable. Where no date reference is made, the latest version of the standard applies.**

3. DEFINITIONS AND ABBREVIATIONS

Asphalt	mixture of aggregate, mineral filler and bituminous binder
Beam	rectangular prismatic specimen of asphalt cut from slab compacted samples
Briquette	cylindrical asphalt test specimen compacted with either the Marshall or gyratory compactor
Bulk density of mix (BD_{MIX})	mass per unit volume, including the air voids, of a bituminous mixture at a known test temperature, expressed in kg/m^3
Constant mass	less than 0,1% change in mass after two successive (more than 1h) periods of oven-drying at a temperature not exceeding 52 ± 3°C

Maximum void-less density (*MVD*)	mass per unit volume of a void-less bituminous mixture at a known test temperature, expressed in kg/m^3, including aggregate, absorbed and free binder and filler, excluding any air voids
Tare	to reset a balance to read zero so that it excludes the mass of the container, under-weighing frame/basket or any other fixtures that may influence mass determination of the specimen

4. APPARATUS

- Electronic balance fine measurement type complying with SANS 1649, with a capacity of at least 8,0 kg and reading to 0,1 g, and fitted with under-weighing capability so that an object can be suspended below the balance and the mass of the object determined.

- Water bath at least 300 mm deep, thermostatically controlled to maintain a temperature of 25 °C ± 1 °C in which an asphalt cylinder, beam or block can be suspended.

- Wire basket or other suitable method of suspending a compacted specimen in water such that the wire basket is completely submerged with a minimum cover depth of 25 mm to the water surface.

- Wire or suspension cord of fixed length for suspending the specimen in water. The fixed length is determined by establishing the shortest length that can be used with the apparatus so that the basket is submerged when taking readings and as little wire or cord as possible is above the water level. Chains, string, cloth or cord that may be buoyant or stretch are not to be used.

- Thermometer capable of measuring from 0 °C to 50 °C with 0,5 °C graduations.

- Brush with soft bristles.

- Cloth of chamois leather or suitable equivalent approximately 750 mm × 750 mm in size.

- AASHTO T331 apparatus vacuum sealing and associated equipment as listed in this standard, including suitable plastic sealing bags as specified.

- Calliper gauge either Vernier or digital, capable of reading to 0,1 mm.

5. SPECIMEN PREPARATION

5.1 SPECIMEN DIMENSION REQUIREMENTS:

5.1.1 Ensure that the minimum specimen dimensions are such that the diameter is greater or equal to four times the maximum aggregate size for cylindrical specimens. The compacted field or laboratory sample thickness is to be at least three times the nominal maximum size of the aggregate in the specimen for both cylindrical and block or beam specimens prior to cutting/trimming. Special care should be taken when the specimen thickness is 35 mm or less as it may have been damaged during the extraction process and must be noted as such in the report. When specimens are to be tested from pavement slabs, ensure to cut slab samples of at least 150 mm × 150 mm out of the compacted mix. Trim the sides of the slabs neatly with a diamond saw such that the finally trimmed blocks measure approximately 100 mm × 100 mm.

5.2 SPECIMENS PREPARED IN THE LABORATORY:

5.2.1 Ensure that the specimens are briquettes compacted in the laboratory as described in SANS 3001-AS1 or cylindrical or beam type specimens prepared as per Manual 39, which contain no moisture. Store specimens as described in 5.5.

5.3 SPECIMENS OBTAINED FROM SITE OR A MIXING PLANT:

5.3.1 Ensure that the specimens, either core or slabs removed from the compacted pavement, or briquettes from the mixing plant, are dried to constant mass, as per drying procedure in 5.4, prior to testing. This is of particular importance where specimens have been extracted from compacted layers and have undergone coring, cutting and trimming and the likelihood that moisture will be present within the specimen is high.

5.3.2 Check that cores or slabs were not damaged in any manner when extracted from the layer or during transporting and handling prior to testing. Note any damage in the test report and mark the result as questionable. Store the samples as described in 5.5.

5.3.3 Trim off any prime, tack coat or other material adhering to the underside of the core or slab to ensure that the specimen is free of any foreign matter.

5.4 SPECIMEN DRYING

5.4.1 To ensure that no moisture is present in the specimen, the specimen is to be dried either by oven, air-drying or other suitable means at a temperature not exceeding the maximum of 52 ± 3 °C until a state of constant mass is achieved. Special care should be taken that no distortion of the specimen is inflicted during the drying process that may influence specimen volume determination.

5.5 SPECIMEN STORAGE

5.5.1 To avoid distortion of specimens, after removal from the mould or after from trimming, store the specimens with the flat side on a level surface at or below 25 °C until required for testing.

5.5.2 If specimens will not be tested within 24hr, store specimens in an environmentally controlled area at a temperature of 20 ± 1 °C such that:

5.5.2.1 Specimen is supported on a flat, clean surface;

5.5.2.2 Specimens are not stacked upon one another.

6. DENSITY DETERMINATION SELECTION CRITERIA

6.1 GENERAL

Compacted specimens of asphalt consist of aggregate, filler, binder, internal voids containing air and possibly water, and external surface voids on the aggregate. The extent to which surface voids and interconnected voids are taken into account in conjunction with the level of risk associated with the use of the determined Bulk Density result will serve as a guide to the testing procedure required, as set out in 6.2.

6.2 PROCEDURE SELECTION

The following section describes the three procedures that may be selected for the determination of the Bulk Density (BD_{MIX}) of compacted asphalt specimens and the conditions under which they may be utilised:

6.2.1 Bulk density of compacted EME specimens using the saturated surface dry (SSD) method:
According to SABITA Manual 33, in assessing the workability of EME mixes, the SSD procedure is implied in the bulk relative density determination procedures using gyratory compaction, all as per ASTM D6925.
Though not exhaustive, some of the factors that are recommended to be considered during the risk

evaluation include:

- Influence of possible increased asphalt layer permeability on the integrity of the surfacing and base layers;
- Possible loss of bond between the surfacing and base layers;
- Binder-aggregate interface bond loss (stripping);
- Increased risk of pavement cracking;
- Risks associated with premature binder ageing;
- Degree of possible distortion between true voids and SSD determined voids due to interconnectedness of surface voids (i.e., Water absorption into specimen to be monitored closely. Mixtures such SMA or similar have a higher probability of this occurrence.);
- Degree of possible post-construction consolidation (secondary traffic compaction) leading to premature rutting.

6.2.2 Bulk density determined by automatic vacuum sealing method as per AASHTO T331:
Where the Bulk Density is to be determined and utilised in the establishment, control and assurance of Manual 35 designs and the associated performance testing as detailed within Manual 19, 35 and 39; testing shall be done according to this standard.

6.2.3 Bulk density determination by direct dimensional measurement:
This procedure for Bulk Density determination is to be used on specimens with a regular surface and geometric shape, that have intended high void contents, i.e. in excess of 12%, as is the case for porous asphalt mixes designed in accordance with SABITA Manual 17.

7. SPECIMEN COMPACTED DENSITY DETERMINATION

7.1 GENERAL

Once the specimen density determination procedure has been selected as per 6.2 and the specimen(s) prepared as per Section 5 are dry and ready for testing, the selected/specified testing procedure is to be followed.

7.2 DETERMINATION OF DRY MASS IN AIR:

7.2.1 By rubbing the edges of two specimens together, remove the rough edges from the specimen that may potentially puncture vacuum sealing bags or give false measurements during direct dimensional testing.

7.2.2 Clean the specimen with a soft brush to remove any loose material.

7.2.3 Weigh the specimen in air and record the mass, M_1, to the nearest 0,1 g.

7.3 DETERMINING THE VOLUME OF THE SPECIMEN:

Use the applicable one of the following three procedures to determine the volume of the specimen, dependent on the risk imposed by the factors stated in 6.2 and the test requirement.

7.3.1 Volume determination using saturated surface-dry condition (See NOTES 2,3 and 4):

7.3.1.1 Suspend the wire basket or specimen holder on a fixed length of suspension wire or cord under the balance such that the suspended specimen holder is fully immersed in the water bath below the balance during testing. Also ensure that the wire/line is free hanging.

7.3.1.2 Tare the balance and then place the specimen in the specimen holder such that it is completely submerged and is free of any air bubbles.

7.3.1.3 Immerse the specimen in the water bath at a temperature of 25 °C ± 1 °C for 4 min such that all faces of the specimen are equally exposed to the water. When the specimen's temperature is more than 2 °C above the temperature of the water in the water bath, pre-condition the specimen in 25 °C air so that a constant temperature is achieved throughout the specimen prior to immersion.

7.3.1.4 Weigh the specimen in the water and record the mass, M_3, to the nearest 0,1 g once the mass reading stabilises.

7.3.1.5 Remove the specimen from the water and dry it by blotting with the damp cloth to obtain a saturated surface-dry condition. The time of handling from removal out of the water bath to mass determination should take no longer than 10 seconds. Do not tap the specimen on the working or other surface to dislodge the water. Weigh the specimen in air and record the mass, M_2, to the nearest 0,1 g. (See Annex A for recommended blotting procedure)

***NOTE 2**: Saturated-surface-dry is achieved when only the external voids of the specimen are filled with water and there is no free water on the smooth surfaces of the specimen.*

***NOTE 3**: The cloth is considered damp when no free water can be wrung out of the cloth after it has been completely submerged in water*

***NOTE 4**: To achieve the desired level of consistency, it is recommended that the specimen be immersed on the under-weighing specimen holder from commencement of immersion, to ensure a stable reading can be taken within the required time. The time of specimen immersion within water is not to exceed 5 minutes.*

7.3.2 Volume determination by automatic vacuum sealing:

Follow procedure as outlined within AASHTO T331 for the determination of BD_{MIX}. All prescriptions and requirements detailed within the AASHTO T331 method are to be followed, however allowance is made for addition of additional temperature correction factors where water temperature is not 25°C. These factors are provided within Table 1 for use during calculation.

In order to maintain quality, consistency and to guard against common risk of error the following precautions should be taken and verified before, during or after testing as applicable:

- Care is required that vacuum sealing bags are of such a nature that they carry the required approval from the instrument manufacturer or similar to ensure that density and buoyancy effects are in line with that used during the method calibration. Bag approval criteria is to consider at least the vacuum sealing bag density, thickness and size.

- Device sealing strip is to be inspected regularly to ensure that damage to Teflon strip does not cause localised overheating to the vacuum sealing bag which may cause water to penetrate the bag during testing.

- Care should be taken during bag size selection, to ensure that the bag mass to specimen mass ratio is maintained according to calibrated buoyancy effects. The appropriate selection is further required to ensure that there is sufficient room to obtain optimal sealing of the specimen without stretching or tearing of the vacuum seal bag.

- Ensure that the test specimen has equilibrated to the required test temperature of 25°C ± 1 °C prior to vacuum sealing as to avoid any specimen deformation or artificial compaction during the vacuum sealing process.

- Ensure that the device manufacturer guidelines for routine maintenance is adhered to such that vacuum is maintained at the required pressures at all times.

- Ensure that the vacuum sealing bag is inspected for possible defects prior to sealing to limit need for additional drying and retesting of specimens unnecessarily.

- Once a bag is sealed around the specimen, care must be taken when removing the specimen from the vacuum sealing device sealing strip to avoid damage to the seal section that may allow water penetration into the bag and/or specimen during testing.

- When the sealed specimen is transferred into the waterbath for testing, care is to be taken to avoid:

  (a) Entrapment of air below the bag and specimen when the sealed specimen is lowered into the water. Lowering the sealed specimen into the water with the smallest dimension is known to assist in avoiding such air entrapment.

  (b) Entrapment of air within the bag folds when lowering the sealed specimen into the water. Ensuring that the vacuum sealed bag is not folded in any way prior to submerging the sealed specimen into the water would serve as preventive measure.

  (c) Incorrect mass in water readings due to partially submerged vacuum sealing bag. Ensuring that the specimen holder/basket attached to the balance underweighing mechanism is submerged to such a depth that there is adequate space for the specimens to be tested as well as at least 50mm of water cover would aid in avoiding error in this regard.

- Confirmation of the specimen mass after removal from the sealing bag once testing in water is completed is a critical step toward assuring accuracy and should as such not be overlooked.

  As a critical note: The calculation to determine the volume of water displaced performed as per the AASHTO T331 method takes into account the specimen volume as well as the volume and buoyancy effect of the vacuum sealing bag used during the testing. Appropriate calculations are to be used at all times to ensure that the final BD is reflective of the specimen only. Care is to be taken that the water density and temperature correction is done correctly to ensure both bag and sample effects are considered.

7.3.2 Volume determination by direct dimensional measurement, where expected specimen void content > 12%:

Measure the specimen dimensions: length, *l*, breadth, *b* and height, *h*, or diameter, *d*, accurately to 0,1mm with a calliper gauge. Take five measurements of each dimension, evenly spaced across the specimen. The mean value for each dimension is then determined and recorded for calculation.

8. CALCULATIONS

8.1 BULK DENSITY OF COMPACTED ASPHALT MIX (BD_{MIX}):

8.1.1 When the procedure given in 6.2.1 has been used, use the following equation to calculate the BD_{MIX}:

$$BD_{MIX} = \frac{M_1}{M_2 - M_3} \times \rho_w$$

Where:

BD_{MIX} is the bulk density of the mixture, expressed in kilograms per cubic metre (kg/m^3);

M_1 is the dry mass of the specimen, expressed in grams (g)[5];
M_2 is the saturated surface-dry mass of the specimen, expressed in grams (g);
M_3 is the mass of the specimen in water, expressed in grams (g);
ρ_w is the density of water at test temperature as shown in Table 1 (column 3), expressed in kilograms per cubic metre (kg/m^3).

Once the specimen volume determination is complete, it is pertinent that the water absorbed (%) by the specimen is determined:

$$W_{ABS} = 100 \times \frac{M_2 - M_1}{M_1}$$

Where:

W_{ABS} is the percentage water absorbed by the specimen.

Table 1 — Density versus temperature of water

1	2	3
Water temperature (°C)	**Correction factor**	**Water density (kg/m^3)**
24	1,0003	997,4
25	1,0000	997,1
26	0,9997	996,8

8.1.2 When the procedure given in 6.2.2 has been used, use the equations as per AASHTO T331 to calculate the BD_{MIX}.

8.1.3 When the direct dimensional measurement procedure given in 6.2.3 has been used where voids > 12%, calculate the volume from direct measurements using the following equation to allow calculation of the BD_{MIX}.

$$V_{MIX} = l \times b \times h\ (block\ or\ beam\ specimens)$$

$$or$$

$$V_{MIX} = \frac{\pi \times d^2}{4} \times h\ (cores\ or\ briquette\ specimens)$$

Once the volume of the specimen has been computed, calculate BD_{MIX} as follows:

$$BD_{MIX} = \frac{M_1}{V_{MIX}} \times 10^6$$

Where:

l, *b*, *h* and *d* are specimen dimensions, expressed in millimetres (mm);

V_{MIX} is the volume of the mix by direct dimensional measurement, expressed in cubic millimetres (mm^3).

NOTE 5: A worked example of the calculation method is given in Annex B

8.2 VOIDS IN THE MIX (VIM):

The VIM is the difference between the maximum void-less density (MVD) and the BD, expressed as a percentage of the MVD.

Determine the MVD of the mix as described in ASP 9 and calculate the percentage voids in the mix using the following equation:

$$VIM = 100 \times \frac{MVD - BD_{MIX}}{MVD}$$

Where:

VIM is the percentage difference between MVD and BD_{MIX};

MVD is the void-less density of the mix (see ASP9);

expressed in kilograms per cubic metre (kg/m^3).

8.3 PERCENTAGE VOLUME OF VOIDS IN THE MINERAL AGGREGATE (VMA):

The *VMA* is the volume of voids in the bulk compacted mix (excluding binder) expressed as the percentage difference between the volume of the aggregate in the mix and the bulk compacted volume of the mix.

Determine the *VMA* using the following equation:

$$VMA = 100 - \left(\frac{(100 - B) \times (BD_{MIX})}{BD_A}\right)$$

Where:

VMA is the percentage volume of voids in the Mineral Aggregate of the bulk compacted mix;

B is the percentage binder in the mix;

BD_A is the bulk density of total aggregate (see SANS 3001-AG20 and SANS 3001-AG21), expressed in kilograms per cubic metre (kg/m^3).

8.4 PERCENTAGE OF VOIDS FILLED WITH BINDER (VFB):

The *VFB* is the percentage of voids in the bulk mix filled with effective binder, expressed as a percentage of the volume of voids in the bulk compacted mix.

First determine V_{BEF} using the following equation:

$$V_{BEF} = \frac{B_{EF} \times BD_{MIX}}{BD_B}$$

Then determine *VFB* using the following equation:

$$VFB = \frac{100 \times V_{BEF}}{VMA}$$

Where

V_{BEF} is the volume of effective binder expressed as a percentage of the volume of the bulk compacted mix;

B_{EF} is the percentage of effective binder in the mix determined in ASP9 (i.e. the total binder less the binder absorbed);

BD_B is the bulk density of the binder, expressed in kilograms per cubic metre (kg/m^3);

VFB is the percentage of voids in the bulk compacted mix filled with binder.

9. REPORTING

Report the BD_{MIX} to the nearest kilogram per cubic metre (kg/m^3), *VIM*, *VMA* and *VFB* to one decimal place in percentage (%), and water absorption to two decimal places in percentage (%), as well as the following information:

a) the type of mix and where the sample was obtained; and

b) any deformation or damage to the specimens.

10 PRECISION AND BIAS

For the precision and bias requirements to be used for results analysis pertaining to the current protocol refer to SANS 3001 AS 10 and AASTO T331 requirements as applicable.

ANNEX A

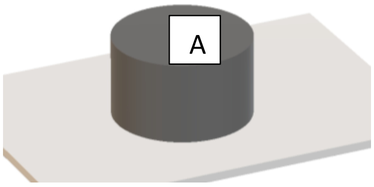
(INFORMATIVE)

Saturated Surface Dry (SSD) blotting Procedure

Though multiple procedures are available for the execution of the blotting of the specimens to obtain the SSD condition during 6.2.1, the following procedures for blotting the specimen are recommended in order to maintain the handling time within specified limits and to avoids any tapping of the specimen against surfaces. The final option selected is subject to the outcome of the internal method validation of the laboratory executing the test.

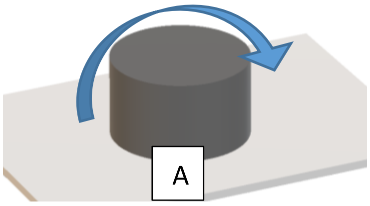
a) Perform the following specimen orientation manoeuvres upon removing specimen from the water bath:

i)



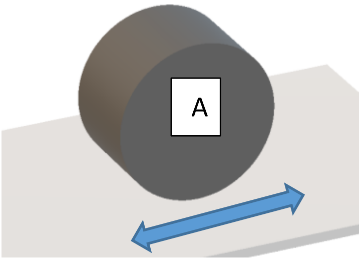
Place specimen on single flat face onto damp cloth.

ii)



Rotate specimen 180° and place specimen on opposite flat face on damp cloth.

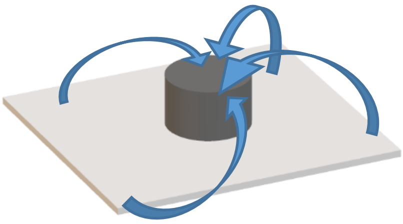
iii)



Rotate specimen 90° and place specimen such that you can role the specimen forward and backward in such a manner that the entire circumference comes into contact with the cloth as required to remove any surface moisture.[6]

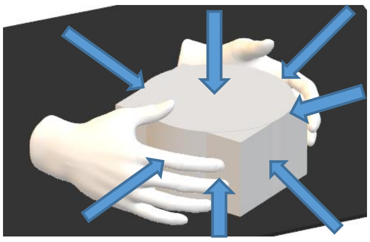
b)

i)



Place the specimen in the centre of the damp cloth and fold the 4 corners of the cloth over the specimen.

ii)



Firmly squeeze the circumference of the enclosed specimen, without shaking, tapping or any other mechanical force, to remove any surface moisture. Upon completion, remove from cloth and determine SSD mass.

***NOTE 6*: Similar procedures to be followed for block density specimens, aside from the rolling, where placement will just continue similar to flat face until all edges have been blotted dry.**

ANNEX B

(INFORMATIVE)

Example of the calculation procedure (Procedure 6.2.2 not included as this is described in AASHTO T331)

B.1 General: Use the information given in table B.1 to calculate B.2 to B.4.

Table B.1 — Information for example calculation

1	2	3
Conditions	**Symbol**	**Mass**
Dry Mass	M_1	1188 g
Saturated Surface Dry	M_2	1193 g
Submerged in water	M_3	705 g
Temperature factor @ 25 °C	ρ_w	997,1 kg/m^3

B.2 Calculate the water absorbed (see 8.1.1):

B.2.1 The water absorbed using the following equation and substituting *M*$_1$:

$$W_{ABS} = 100 \times \frac{M_2 - M_1}{M_1} = 100 \times \frac{1193 - 1188}{1188} = 0{,}42\ \%$$

B.3 Calculate the bulk density of the mix:

B.3.1 Calculate the BD_{MIX} when using procedure 6.2.1 using the following equations:

$$BD_{MIX} = \frac{M_1}{M_2 - M_3} \times \rho_w = \frac{1188}{1193 - 705} \times 997{,}1 = 2427\ kg/m^3$$

B.3.2 Calculate the V_{MIX} *and* BD_{MIX} when using procedure 6.2.3 when *specimen voids* > 12 % by using the following information for a cylindrical example:

Table B.3 — Information for example calculation

Specimen		
Diameter (*d*)	Height (*h*)	Dry Mass (M_1)
101 mm	63 mm	1224,9 g

$$V_{MIX} = \frac{\pi \times d^2}{4} \times h = \frac{\pi \times 101^2}{4} \times 63 = 504\,706{,}34\ mm^3$$

$$BD_{MIX} = \frac{M_1}{V_{MIX}} \times 10^6 = \frac{1224.9}{504\,706{,}34} \times 10^6 = 2427\ kg/m^3$$

B.4 Calculate the VIM (see 8.2):

Calculate the voids in the mix, using the following equation:

MVD: 2534 kg/m^3 (determined as described in ASP 9)

BD_{MIX}: 2427 kg/m^3

$$VIM = 100 \times \left(\frac{MVD - BD_{MIX}}{MVD}\right) = 100 \times \left(\frac{2534 - 2427}{2534}\right) = 4{,}2\ \%$$

B.5 Calculate the *VMA* (see 8.3):

Calculate the percentage volume of voids in mineral aggregate in the bulk compacted mix, using the following equation:

$BD_{A:}$ 2750kg/m^3 (determined as described in SANS 3001 AG20 and AG21)
B: 5% (determined as per SANS 3001 AS20)
$BD_{MIX:}$ 2427kg/m^3

$$VMA = 100 - \left(\frac{(100 - B) \times (BD_{MIX})}{BD_A}\right) = 100 - \left(\frac{(100 - 5) \times (2427)}{2750}\right) = 16{,}2\ \%$$

B.6 Calculate the VFB (see 8.4):

Calculate the percentage of voids in the bulk compacted mix filled with effective binder, using the following equations:

B_{EF}: 4.47% (determined as described in ASP9)

$$V_{BEF} = \left(\frac{B_{EF} \times BD_{MIX}}{BD_B}\right) = \left(\frac{4.47 \times 2427}{1020}\right) = 10{,}6\ \%$$

$$VFB = \left(\frac{100 \times V_{BEF}}{VMA}\right) = \left(\frac{100 \times 10.6}{16.2}\right) = 65{,}8\ \%$$

11. REFERENCES

ASTM D 1188, *Standard test method for bulk specific gravity and density of compacted bituminous mixtures using coated samples.*

ASTM D 2726, *Standard test method for bulk specific gravity and density of non-absorptive compacted bituminous mixtures.*

EN 12697-6, *Bituminous mixtures – Test methods for hot mix asphalt – Part 6: Determination of bulk density of bituminous specimens.*

TMH 1, *Standard methods of testing road constructions materials.*

TMH 5, *Sampling methods for road construction materials*.

AASHTO T166, *Bulk specific gravity (G_{mb}) of compacted asphalt mixtures using saturated surface dry specimens*

AASHTO T331, *Standard Method of Test for Bulk Specific Gravity (G_{mb}) and Density of Compacted Asphalt Mixtures Using Automatic Vacuum Sealing Method*

ASP9: PROTOCOL FOR DETERMINING THE MAXIMUM VOIDLESS DENSITY OF ASPHALT MIXTURES

1. INTRODUCTION

This test protocol describes the procedure for determining the maximum voidless density of asphalt specimens prepared for design volumetric properties and/or performance testing.

2. NORMATIVE REFERENCES

- SANS 1649, Non-automatic self-indicating, semi-self-indicating and non-self-indicating weighing instruments with denominated verification scale intervals.
- SANS 3001-AG20, Civil engineering test methods – Part AG20: Determination of the dry bulk density, apparent density and water absorption of material retained on the 5 mm sieve.
- SANS 3001-AG21, Civil engineering test methods – Part AG21: Determination of the dry bulk density, apparent density and water absorption of aggregate particles passing the 5 mm sieve.
- SANS 3001-PR1, Civil engineering test methods – Part PR1: Determination of uncertainty of measurement, repeatability, reproducibility and bias.
- AASHTO T331, Standard Method of Test for Bulk Specific Gravity (G_{mb}) and Density of Compacted Asphalt Mixtures Using Automatic Vacuum Sealing Method
- SABITA Manual 39, Laboratory testing Protocols for Binders and Asphalt – Part ASP1: Protocol for the compaction of asphalt specimens in the gyratory compactor.
- SABITA Manual 39, Laboratory testing Protocols for Binders and Asphalt – Part ASP8: Bulk Density of Compacted Asphalt Mixtures.
- SABITA Manual 35, Design and use of asphalt in road pavements
- SANS 3310-1/ISO 3310-1, Test sieves – Technical requirements and testing – Part 1: Test sieves of metal wire cloth.
- SANS 3310-2/ISO 3310-2, Test sieves – Technical requirements and testing – Part 2: Test sieves of perforated metal plate.
- TMH 5, Sampling methods for road construction materials.
- SANS 3001 AS10, Determination of the bulk density and void content of compacted asphalt.
- SANS 3001 AS11, Determination of the maximum void-less density of asphalt mixes and the quantity of binder absorbed by the aggregate.
- ASTM D 2041: Standard Test Method for Theoretical Maximum Specific Gravity and Density of Asphalt Mixtures.

***NOTE 1**: For dated references, only the cited edition is applicable. Where no date reference is made, the latest version of the standard applies.*

3. DEFINITIONS AND ABBREVIATIONS

Asphalt	mixture of aggregate, mineral filler and bituminous binder
Constant mass	less than 0,1% change in mass after two successive (more than 1h) periods of oven-drying

Maximum void-less density (MVD)	mass per unit volume of a void-less bituminous mixture at a known test temperature. It includes the aggregate, absorbed binder and the free binder in the mix excluding any air voids
Surfactant	wetting agent
Tare	to reset a balance to read zero so that it excludes the mass of the container and any other fixtures that may influence mass determinations

4. APPARATUS

• Electronic balance	fine measurement type complying with SANS 1649, with a capacity of at least 8,0 kg and reading to 0,1 g
• Flask/Bowl	of at least 2 L capacity, or other suitable container of 3 L capacity when dealing with larger aggregate size mixtures
• Vacuum Pump	capable of maintaining vacuum pressure below a maximum of 30 mm mercury (Hg), fitted with a manometer or a vacuum gauge fitted close to the pump to ensure there is no difference in the reading between the pump and the actual manometer/gauge reading.
• Water bath	at least 150 mm deep, thermostatically controlled to maintain a temperature of 25 °C ± 1 °C in which the majority of the flask is covered
• Thermometer	capable of measuring from 0°C to 50°C with 0,5°C graduations.
• Spout stopper	of rubber with suitable tubing
• Glass plate	flat glass plate suitable for closing the mouth of the flask
• Stirring rod	to agitate the particles inside the flask to aid in removing air bubbles
• Surfactant	suitable commercial wetting agents, eg. Teepol
• Sieves	that comply with SANS 3310-1 or SANS 3310-2, as applicable

5. SPECIMEN PREPARATION

5.1 Specimen mass requirements:

5.1.1 Ensure that the minimum specimen size conforms to the recommendations as outlined in Table 1. Care is to be taken when dealing with a mixture containing varying density such that a flask of suitable volume and/or suitable sample size (specifically volume of material) is used. If cores or slab samples are used for test specimen preparation, it must be noted that a single core or cut block may not contain sufficient material to obtain a satisfactory result on the test specimen. When multiple core or block specimens are to be combined to obtain the test specimen, care should be taken to ensure that the section under evaluation is adequately represented by the specimen(s). Further thereto it must be ensured that aggregate particles with cut faces be removed from the specimen as this will cause misrepresentation of the test result.

5.2 Specimens prepared in the laboratory:

5.2.1 Specimens mixed in the laboratory are to be prepared as per the procedure outlined within ASP1. Ensure that the aggregate consists of all the different fractions that will be used in the proportional blend to achieve the required grading.

5.2.2 Once mixing has been completed, allow the specimen to cool to room temperature.

5.2.3 Loosen the specimen, such that there are no lumps or agglomerations larger that 7mm. (See NOTE 2)

NOTE 2: Agitating the material during the cooling process is recommended such that additional reheating is avoided and to maintain the specimen in a loose state. Use of mechanical means to break agglomerates is not permitted.

5.3 Specimens obtained from site or a mixing plant:

5.3.1 Ensure that the specimens, either core(s) or slabs removed from the compacted pavement, or briquettes from the mixing plant, are dried to constant mass as per section 5.4, prior to testing when it is suspected that moisture is present within the specimen. Take a field sample in accordance with TMH 5.

5.3.2 As the original sample is in a compacted state when obtained from coring or slab cutting, heat the sample to a temperature not exceeding 110 °C, to allow loosening of the sample for specimen preparation. Refer to 5.1.1 for additional aspects of preparation for field obtained samples.

5.3.3 Once the representative sample has been obtained, proceed to final preparation as outlined in 5.2.2 and 5.2.3.

5.4 Specimen Drying

5.4.1 When core or slab samples are to be used to prepare MVD specimens, care must be taken to ensure that the specimen is dried either by oven, air-drying or other suitable means at a temperature not exceeding 52 ± 3°C until a state of constant mass is achieved.

Table 1 — Minimum sample sizes

1	2
Nominal maximum particle size (mm)	**Recommended specimen size (g)**
≥ 37,5	3 000 (See note 3)
20 to 28	2 500 (See note 3)
≤ 14	1 500

NOTE 3: To ensure that a suitable volume of material is used during the testing process for larger aggregate mixtures, use of a suitable container with at least a 3 L volume is recommended for use.

6. MVD DETERMINATION PROCEDURE

6.1 De-airing the mixture

6.1.1 Clean, dry and weigh the flask/bowl and glass plate and record the mass, M_1, to the nearest 0,1 g.

6.1.2 Fill the flask to approximately one third full with the mixture prepared in accordance with section 5 of suitable size as per Table 1, weigh and record the mass, M_2, to the nearest 0,1 g. Separate the particles of the mixture manually as much as possible to avoid lumps or agglomerates larger than 7 mm in the flask.

NOTE 4: To ensure that there is no entrapment of air within the fine proportion of the mixture, lumps/agglomerates are to be broken down as far as reasonably practical.

6.1.3 Add water to the flask until the sample is completely covered by 5 cm of water without filling the flask.

NOTE 5: It is recommended that the fill height of the water be such that there is sufficient room to prevent any water from entering the vacuum system during pumping and/or agitation.

6.1.4 Add two drops of a 10 % solution of surfactant to facilitate the removal of entrapped air.

NOTE 6*: Care is to be taken not to overdose the surfactant solution or using wetting agents that are very active, as it will cause frothing under reduced pressure.

6.1.5 Attach the vacuum pump to the flask, and ensure a tight seal is obtained.

6.1.6 Reduce the pressure in the system, such that the vacuum pressure is maintained below a maximum of 30 mm mercury. At intervals of approximately 2 min agitate the flask by tapping the sides with the hand while holding it firmly and maintain the reduced pressure for a period of 15 min ± 2 min.

***NOTE 7:* As the mercury reading increases, i.e. moves closer to 30mm, the effectiveness of the vacuum in removing entrapped air decreases. It is recommended that the pressure be maintained as close to 25mm Hg to promote consistency of testing and the pressure should be achieved within 2 minutes of starting the test. If the pressure is not achieved during this period, the test is to be restarted. At the reduced pressure the water may start to boil. Excessive boiling is not desirable and may be stopped by increasing the pressure slightly until the boiling stops, ensuring that the pressure remains below 30 mm of mercury at all times.**

6.1.6 Restore the flask gradually to atmospheric pressure and stir with the stirring rod. Ensure that no bubbles are visible in the sample after 1 min. If bubbles are still present repeat 6.1.5. Ensure that no air is reintroduced while stirring and that the sample remains covered with water at all times.

NOTE 8*: Bubbles will always be visible at the reduced pressure but should disappear when the pressure is returned to atmospheric pressure. The pressure should be reduced gradually to ensure that the mercury is not ejected from the manometer when using a mercury manometer as well as ensuring the integrity of the flask is maintained.

6.2 Determination of the mass and volume of the de-aired mixture

6.2.1 Fill the flask with water. Tilt the flask slightly to allow the water to run down the inside of the flask to minimise the bubbles that form at the interface. Ensure that the sample remains covered at all times.

6.2.2 Condition the contents of the flask to a temperature of 25 °C ± 1 °C by placing the flask in the water bath for 10 min. Measure the temperature inside the specimen to confirm that the specimen is at the correct temperature.

6.2.3 Make sure the flask is full of water and seal the mouth with the glass plate ensuring that no air is trapped in the flask. Wipe off all excess water on the outside of the flask as well as the glass plate and dry without disturbing the seal created at the interface of the flask and the glass plate. Weigh the flask assembly including the contents and record the mass, M_3, to the nearest 0,1 g.

6.2.4 Empty the flask, clean it, refill with water at a temperature of 25 °C ± 1 °C and seal the mouth with the glass plate ensuring that no air is trapped in the flask. Weigh the flask assembly filled with water and record the mass, M_4, to the nearest 0,1 g.

6.2.5 When the pores of the aggregate are not thoroughly sealed by the bituminous film, they may become saturated with water during the vacuum procedure to determine if this has occurred, proceed as follows:

a) Drain the water from the container. Decant the water through a 75 µm sieve to prevent the loss of fines particles.

b) Break several of the large pieces of aggregate and examine the broken surfaces for wetness. When there is no doubt that the aggregate is dry proceed to 6.4. When it is suspected that the aggregate has absorbed water, continue to 6.3 to determine if a correction is needed.

***NOTE 9:* When utilising a glass flask, care should be taken to ensure that a firm grip be maintained on the flask when moving the flask and contents around to avoid injury.**

6.3 Porous aggregate mixes

If the outcome of the aggregate wetness verification in 6.2.5 indicates the presence of water absorbed by the aggregate:

6.3.1 Spread the sample on a flat tray with a non-absorptive surface and place in front of a fan to remove the surface moisture. Break agglomerations of the mixture by hand. Stir the sample intermittently for approximately 2 h in such a way that the aggregate particles are rolled over rather than simply moved horizontally on the tray. Take care to prevent the loss of particles of the asphalt mixture.

6.3.2 Weigh the tray and the sample at 15 min intervals. When the loss in mass is less than 0,05 % for successive readings, the sample is considered to be surface dry.

6.3.3 Weigh the surface dry aggregate and record the mass, M_5, to the nearest 0,1 g.

6.4 Bulk density of aggregate and binder

To carry out the calculations within section 7, the following is required:

6.4.1 Determine the bulk density (BD_A) of the aggregate in accordance with SANS 3001-AG20 and SANS 3001-AG21.

6.4.2 For the purpose of the calculations, the bulk density of penetration grade binder, (BD_B), may be taken as 1 020 kg/m^3. Where modified binders are used obtain the BD_B of the binder from the supplier.

7. CALCULATIONS

7.1 Maximum void-less density

Calculate the maximum void- less density (MVD) of the bituminous mixture to the nearest 1 kg/m^3 using the following equations:

a) When no water is absorbed by the aggregate (see 6.2):

$$MVD = \frac{(M_2 - M_1)}{\frac{((M_4 - M_1) - (M_3 - M_2))}{\rho_w}}$$

where:

MVD is the maximum void-less density of the mix, expressed, in kilograms per cubic metre (kg/m^3);

M_1 is the mass of the flask assembly, expressed in grams (see 6.1.1);

M_2 is the mass of the flask assembly and sample, expressed in grams (g) (see 6.1.2);

M_3 is the mass of the flask assembly and sample filled with water, expressed in grams (g) (see 6.2.3);

M_4 is the mass of the flask assembly filled with water, expressed in grams (g) (see 6.2.4);

ρ_w is the density of water at test temperature as shown in table 2, expressed in kilograms per cubic metre (kg/m^3);

b) When water is absorbed by the aggregate (see 6.2 and 6.3):

$$MVD = \frac{(M_2 - M_1)}{\frac{\left((M_4 - M_1) - \left(M_3 - (M_5 + M_1)\right)\right)}{\rho_w}} = \frac{(M_2 - M_1)}{\frac{\left((M_4 - M_3 + M_5)\right)}{\rho_w}}$$

where:

M_5 is the mass of the aggregate surface dry in air, expressed in grams (g).

Table 2 — Water density at varying temperature

1	2
Water temperature (°C)	**Water density (kg/m3)**
24	997,4
25	997,1
26	996,8

7.2 Binder absorption

7.2.1 In the following calculations it is important to remember that binder, B, and effective binder, B_{EF}, are expressed as a percentage of the mass of the mix (aggregate plus binder). Absorbed binder, B_{ABS}, is expressed as a percentage of only the dry aggregate in the mix.

Calculate the total volume of the aggregate and the binder in the sample using the BD_A and BD_B (see 6.4), and the following equations:

For the mass of the binder in the mix:

$$M_B = B \times \frac{(M_2 - M_1)}{100}$$

where:

M_B is the mass of binder in the mix, expressed in grams (g);

B is the percentage binder in the mix and is expressed as a percentage of the mass of the mix (aggregate plus binder).

For the mass of the aggregate in the mix:

$$M_A = \frac{(100 - B) \times (M_2 - M_1)}{100}$$

where:

M_A is the mass of aggregate in the mix, expressed in grams (g).

For the volume of the binder in the mix:

$$V_B = \frac{1000 \times M_B}{BD_B}$$

where:

V_B is the volume of binder in the mix, expressed in cubic centimetres (cm^3);

BD_B is the bulk density of the binder, expressed in kilograms per cubic metre (kg/m^3) (see 6.4).

For the volume of the aggregate in the mix:

$$V_A = \frac{1000 \times M_A}{BD_A}$$

where:

V_A is the volume of aggregate in the mix, expressed in cubic centimetres (cm^3);

BD_A is the bulk density of the aggregate, expressed in kilograms per cubic metre (kg/m^3) (see 6.4)

For the total volume of the binder and the aggregate in the mix:

$$V_T = V_B + V_A - V_{BABS}$$

where:

V_T is the total volume of aggregate and binder in the mix, taking the binder absorbed into the aggregate in consideration, expressed in cubic centimetres (cm^3).

V_{BABS} is the volume of binder absorbed by the aggregate, expressed in cubic centimetres (cm^3).

7.2.2 Calculate the de-aired volume of the mix using the following equations:

a) When no water is absorbed by the aggregate (see 7.1(a)):

$$V_{DA} = 1000 \times \frac{((M_4 - M_1) - (M_3 - M_2))}{\rho_w}$$

where:

V_{DA} is the volume of the de-aired mix, expressed in cubic centimetres (cm^3);

ρ_w is the density of water at the test temperature.

b) When water is absorbed by the aggregate (see 7.1(b)):

$$V_{DA} = 1000 \times \frac{((M_4 - M_1) - (M_3 - (M_5 + M_1)))}{\rho_w} = 1000 \times \frac{(M_4 - M_3 + M_5)}{\rho_w}$$

NOTE 10: ***From volumetric analysis, as depicted in Figure 1, the total volume of the aggregate and binder in the mix (V_T), is therefore equal to the volume of the mixture excluding air, i.e. de-aired volume of mix (V_{DA}), therefore: $V_T = V_{DA}$***

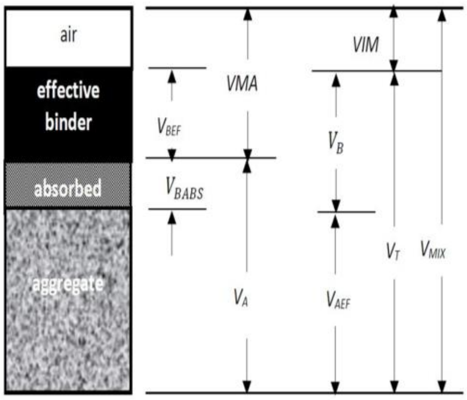


Figure 1: Definition of Mass and Volumes of Mix Components

7.2.3 Calculate the volume of binder absorbed

$$V_{BABS} = V_A + V_B - V_{DA}$$

7.2.4 Calculate the mass of binder absorbed

$$M_{BABS} = \frac{(V_{BABS} \times BD_B)}{1000}$$

where:

M_{BABS} is the mass of the binder absorbed by the aggregate, expressed in grams (g).

Calculate the percentage of binder absorbed by the aggregate, using the following equation:

$$B_{ABS} = 100 \times \frac{M_{BABS}}{M_A}$$

where:

B_{ABS} is the percentage binder absorbed by the aggregate in the mix and is expressed as a percentage of the mass of the dry aggregate in the mix.

Calculate mass of effective binder in the mix, expressed in grams (g)

$$M_{BEF} = M_B - M_{BABS}$$

7.2.7 Calculate the percentage of effective binder in the mix, using the following equation:

$$B_{EF} = 100 \times \frac{M_{BEF}}{(M_A + M_B)}$$

where:

M_{BEF} is the mass of effective binder in the mix, expressed in grams (g);

B_{EF} is the percentage of effective binder in the mix, expressed as a percentage of the mass of the mix (aggregate plus binder).

8. REPORTING

Report the results obtained, with the binder absorption to one decimal place in percentage (%), the maximum void-less density to the nearest 1 kg/m^3, and also include the following:

the type of mix;

the size of specimen; and

the type of sample: such as laboratory mixed field sample or core sample.

9. PRECISION AND BIAS

The precision and bias requirements to be used for results analysis pertaining to the current method relates by procedure as follows:

Reference standard: Precision prescriptions provided in SANS 3001 AS11 (2011):

The values in Table 3 for sigma, σ, and the required precision E (two-sided case and precision category PV, variable) for the statistical analysis of the test results as described in SANS 3001-PR1 is to be used:

Table 2 — SANS 3001 AS11 Precision Prescriptions

Aspect	***σ***	***E***
MVD	5.0	3.5

PROTOCOLS FOR

BITUMEN STABILISED MATERIALS

BSM1: DETERMINING THE FOAMING CHARACTERISTICS OF BITUMEN

1. SCOPE

This test method concerns the determination of the foaming characteristics of bitumen.

2. REFERENCES

The following referenced documents are indispensable for the application of this test method. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies. Information on currently valid national and international standards can be obtained from the SABS Standards Division.

- ASTM D140. Standard Practice for Sampling Bituminous Materials.
- SANS 1649. Non-automatic self-indicating, semi-self-indicating and non-self-indicating weighing instruments with denominated verification scale intervals.
- TG2, Test Method BSM2. Laboratory mix design of bitumen stabilised material.
- TG2, Test Method BSM3. Vibratory hammer compaction for test specimens of bitumen stabilised material.
- TG2, Test Method BSM4. Determination of the indirect tensile strength of bitumen stabilised material.
- TG2, Test Method BSM5. Determination of the shear properties of bitumen stabilised material (Triaxial test).
- TG2, Bitumen Stabilised Materials guideline for the Design and Construction of Bitumen Emulsion and Foamed Bitumen Stabilised Materials.
- TMH 5, Sampling methods for road construction materials. Pretoria.

3. DEFINITIONS

3.1 Binder

Bitumen and bituminous materials. Avoid using the term “binder” with bitumen stabilised materials (BSMs). This is important as it disassociates BSMs from asphalt.

3.2 Binder content

This is another term that is associated with asphalt. It is preferable to talk about “added bitumen” for BSMs, mainly because, unlike asphalt, you cannot determine the “binder content” after the event.

3.3 Bitumen stabilised material (BSM)

Granular, previously cement treated or reclaimed asphalt material blends, stabilised either using bitumen emulsion or foamed bitumen.

3.4 Constant mass

Less than 0.1% change in mass after two successive (more than 4 hours) periods of oven-drying.

3.5 Expansion ratio

A measure of the viscosity of the foamed bitumen, calculated as the ratio of the maximum volume of the foam relative to the original volume of bitumen.

3.6 Half-Life

A measure of the stability of the foamed bitumen, calculated as the time taken in seconds for the foam to collapse to half its maximum volume.

4. APPARATUS

4.1 Electronic balance

Fine measurement type that complies with SANS 1649, with a capacity of 10 kg and reading to 0.1 g.

4.2 Foamed bitumen laboratory unit

Capable of producing foamed bitumen at a rate of between 50 g and 200 g per second. The method of producing the foamed bitumen shall closely simulate that used in full-scale production. The laboratory unit shall be equipped with a thermostatically controlled kettle, capable of holding a mass of 10 kg of bitumen at a constant temperature within the range of 160 and 190 ± 5 °C. The unit is to be equipped with a water injection system, where the mass of water injected into the hot bitumen can be variable from 0 to 5 ± 0.25%, by mass of the bitumen. To assist in achieving a uniform foam, the water is injected together with compressed air. The unit shall be capable of producing a predetermined mass of foamed bitumen directly into the container or laboratory mixer.

***NOTE 1*: A Wirtgen WLB10S is an example of a suitable foamed bitumen laboratory unit available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement of this product.**

4.3 Cylindrical metal container
With a diameter of 275 ± 5 mm and a capacity of 20 litres.

4.4 Calibrated dipstick
Metal, with prongs. The spacing between each of the 5 prongs indicates the expansion of a mass of 500 g of bitumen when foamed in the container.

4.5 Stopwatch
Reading to 1 second.

4.6 Safety equipment
Including:

a) safety glasses or face shield
b) protective gloves, well insulated and capable of withstanding 200 °C
c) long sleeved jacket

4.7 Sealable steel containers
Capacity approximately 5 litres.

4.8 Drying ovens
Capable of maintaining a temperature range of 105 °C to 180 °C with continuous draft or by convection, for preheating the bitumen prior to testing. A second oven to preheat the cylindrical metal container to 75 °C.

4.9 Plastic beaker
Capacity 0.5 litres.

4.10 Glass measuring cylinder
Capacity 50 millilitres.

4.11 Suitable waste receptacle
Capacity 10 litres, for bitumen and foamed bitumen.

5. HAZARDS

Warning: *The temperature of the bitumen in the kettle is high, while the foamed bitumen will also cause injury. Pouring hot bitumen into the unit's kettle is also hazardous. Therefore, exercise caution at all times when preparing the unit and carrying out the testing as failure to do so could result in serious injury or severe burns.*

It is important to follow the foamed bitumen unit's manufacturer's instruction manual carefully and take all necessary precautions.

6. PRINCIPLES

The objective is to determine the percentage of water required to produce the best foaming characteristics of a particular source of bitumen at three (3) different bitumen temperatures. The aim is to produce foamed bitumen with the largest expansion ratio and with the longest possible half-life.

7. PREPARATION

7.1 Preparation of the bitumen

- 7.1.1 Obtain the sample of bitumen in accordance with TMH 5, sealing it in the containers. Sample a minimum of 10 litres for determining the bitumen's foam characteristics.
- 7.1.2 Heat the bitumen in the oven to a temperature of 100 °C.

7.2 Preparation of the laboratory unit

- 7.2.1 Connect the unit to the electrical power supply and switch on.
- 7.2.2 Set the temperature control on the foamed bitumen laboratory unit to heat the kettle to the predetermined temperature (usually between 160 °C and 180 °C).

<u>NOTE 2</u>: The temperature set to produce the foamed bitumen depends upon the grade of bitumen, as well as for an investigation of the effect of bitumen temperature on foaming characteristics.

- 7.2.3 Remove the containers with the bitumen from the oven and pour 10 litres into the kettle.
- 7.2.4 Once the temperature of the bitumen in the kettle is above 140 °C, start the bitumen pump to circulate it through the system and continue circulating whilst heating to achieve the required temperature.
- 7.2.5 Fill the unit's water reservoir with potable water.
- 7.2.6 Weigh the 20 litre cylindrical metal container on the balance (M_1) and place it under the foamed bitumen outlet.
- 7.2.7 Set the bitumen discharge rate using the manufacturer's instructions, typically 100 g per second for 5 seconds.
- 7.2.8 Discharge bitumen for the pre-set time period into the container and weigh it again (M_2). Determine that the mass of bitumen discharged is 500 g ± 10 g. Empty the container into the waste receptacle. If the mass of discharged bitumen in the container falls outside this range, repeat the procedure, adjusting the timer until the discharge rate complies.
- 7.2.9 Set the injection rate with the water flow meter using the manufacturer's instructions. Discharge water for the pre-set period into the beaker. Transfer the water into the measuring cylinder and read the volume of water discharged. Determine that the volume of water is within 5% of the prescribed volume. If the volume of water falls outside this range, repeat the procedure, adjusting the water flow meter until the discharge rate complies.
- 7.2.10 Ensure that the dip stick is clean and the 20 litre cylindrical metal container is empty. Pre-heat the container in the oven for at least 5 minutes at 75 °C.

8. PROCEDURE

8.1 Circulate the bitumen in the kettle through the system at 160 °C for at least 5 minutes prior to testing.

8.2 Check that the correct settings have been made for the timer for the foamed bitumen discharge as well as the flow meter for the water injection.

<u>NOTE 3</u>: The water flow meter is normally set to inject 2% of water by mass of bitumen for the first testing cycle.

8.3 Discharge foamed bitumen into the pre-heated container for the time required to spray 500 g of bitumen.

8.4 Start the stopwatch immediately the foamed bitumen discharge starts.

8.5 Use the dipstick to estimate (to the nearest whole number) the maximum height that the foamed bitumen reaches in the container and record it as the maximum Expansion Ratio (ER = h_1).

8.6 Continue to measure the time that the foam takes to dissipate to half of its maximum volume in the container. When this point is reached, stop the stopwatch. Record the lapsed time to the nearest second as the foamed bitumen's half-life ($\tau_{½\,1}$). Decant the foamed bitumen into the waste receptacle.

8.7 Repeat 8.3 to 8.6 at least three times until similar results are obtained (h_2, $\tau_{½\,2}$; h_3, $\tau_{½\,3}$).

8.8 Calculate and record the average values for the three most similar pairs of results for expansion ratio and half-life.

8.9 Determine the expansion ratio and half-life at other percentages of water, following steps 8.3 to 8.7. Typically, these values are determined by injecting 1%, 2%, 3%, and 4% of water by mass of bitumen.

8.10 Repeat 8.1 to 8.9 with the bitumen at two higher temperatures.

<u>NOTE 4</u>: i. Typically, the two higher temperatures used for testing are 170 and 18 °C.

ii. Under no circumstance should the bitumen be heated above 190°C.

9. CALCULATIONS

9.1 Calculate the mass of bitumen discharged in 5 seconds. See Paragraph 7.2.7.

$$M_{bit} = (M_2 - M_1)$$

where:

M_{bit} *mass of bitumen discharged in 5 seconds*
M_1 *mass of empty cylindrical metal container, expressed in grams (g)*
M_2 *mass of container and bitumen, expressed in grams (g)*

9.2 Prepare a chart to plot the expansion ratio and half-life at the different percentages of water injected into the bitumen, in the following way:

<u>Horizontal axis:</u> Rate of injected water
Unit: Percentage (%)
Scale: 20 mm = 0.5%

<u>Vertical axis</u>:

Left Ordinate:	Expansion Ratio	Right Ordinate:	Half-life
Unit:	Times expansion	Unit:	seconds
Scale:	5 mm = 1 time	Scale:	5 mm = 2 seconds

9.3 Plot the Expansion Ratio obtained at each rate of injected water and join the points together.

9.4 Repeat 9.3 for Half-life.

9.5 Select the Optimum Water Injection Rate as the average of the two water contents required to meet the minimum requirements for Expansion Ratio and Half-life, respectively (see Chapter 4). An example of the chart is shown in Figure B1.

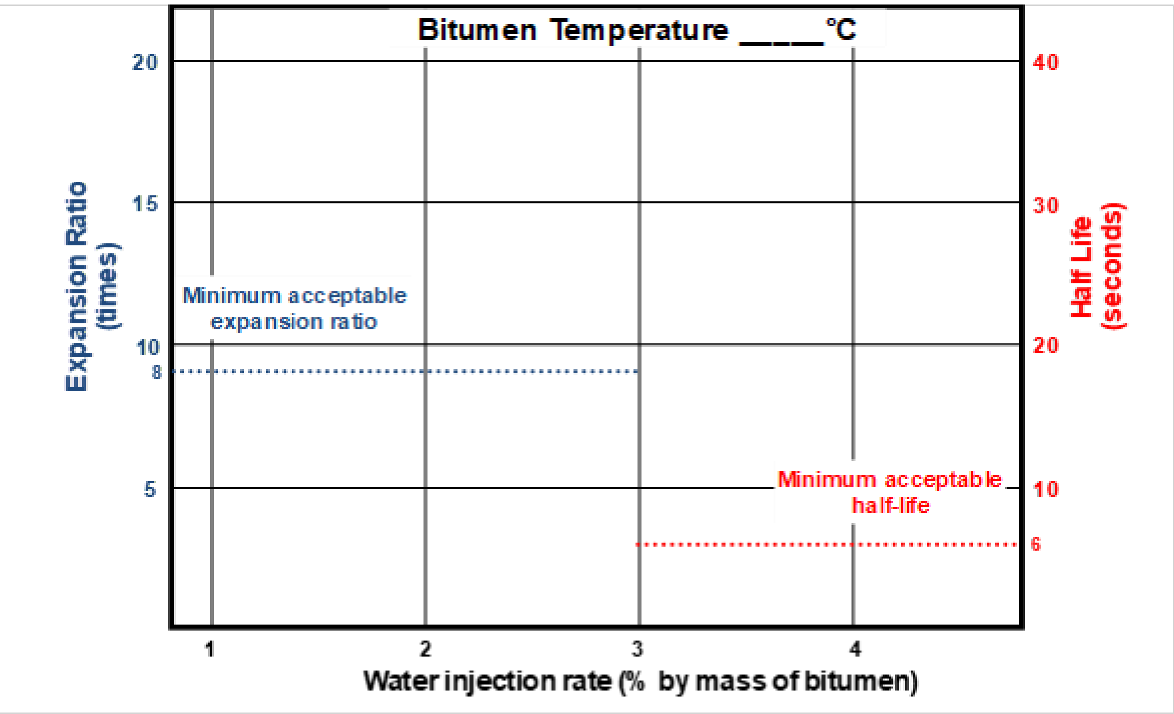


Figure B.1 Chart to Determine Optimum Water Injection Rate

10. TEST REPORT

Report the Optimum Water Injection Rate and the measured foam characteristics on a suitable form, including details of the bitumen being foamed. An example of such a form is shown below.

<table>
<tr><td colspan="10">Influence of Water Injection Rate on the Expansion Ratio and Half-life</td></tr>
<tr><td colspan="2">Date</td><td colspan="3"></td><td colspan="2">Bitumen type</td><td colspan="3"></td></tr>
<tr><td colspan="2">Tested by</td><td colspan="3"></td><td colspan="2">Injection time</td><td colspan="3">(sec)</td></tr>
<tr><td colspan="2"></td><td colspan="3">Bitumen</td><td colspan="2">Water</td><td colspan="3">Air</td></tr>
<tr><td colspan="2">Temperature (°C)</td><td colspan="3"></td><td colspan="2"></td><td colspan="3"></td></tr>
<tr><td colspan="2">Pressure (bar)</td><td colspan="3"></td><td colspan="2"></td><td colspan="3"></td></tr>
<tr><td colspan="2">Water Injection</td><td colspan="2">1st measurement</td><td colspan="2">2nd measurement</td><td colspan="2">3rd measurement</td><td colspan="2">Average</td></tr>
<tr><td>Injection (%)</td><td>Flow (l/hr)</td><td>ER</td><td>$\tau_{½}$</td><td>ER</td><td>$\tau_{½}$</td><td>ER</td><td>$\tau_{½}$</td><td>ER</td><td>$\tau_{½}$</td></tr>
<tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td><td></td></tr>
<tr><td colspan="2">Flow rate</td><td colspan="3">(g/sec)</td><td colspan="2">As shown below</td><td colspan="3">On / Off</td></tr>
</table>

ANNEXURE A: Calculation Procedures

A.1 Calculate the Mass of Bitumen Discharged in 5 seconds (see Paragraph 9.1)

Use the information below to calculate the mass of bitumen discharged:

$$M_{bit} = (M_2 - M_1) = (696 - 194) = 502\ g$$

Where:

M_{bit} = *mass of the bitumen discharged in 5 seconds*
M_1 = *mass of the 20 litre container (194 g)*
M_2 = *mass of the container and bitumen (696 g)*

A.2 Determination of the Optimum Water Injection Rate

The following example explains how the graph is compiled from measurements of the foaming characteristics. Appropriate minimum acceptable values for the Expansion Ratio and Half-life are employed (explained in Chapter 4) to determine the Optimum Water Injection Rate for a specific bitumen temperature.

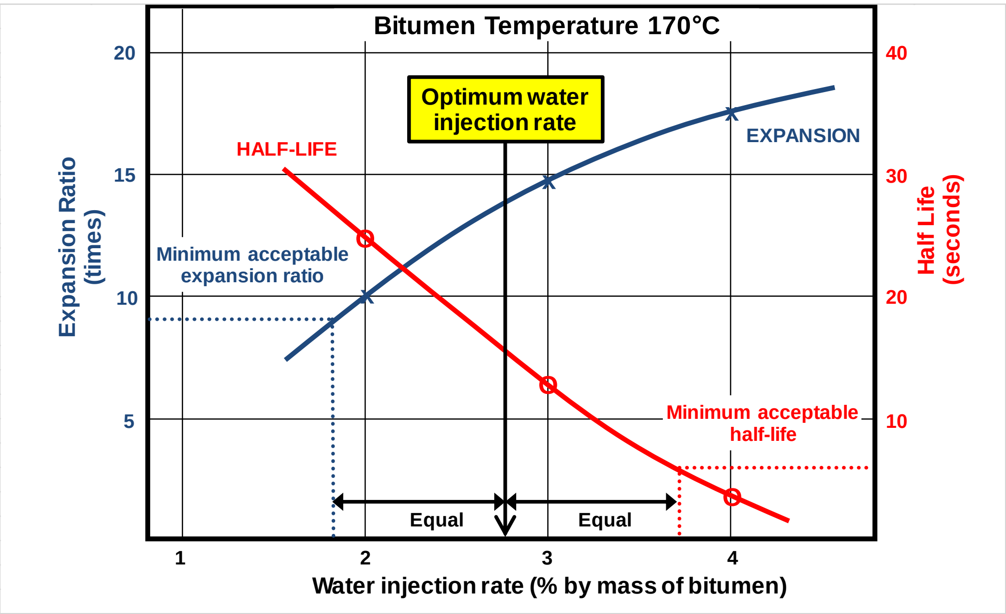
Assume that foamed bitumen is to be used as a stabilising agent on a project in a warm climatic region and the bitumen to be foamed is a standard 70/100 Pen Grade. Chapter 4 provides the minimum acceptable Expansion Ratio and Half-life values of 8 time and 6 seconds respectively.

The figures in the table below are typical measurements taken in the laboratory for three water injection rates with the bitumen at a temperature of 170 °C.

Table B.1 Influence of Water Injection Rate on the Expansion Ratio and Half-Life

<table>
<tr><td colspan="2">Date</td><td colspan="3">28/02/2019</td><td colspan="3">Bitumen type</td><td colspan="2">70 / 100 Pen grade</td></tr>
<tr><td colspan="2">Tested by</td><td colspan="3">PvW</td><td colspan="3">Injection time</td><td colspan="2">5 (sec)</td></tr>
<tr><td colspan="2"></td><td colspan="3">Bitumen</td><td colspan="3">Water</td><td colspan="2">Air</td></tr>
<tr><td colspan="2">Temperature (°C)</td><td colspan="3">170</td><td colspan="3">23</td><td colspan="2">24</td></tr>
<tr><td colspan="2">Pressure (bar)</td><td colspan="3">3</td><td colspan="3">4</td><td colspan="2">4</td></tr>
<tr><td colspan="2">Flow rate</td><td colspan="3">100 (g/sec)</td><td colspan="3">As shown below</td><td colspan="2">On / Off</td></tr>
<tr><td colspan="2">Water Injection</td><td colspan="2">1st measurement</td><td colspan="2">2nd measurement</td><td colspan="2">3rd measurement</td><td colspan="2">Average</td></tr>
<tr><td>Injection (%)</td><td>Flow (l/hr)</td><td>ER</td><td>$\tau_{1/2}$</td><td>ER</td><td>t</td><td>ER</td><td>$\tau_{1/2}$</td><td>ER</td><td>$\tau_{1/2}$</td></tr>
<tr><td>2</td><td>7.2</td><td>12</td><td>26</td><td>10</td><td>22</td><td>8</td><td>25</td><td>10</td><td>24.5</td></tr>
<tr><td>3</td><td>10.5</td><td>12</td><td>11</td><td>15</td><td>12</td><td>16</td><td>15</td><td>14.5</td><td>12.5</td></tr>
<tr><td>4</td><td>14.4</td><td>18</td><td>3</td><td>16</td><td>5</td><td>17</td><td>4</td><td>17</td><td>4</td></tr>
</table>

The average values for Expansion Ratio and Half-life for each of the three water injection rates are then plotted on the graph, together with the minimum acceptable foaming characteristics.



The Optimum Water Injection Rate is determined as follows:

i. Determine the water injection rate for where the minimum acceptable Expansion Ratio and Half-life lines intersect the relevant curve:

a)	*Water injection for minimum acceptable expansion:*	*1.9%*
b)	*Water injection for minimum acceptable half-life:*	*3.7%*

ii. The Optimum Water Injection Rate is the water injection rate that falls mid-way between the above two values:

The difference between the two minimum water injections rates:
3.7 – 1.9 = 1.8%
Divide the difference in two:
1.8 ÷ 2 = 0.9%
The Optimum Water Injection rate is:
1.9 + 0.9 = 2.8%

RESULT: The Optimum Water Injection Rate for bitumen at 170 °C is 2.8% (by mass of the bitumen)

BSM2: LABORATORY MIX DESIGN OF BITUMEN STABILISED MATERIAL

1. SCOPE

This test method describes a mix design procedure for laboratory preparation, mixing, compacting, curing and testing of bitumen stabilised material using either foamed bitumen or bitumen emulsion.

2. REFERENCES

The following referenced documents are indispensable for the application of this method. All normative documents are subject to revision and, since any reference to a normative document is deemed to be a reference to the latest edition of that document. Parties to agreements based on this document are encouraged to take steps to ensure the use of the most recent editions of the normative documents indicated below. Information on currently valid national and international standards can be obtained from Standards South Africa.

- SANS 1649, Non-automatic self-indicating, semi-self-indicating and non-self-indicating weighing instruments with denominated verification scale intervals.
- SANS 3001-GR1, Civil engineering test methods Part GR1: Wet preparation and particle size analysis.
- SANS 3001-GR10, Civil engineering test methods Part GR2: Determination of the one-point liquid limit, plastic limit, plasticity index and linear shrinkage.
- SANS 3001-GR20, Civil engineering test methods Part GR20: Determination of the moisture content by oven-drying.
- SANS 3001-GR30, Civil engineering test methods Part GR30: Determination of the maximum dry density and optimum moisture content.
- TG2, Test Method BSM1, Determination of the foaming characteristics of bitumen.
- TG2, Test Method BSM3, Vibratory hammer compaction for test specimens of bitumen stabilised material.
- TG2, Test Method BSM4, Determination of the indirect tensile strength of bitumen stabilised material.
- TG2, Test Method BSM5, Determination of the shear properties of bitumen stabilised material (Triaxial test)
- TMH 5, Sampling methods for road construction materials.

3. DEFINITIONS

3.1 Bitumen stabilised material (BSM)
Granular, previously cement treated or reclaimed asphalt material blends, stabilised either using bitumen emulsion or foamed bitumen.

3.2 Foamed bitumen
Vaporizing cold water in hot bitumen to create foam.

3.3 Optimum fluid content (OFC)
The percentage (by mass) of the combination of bitumen emulsion and water required to achieve the maximum dry density (MDD) of material treated with bitumen emulsion.

3.4 Raw
Untreated material (gravel, crushed stone or reclaimed asphalt).

4. APPARATUS

4.1 Foamed bitumen laboratory unit
Capable of producing foamed bitumen at a consistent rate of between 50 g/sec and 200 g/sec, equipped with a thermostatically controlled kettle of capacity 10 kg of bitumen, operating at a constant temperature ± 5 °C within a range of 160 °C and 190 °C. The unit is to be equipped with an expansion chamber (similar to that used in full-scale production) into which water and hot bitumen is injected under pressure to create the foamed bitumen. The unit is to be capable of injecting compressed air and a set amount of water into the hot bitumen to produce a uniform foam. The amount of water added by mass is to be up to 5% of the mass

of the bitumen and added at a controlled amount ± 0.25%. The unit shall be capable of producing a mass of foamed bitumen within 5 g of the prescribed amount, directly into a container or laboratory mixer. The unit is coupled directly to the pug mill mixer and is to inject the foamed bitumen directly into the running mixer uniformly across the full length of the mixer (between the two paddle shafts) by means of a fan jet fitted to the discharge end of the expansion chamber.

4.2 Pug mill mixer

Capacity 30 kg, with twin shafts and variable rotation speed control, graduated from a minimum to a maximum rotation speed of 150 rpm.

***NOTE 1*: Wirtgen WLB10S and WLM30 are examples of suitable foamed bitumen and pug mill units available commercially. This information is given for the convenience of users of this standard and does not constitute an endorsement of this product.**

4.3 Steel tamper or small laboratory crusher

A small, laboratory-scale jaw crusher that is capable of resizing aggregates to minus 20 mm is recommended

4.4 Electronic balances

Fine measurement type complying with SANS 1649. One with a capacity of 15 kg reading to 5 g and one with a capacity of 2 kg reading to 0.1 g.

4.5 Suitable basin

About 500 mm diameter for mixing.

4.6 Containers

Mixing basin (or bath) 200 litres capacity.

Airtight containers of capacity:
a) 20 litres for stabilised material.
b) 1 litre for moisture content determination.

4.7 Sieves

Comply with SANS 3310-2, that comprise a 450 mm diameter sieve set with pan, which consists of sieves with nominal aperture sizes: 20 mm, 14 mm and 5 mm.

4.8 Litmus paper

5. PRINCIPLES

The mix design procedure requires step-wise and iterative ITS testing to select the most effective active filler and the optimum amount of bitumen stabilising agent, followed by triaxial testing to determine the shear properties of the treated material. At various stages the mix designer has to make selections based on the outcome of ITS tests, so that the next step can be carried out. The procedure is outlined in Table B.2.

Table B.2. Mix Design Procedure

Stage	Step	Action by	Description of Action Required
Preliminary	1	Laboratory	Carry out preliminary tests to determine the moisture content, grading and plasticity of the field sample.
	2	Designer Input	Approve material or specify material and blending proportions to adjust grading and plasticity.
	3	Designer Input	Select type of bitumen stabilising agent – foamed bitumen or bitumen emulsion.
	4	Laboratory	Proportion the material and reduce to minus 20 mm maximum size.
	5	Laboratory	Test the blended material to determine the MDD and OMC.
	6	Laboratory	Test the bitumen stabilising agent to determine its properties. Evaluate foamed bitumen characteristics or obtain bitumen emulsion certification.
Effect of active filler	7	Laboratory	Mix 3 samples with the same amount of bitumen; one with 1% lime, one with 1% cement and one with no active filler.
	8	Laboratory	Compact specimens, cure and ITS test to determine the wet and dry values.
	9	Designer Input	Select the type of active filler.
Optimum bitumen application rate	10	Laboratory	Mix 3 further samples with the active filler (step 8) and at 3 different bitumen application rates in 0.2% intervals either side of previous mix (step 6) i.e. 2 mixes lower + 1 higher
	11	Laboratory	Compact specimens, cure and ITS test to determine the wet and dry values.
	12	Designer Input	Select optimum bitumen application rate.
Triaxial test	13	Laboratory	Mix samples with the active filler (selected in step 8) and bitumen at the optimum application rate (selected in step 11).
	14	Laboratory	Compact specimens, cure and perform Triaxial testing to determine the shear properties.
Outcome	15	Laboratory	Report ITS and Triaxial results for the mix design.

6. PREPARATION OF SAMPLES AND PRELIMINARY TESTING

6.1 General

During the preparation procedure, input from the BSM mix designer is required to specify approval or modification of the raw material (by blending) and the type of bitumen stabilising agent to be used. The preparation procedure is dependent on whether bitumen emulsion or foamed bitumen is to be used as the bituminous stabilising agent. The paragraphs dealing with the other stabilising agent should be ignored and the specified design inputs should be clearly indicated on the test report.

Sample the field material to be stabilised in accordance with TMH5. A large sample of at least 500 kg for each mix design is recommended. This quantity enables preliminary testing to be carried out and for fractions of material to be discarded during the preparation stage.

6.2 Test sample sizes

The size of the test samples will depend on the extent of the testing programme envisaged. Table B.3 provides a guideline for the quantity of air-dried material that will be required to manufacture test specimens for a normal BSM design.

Table B.3 Guideline for Sample Size Requirements for BSM Mix Design

Testing Phase	**Specimen size (mm)**	**Number of specimens**	**Maximum mass of each specimen (kg)**	**Number of mixes (kg /mix)**	**Total mass required (kg)**
Preliminary tests	-	-	-	-	100
Effect of active filler	150 ϕ x 95	6 x 3 = 18	4.5	3 (26) *	60
Optimum bitumen application rate	150 ϕ x 95	6 x 3 = 18	4.5	3 (26) *	60
Shear properties	150 ϕ x 300	10	15	5 (30)	150
Allowance for repeat testing	-	-	-	-	80
				Total	450

NOTE: 2: **20 kg is the minimum mass for effective mixing in a pug mill mixer**

6.3 Preliminary tests

6.3.1 Mix the field sample and take out a representative sample for preliminary testing.

6.3.2 Carry out the moisture content, grading analysis and Atterberg Limits tests as listed in Table B.4, on representative specimens taken from the preliminary testing sample.

Table B.4 List of Preliminary Tests

Test	**Standard**
Determination of the moisture content (W_M)	SANS 3001-GR20
Grading analysis (Wet preparation and particle size analysis)	SANS 3001-GR1
Atterberg Limits (Determination of the one-point liquid limit, plastic limit, plasticity index and linear shrinkage)	SANS 3001-GR10
Determination of maximum dry density and optimum moisture (or fluid) content *	SANS 3001-GR30 or Annexure A *

***NOTE 3:** The test method for determination of dry density / optimum moisture (or fluid) content will depend on the type of stabilising agent used (foamed bitumen or bitumen emulsion)*

6.3.3 Report the results of the tests carried out in 6.3.2 to the mix designer and obtain instructions as to the type of bitumen stabilising agent to be used (foamed bitumen or bitumen emulsion), whether the raw material is suitable or requires blending and, if so, the type of material to be added and the proportions.

6.3.4 Depending on the instructions in 6.3.3, carry out the material blending specified or use the raw material as is, and test the material for maximum dry density and optimum moisture or fluid content (see Table B.4). When the raw material has been modified by blending additional material, carry out a further grading analysis as described in Table B.5.

6.3.5 Depending on the type of bitumen stabilising agent to be used (see 6.3.3) either determine the foaming characteristics of the bitumen as described in Test Method BSM1 or test the emulsion as described in 6.3.6.

NOTE 4**: Bitumen of minimum 70 Pen is required to ensure that the bitumen is suitable for foaming.**

6.3.6 When bitumen emulsion is specified, check the manufacturer's certificate for type, bitumen content and date of manufacture of the emulsion. Only stable grade emulsion is suitable for bitumen stabilisation. Where such certification is not available, carry out the following tests:

6.3.6.1 Check the charge on the bitumen emulsion using a strip of litmus paper. When the paper turns red the emulsion is cationic (with a pH < 7). When the paper turns blue the emulsion is anionic (with a pH > 7).

6.3.6.2 Check the stability of the emulsion by adding approximately 83 g of emulsion to a cement paste (i.e. 50 g of ordinary Portland cement + 17 g water premixed with a spatula) and mix in a tin by stirring vigorously with a spatula. The outcomes are evaluated as follows:

- If the mix remains smooth after 60 seconds of mixing, then you have a Slow Set (Stable Grade) emulsion
- If the mix breaks and coagulates quickly (<20 secs of mixing) you have a Rapid Set (Spray Grade) emulsion
- Intermediate break time and some coagulation would most likely indicate a Medium Set (Premix Grade) emulsion

6.3.6.3 Check the compatibility of the emulsion with the proposed mixing water by adding 50 millilitres of water to 50 millilitres of emulsion and gently turning the cylinder upside-down several times whilst sealing the top by hand. Allow the fluid mixture to stand for 30 minutes. If any bitumen separation or coarsening of the fluid is observed there is a compatibility problem and another source of water should be used.

6.4 Sample Proportioning

6.4.1 From the grading analysis determined in Paragraphs 6.3.2 or 6.3.4, calculate the proportions retained, expressed as a percentage of the total sample, as follows:

A = percentage retained on the 20 mm sieve
B = percentage retained on the 14 mm sieve
C = percentage retained on the 5 mm sieve
D = percentage passing the 5 mm sieve

6.4.2 Air-dry the field sample sufficiently so that the material can be sieved into separate fractions.

6.4.3 Sieve the sample through the 20 mm, 14 mm and 5 mm sieves, in order of decreasing size, into a pan.

6.4.4 Crush lightly the material retained on the 20 mm sieve so that it just passes the 20 mm sieve. Sieve the crushed material through the 14 mm sieve and add the material retained to the minus 20 mm plus 14 mm fraction. Discard the fraction passing the 14 mm sieve that was generated by crushing.

6.4.5 Using the proportions given in Table B.5 reconstitute the fractions (see 6.4.4) to provide samples for ITS and Triaxial tests, and store in sealed containers. An example of the procedure is given in Annexure B.

NOTE 5**: This procedure allows for different sizes of samples to be produced with a similar grading**

Table B.5 Representative Proportions by Mass to be used for Test Sample Preparation

Fraction	Percentage of Total Mass
< 20 mm to ≥ 14 mm	A + B
< 14 mm to ≥ 5 mm	C
< 5 mm	D

7. MIX DESIGN

7.1 General

The following paragraphs describe the iterative mix design procedure to determine the active filler type and the optimum bitumen application rate.

NOTE 6: ***The residual bitumen content for emulsion is assumed to be 60% in the following sections.***

Table B.6 Guidelines for Estimating Optimum Bitumen Addition

Fraction < 0.075mm (%)	Bitumen addition (% m/m dry aggregate) Fraction < 4.75mm		Typical Material
	< 50%	> 50%	
< 4	1.8	2.0	Recycled asphalt (RA)
4 – 7	2.1	2.3	RA Graded Crushed Stone Natural Gravel Blends
7 – 10	2.4	2.6	
> 10	2.6	2.9	Gravels Sands

7.2 Active filler selection

7.2.1 Mix the bulk sample prepared in Paragraph 6.4.5 and riffle out sufficient material for 3 batches of ITS tests (about 78 kg, see Table B.3).

7.2.2 Use Table B.6 to select an estimated optimum (residual) bitumen content for either foamed bitumen or emulsion. For example, the grading yields 55% < 4.75 mm and 6% < 0.075 mm, so 2.3% of bitumen addition is required. Mix each batch of material: one without active filler, one with 1% cement and one with 1% lime.

7.2.3 Because pug mill mixers require a minimum mass for effective mixing, mix each of the three batches using the minimum mass of material recommended by the manufacturer of the pug mill (26 kg for the Wirtgen WLM 30).

7.2.3.1 Measure out 26 kg from the bulk sample material, place in the pug mill mixer and spread uniformly along the length of the mixer.

7.2.3.2 Measure out the required amount of active filler as calculated in Paragraph 9.1.2. Sprinkle the active filler uniformly on top of the spread sample material. Close the lid of the mixer and mix for 30 seconds with the pug mill set at the medium rotation speed. Stop the mixer and leave it to stand for at least 2 minutes to allow the dust to settle.

7.2.3.3 Following the procedure described in Paragraphs 8.2 (bitumen emulsion) or 8.3 (foamed bitumen), add and mix the required amount of water and bitumen stabilising agent to the material in the mixer.

7.2.3.4 Measure the temperature of the material to the nearest 1 °C.

7.2.4 Compact, cure and ITS test the specimens (3 dry and 3 soaked per batch) as described in Test Methods BSM3 and BSM4.

7.2.5 Report the ITS values to the mix designer and obtain instructions regarding the type of active filler to be used. In special circumstances, the mix designer may require an active filler content other than 1%.

7.3 Optimum bitumen application rate

7.3.1 As described in 7.2.1, prepare sufficient material for 3 batches of ITS tests (about 78 kg, see Table B.3).

7.3.2 As described in 7.2.3, mix the required amount and type of active filler specified by the mix designer (see 7.2.5) to the sample.

7.3.3 As described in 8.2 or 8.3, prepare and mix each of the three batches with the bitumen application rate recommended in Table B.6. With experience, and depending on the ITS values obtained in 7.2, other ranges of bitumen application rates may be used. However, small increments i.e. less than 0.2%, are not recommended.

***NOTE 7*: The intention of Table B.6 is to bracket the optimum bitumen application rate. In the case where the raw material is of high quality, such as reclaimed asphalt and crushed stone base, the application rates may all be reduced to below the 2.3% used to determine the effect of active filler (Paragraph 7.2).**

Table B.7 Selection of Bitumen Application Rates

Bitumen Stabilising Agent type	Bitumen Application Rate per Batch			
	Batch 1	Batch 2	Already tested	Batch 3
Emulsion	3.2% (1.9% RBC)	3.5% (2.1% RBC)	3.8% (2.3% RBC)	4.2% (2.5% RBC)
Foam	1.9% BC	2.1%	2.3%	2.5%
BC = Bitumen content. RBC = Residual bitumen content. Active Filler for all batches determined in Section 7.2.				

7.3.4 Compact, cure and ITS test the specimens manufactured from each batch (3 dry and 3 soaked) as described in Test Methods BSM3 and BSM4.

7.3.5 Plot and report the ITS_{DRY} and ITS_{WET} values against the respective bitumen application rate (including the values obtained in 7.2.6 for determining the effect of active filler) to the mix designer and obtain instructions regarding the optimum bitumen application rate to be used.

7.4 Triaxial test specimens

7.4.1 Mix the bulk sample prepared in 6.4.5 and riffle out sufficient material for 5 batches required for triaxial testing (about 150 kg, see Table B.3).

7.4.2 Mix the required bitumen application rate and type of active filler specified by the mix designer (see 7.2.5) into each of the batches.

7.4.3 Transfer the mixed material from each batch into a suitable container and cover with a moist hessian cloth to prevent moisture loss.

7.4.4 Thoroughly mix the material in the container before compacting, curing and testing the specimens, as described in Test Methods BSM3 and BSM5.

8. MIXING

8.1 General

Because pug mill mixers require sufficient material to be placed in the mixer for effective mixing, it is recommended that not less than 20 kg is used for any one mix or batch.

The mixing procedure described assumes that the mixing operation will be followed directly by compaction, curing and testing of each set of specimens. Depending on the capabilities of the laboratory carrying out the testing this approach may be varied, providing that during any pauses permitted in the procedure, the material is stored in clearly marked air-tight containers. The type of bitumen stabilising agent is specified by the **mix designer** in Paragraph 6.3.3. Ignore references to the other bitumen stabilising agent in the following paragraphs.

8.2 Bitumen emulsion stabilisation

8.2.1 Spread out the material in the pug mill mixer uniformly along the length of the mixer.

***NOTE 8**: The raw material was measured out and the active filler mixed in as described in Section 7.2.3, 7.3.2 and 7.4.2.*

8.2.2 Ensure that the temperature of the mixing water is 25 °C ± 1.5 °C. Measure out the amount of water that needs to be added (V_W) to achieve the required OFC (see 9.1.4 for calculations and Annexure A for OFC determination).

8.2.3 Add the water to the material by sprinkling uniformly over the surface of the material in the mixer. Mix for 30 seconds at the maximum speed of rotation.

8.2.4 Ensure that the temperature of the bitumen emulsion is 60 °C ± 2.0 °C. Where no heating mechanism is available discretion may be exercised to mix at 25 °C (minimum). Measure out the required amount of bitumen emulsion that needs to be added (see 9.1.2 for calculation). Care should be taken to clearly differentiate between the amount of emulsion and the amount of bitumen in the emulsion. Emulsion of the same type and bitumen content must be used throughout the procedure. To avoid confusion, check that calculations show correctly the amounts of water and emulsion.

***NOTE 9**: For example, 2% SS60 emulsion contains 1.2 % residual bitumen.*

8.2.5 Remove the cover of the mixer and add the bitumen emulsion to the material by sprinkling uniformly over the surface of the material in the mixer. Mix for 30 seconds at the maximum speed of rotation i.e. 144 rpm.

8.2.6 Remove the sample from the mixer, measure the temperature of the mixed material to the nearest 1 °C and place in a marked sealed container.

***NOTE 10**: The aggregate temperature should be measured before and after mixing.*

8.2.7 Manufacture the test specimens within 30 minutes of mixing, as described in Test Method BSM3.

8.3 Foamed bitumen stabilisation

8.3.1 Spread out the material in the pug mill mixer uniformly along the length of the mixer.

***NOTE 11**: The raw material was measured out and the active filler mixed in as described in Paragraphs 7.2.3, 7.3.2 and 7.4.2*.

8.3.1 Ensure that the temperature of the mixing water is 25 ± 1.5 °C. Measure out the amount of water that needs to be added to achieve 60% to 75% of OMC, where coarser materials require a lower percentage of OMC (see Paragraph 9.1.4 for calculation).

8.3.2 Add the water to the material in the pug mill mixer by sprinkling uniformly over the surface of the material in the mixer. Mix for 30 seconds at the maximum speed of rotation.

8.3.3 Couple the foamed bitumen laboratory unit to the mixer. Set the foamed bitumen laboratory unit to add air, bitumen and water at the prescribed rates to manufacture the foam. Add the required quantity of foamed bitumen (see 9.1.2) whilst the mixer is running. Continue to mix for a further 30 seconds at the maximum speed of rotation.

8.3.4 Remove the cover of the mixer and add additional water to bring the moisture content to 100% of OMC. Sprinkle the water uniformly over the surface of the material in the mixer. Mix for 30 seconds at the maximum speed of rotation.

8.3.5 Remove the sample material from the mixer, measure the temperature of the mixed material to the nearest 1 °C and place in a marked sealed container.

8.3.6 Manufacture the test specimens within 30 minutes of mixing, as described in Test Method BSM3.

9. CALCULATIONS

***NOTE 10**:An example of the calculation procedure is given in Annexure C.*

9.1 Calculations required in the procedure

9.1.1 Calculate the air-dried mass of material to the nearest gram, used in the mix using the following equation.

$$\mathrm{M_D} = \frac{\mathrm{M_{AD}}}{1 + \frac{\mathrm{W_M}}{100}}$$

Where:

M_D *oven-dried mass of material to be mixed, in grams (g)*

M_{AD} *air-dried mass of material to be mixed, in grams (g)*

W_M *moisture content of the air-dried material, as determined in SANS 3001-GR20, as a percentage of the oven-dried mass*

9.1.2 Calculate the quantity of required active filler or bitumen stabilising agent to be added to the nearest gram, using the following equation.

$$\mathrm{M_{Ai}} = \mathrm{P_A} \times \frac{\mathrm{M_D}}{100}$$

Where:

M_{Ai} *mass of active filler or bitumen stabilising agent to be added, in grams (g)*

P_{Ai} *specified percentage of active filler or bitumen stabilising agent to be added (%)*

M_D *mass of oven-dry material to be mixed, in grams (g)*

i *either F for filler or S for bitumen stabilising agent*

9.1.3 Calculate the quantity of water to the nearest millilitre, to be added to achieve the OFC when stabilising with bitumen emulsion using the following equations:

$$\mathrm{M_W} = \frac{\mathrm{OFC} \times (\mathrm{M_D} + \mathrm{M_{AF}})}{100} - (\mathrm{M_{AD}} - \mathrm{M_D}) - \mathrm{M_{AS}}$$

Where:

M_W *mass of water to be added, expressed in millilitres (ml)*

OFC *optimum fluid content, expressed as a percentage (%)*

M_{AF} *mass of active filler to be added, expressed in grams (g)*

M_{AS} *mass of bitumen stabilising agent to be added, expressed in grams (g)*

M_{AD} *mass of air-dried material to be mixed, expressed in grams (g)*

$M_W \approx V_W$ *based on the assumption that the density of water is approximately 1 kg/l*

V_W *volume of water to be added, expressed in millilitres (ml)*

9.1.4 Calculate the quantity of water to the nearest millilitre, to be added to achieve the OMC when stabilising with foamed bitumen, using the following equation.

$$\mathrm{M_W} = \frac{\mathrm{OMC} \times (\mathrm{M_D} + \mathrm{M_{AE}})}{100} - (\mathrm{M_{AD}} - \mathrm{M_D})$$

Where:

OMC *optimum moisture content of the material to be stabilised, as a percentage (%) (See Paragraph 8.3.2)*

$M_W \approx V_W$ *based on the assumption that the density of water is approximately 1 kg/l*

NOTE 12: ***75% of the total moisture mass is added before foaming and mixing. The remaining 25% is added after foaming (see Paragraphs 0 and 8.3.4).***

10. TEST REPORT

Report the following where appropriate on a suitable form:

a) Type and percentage of active filler and bitumen application rate required.
b) Initial moisture content of field sample.

c) Preliminary test results:

  i) Particle size analysis
  ii) Liquid limit, plastic limit and plasticity index
  iii) MDD and OMC (or OFC) before mixing

d) Bitumen stabilising agent:

  i) Bitumen emulsion: Source, type, bitumen content, stability and date of manufacture.
  ii) Foamed bitumen: Foaming characteristics determined in Appendix B, Test Method BSM1: *Determining the Foaming Characteristics of Bitumen*.

e) For each batch mixed:

  iii) Date and time of mixing
  iv) Mass of material mixed
  v) Moisture content after air-drying
  vi) Material temperature immediately before and after mixing
  vii) Percentage active filler and bitumen application rate

ANNEXURE A: Determination of the Maximum Dry Density (MDD) and Optimum Fluid Content (OFC) of a Material Treated with Bitumen Emulsion

A.1 Scope

The Optimum Fluid Content (OFC) of a bitumen emulsion stabilised material is the percentage (by mass) of the combination of bitumen emulsion and water required to achieve the maximum dry density (MDD) of the treated material. The MDD and OFC are determined following the same procedure for determining the MDD and optimum moisture content (OMC) of a material, substituting diluted bitumen emulsion for water to achieve the different moisture (fluid) contents.

A.2 Apparatus

This procedure uses the same apparatus as that used to determine the MDD and OMC of the material as described in SANS 3001-GR30. In addition, a 1 000 millilitre measuring cylinder is required.

A.3 Procedure

Follow the same procedure for determining the MDD and OMC as described in SANS 3001-GR30. In the place of water, a fluid consisting of a 50:50 blend of bitumen emulsion and water is used. Add and mix in the prescribed type and amount of active filler to the material to be tested.

To prepare the fluid pour 500 ml of the bitumen emulsion into a 1 000 ml measuring cylinder. Ensure that the bitumen emulsion is the same as that to be used for the bitumen stabilisation mix design. Slowly add 100 ml of clean water. Mix the bitumen emulsion with the water by gently turning the cylinder upside-down several times whilst sealing the top by hand. Continue diluting the bitumen emulsion in this manner by adding further units of 100 ml water until a 1 000 ml of blended fluid is achieved.

Using this diluted bitumen emulsion fluid, measure out the amount required for each specimen sample required in the SANS 3001-GR30 procedure.

ANNEXURE B: Sample Proportioning Procedure (see Paragraph 6.4)

B.1 Grading

The grading of the sample is shown in the table below.

Table B.8 Grading

Sieve Size (mm)	Passing (%)	Retained	
		Percent (%)	**Portion**
20	85	15	A
14	60	25	B
5	12	48	C
Pan	-	12	D

B.2 Crushing and proportioning

A 450 kg sample is sieved through a 20 mm, 14 mm and 5 mm sieve into a pan. The material retained on the 20 mm sieve is lightly crushed to just pass the 20 mm sieve and then added to the minus 20 mm plus 14 mm fraction. The table below gives an example of the proportioning of the sample to obtain a total mass of 80 kg (enough material for three sets of ITS tests).

Table B.9. Sample Proportioning

1	2	3	4	5	6	7
Sample		**Fraction mass after crushing (kg)**	**Amended fractions**		**80 kg sample**	
Fraction (mm)	**Portion**		**Amended portions as per Table B.8**	**Percentage of whole sample[2] %**	**Proportioning calculation**	**Masses required kg**
> 20	A[1]	-	-	-	-	-
< 20 to $\geq$ 14	B	177,5	A + B	15 + 25 = 40	$80 \times \frac{40}{100}$	32
< 14 to $\geq$ 5	C	218	C	48	$80 \times \frac{48}{100}$	38,4
< 5	D	54,5	D	12	$80 \times \frac{12}{100}$	9,6
TOTAL		450				80

1 Material lightly crushed to just pass 20 mm sieve.

2 From Table B.8, Column 3 (Percent Retained).

ANNEXURE C: Calculation Procedures

C.1 General

Use the following information to calculate C.2 to C.3:

M_{AD} = 29 561 g
W_M = 2.2%

P_{AF} = 1.0%

P_{AS} = 3.5% emulsion

OMC = 6.3%

C.2 Calculate the air-dried mass of material used in the mix (see paragraph 9.1.1)

$$M_D = \frac{M_{AD}}{1 + \frac{W_M}{100}} = \frac{29\,561}{1 + \frac{2.2}{100}} = 28\,925 \text{ g}$$

C.3 Calculate the quantity of required active filler and bitumen stabilising agent to be added (paragraph 9.1.2)

$$M_{AF} = P_{AF} \times \frac{M_D}{100} = 1{,}0 \times \frac{28\,925}{100} = 289 \text{ g}$$

and

$$M_{AS} = P_{AS} \times \frac{M_D}{100} = 3{,}5 \times \frac{28\,925}{100} = 1\,012 \text{ g}$$

C.4 Calculate the quantity of water to be added to achieve the OFC (paragraph 9.1.3)

$$M_W = \frac{\text{OFC} \times (M_D + M_{AF})}{100} - (M_{AD} - M_D) - M_{AS}$$

$$M_W = \frac{6.3 \times (28\,925 + 289)}{100} - (29\,561 - 28\,925) - 1\,012$$

$$M_W = 1840{,}428 - 636 - 1012 = 192 \text{ g} \approx 192 \text{ ml}$$

***NOTE 1**: OFC is determined through a sensitivity analysis of MDD versus moisture content at typical binder content. OFC is no longer automatically equated with OMC.*

BSM3: VIBRATORY HAMMER COMPACTION OF TEST SPECIMENS OF BITUMEN STABILISED MATERIAL

1. SCOPE

This test method describes the preparation and compaction of test specimens of bitumen stabilised material (BSM) using a vibratory hammer.

2. REFERENCES

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies. Information on currently valid national and international standards can be obtained from the SABS Standards Division.

- SANS 1649, Non-automatic self-indicating, semi-self-indicating and non-self-indicating weighing instruments with denominated verification scale intervals.
- SANS 3001-GR10, Civil engineering test methods Part GR2: Determination of the one-point liquid limit, plastic limit, plasticity index and linear shrinkage.
- SANS 3001-GR20, Civil engineering test methods Part GR20: Determination of the moisture content by oven-drying.
- SANS 3001-GR30, Civil engineering test methods Part GR30: Determination of the maximum dry density and optimum moisture content.
- TG2, Test Method BSM1, Determination of the foaming characteristics of bitumen.
- TG2, Test Method BSM2, Laboratory Mix Design of Bitumen Stabilised Materials.
- TG2, Test Method BSM4, Determination of the indirect tensile strength of bitumen stabilised material.
- TG2, Test Method BSM5, Determination of the shear properties of bitumen stabilised material (Triaxial test)
- TMH 5, Sampling methods for road construction materials. Pretoria.

3. DEFINITIONS

For the purpose of this document, the following definitions apply:

3.1 Bitumen Stabilised Material (BSM)
Granular, previously cement treated or reclaimed asphalt material blends, stabilised either using bitumen emulsion or foamed bitumen.

3.2 Indirect Tensile Strength (ITS)
The stress at failure generated by the load required to split a cylindrical specimen of bitumen stabilised material.

3.3 Maximum dry density (MDD)
The maximum dry density of the material determined from the peak of the dry density versus moisture content curve obtained as described in SANS 3001-GR30.

3.4 Optimum moisture content (OMC)
The moisture content at which the maximum dry density is achieved.

4. APPARATUS

4.1 Vibratory demolition hammer

Of mass 11.5 ± 0.1 kg with rated power input of 1700 W, an impact rate of 900 to 1 700 beats per minute and impact energy of 23 J ± 1 J per stroke.

Warning: *Prolonged exposure to the noise emitted by a demolition hammer can lead to impaired hearing. The operator should always wear ear plugs and, to protect others in the vicinity, the vibratory hammer compaction unit should be housed in a soundproof cabinet. Working beneath the suspended hammer assembly has the potential of causing bodily harm in the event of the locking device malfunctioning. Such locking device requires regular inspection and maintenance to ensure its functionality. In addition, exercise care when lowering the tamping foot into the mould to prevent damaging fingers or hands.*

NOTE 1:* *A Bosch Demolition Hammer, GSH 11VC Professional, is an example of a suitable product available commercially. This information is given for the convenience of the users of this standard and does not constitute an endorsement of this product.

4.2 Compaction shank

With corrosion resistant steel, with a tamping foot of diameter 145 ± 1 mm, of combined mass 3.0 ± 0.1 kg (see Figure B.2). The shank is machined with an SDS Max fitting to enable it to be fitted into the demolition hammer.

4.3 Compaction block

Concrete with a strength of approximately 25 MPa, that is at least 1 m x 1 m and 300 mm thick, reinforced with 2 layers of steel mesh (Ref 395), one placed 50 mm from the top of the block, the other 50 mm from the bottom. The surface of the block is cast level. The block has attachments such that the mould base plate can be securely and uniformly fixed to the block.

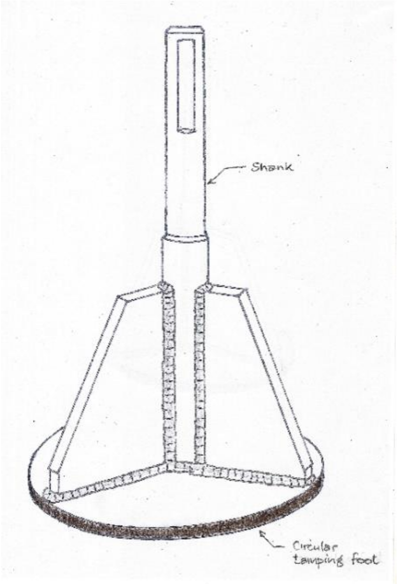


Figure B.2 Compaction Shank

4.4 Frame

With vertical slides, complete with mounting head for the vibratory hammer, that allows the vibratory hammer to be suspended above the mould with the tamping foot centred on the mould. The slides are to ensure free downward movement of the hammer as it compacts the material in the mould. The frame is fitted with a system (connected to the top of the frame) for lifting and lowering the vibratory hammer. The base of the frame is bolted to the compaction block.

4.5 Surcharge weight
Metal, attached to the vibratory hammer mount to achieve a total suspended mass of 33 ± 0.5 kg (including the compaction shank and foot).

***NOTE 2*: A Wirtgen WLV1 frame, is an example of a suitable product available commercially. This information is given for the convenience of the users of this standard and does not constitute an endorsement of this product.**

4.6 Split cylindrical moulds
Corrosion-resistant steel, with two segments:

a) With inside diameter 152 ± 0.5 mm and height of at least 120 mm; and base plate, for ITS test specimens
b) With inside diameter 150 ± 0.5 mm and height of at least 320 mm; and base plate, for triaxial test specimens.

***NOTE 3*: The base plate that fits the 152 mm diameter mould may be used for the 150 mm diameter mould.**

4.7 Interlayer roughening device (IRD)
Diameter 145 ± 1.0 mm, fitted with protruding teeth of length 12 ± 2.0 mm for roughening the upper surface of each compacted layer.

4.8 Carrier plates
Plywood of thickness approximately 15 mm and diameter approximately 160 mm, one for each compacted specimen.

4.9 Electronic balances
Fine measurement type that comply with SANS 1649:

a) With a capacity of at least 30 kg, and that reads to 1 g.
b) With a capacity of at least 10 kg, and that reads to 0.1 g.

4.10 Drying oven
Capable of maintaining a temperature range of 105 °C to 110 °C with continuous draft.

4.11 Containers
With lids that retain moisture, of capacity of at least

a) 2.5 litres for moisture content samples.
b) 20 litres for the mixed treated material.

4.12 Suitable basins
One of diameter approximately 300 mm, one of diameter at least 500 mm and one with a capacity of 200 litres.

4.13 Scoop
Of capacity approximately 500 millilitres.

4.14 Trowel, garden type

4.15 Timer
Capable of reading up to 5 minutes and reading to 1 second.

4.16 Vernier callipers
Measuring to 350 ± 1 mm.

4.17 Spatula

With steel blade, of length approximately 350 mm.

4.18 Marking materials consisting of

a) Marker pen
b) Oil based white paint for marking compacted specimens
c) Thin artist's brush

5. PREPARATION OF THE TEST SPECIMENS

5.1 Sample preparation

5.1.1 Preparation of BSM samples mixed in the laboratory as described in TG2 Test Method BSM2.

***NOTE 4**: The procedure in TG2 Test Method BSM2 requires the mix designer to specify the percentagesby mass of active filler and stabilizing agent to be added; and the type of bituminous stabilizing agent.*

5.1.2 Preparation of samples from field mixed BSM.

5.1.2.1 Sample the BSM in the field as described in TMH 5.

5.1.2.2 Obtain the maximum dry density (MDD) and optimum moisture content (OMC) from routine field control testing.

5.2 Determination of sample size

Use the data from Table B.10 and the formula in 7.1.2 to determine the amount of BSM required to prepare the test specimens.

Table B.10. Specimen Requirements

Requirements	ITS test	Triaxial test
Number of specimens per set	6	10
Mass of BSM required per set (kg)	26	150
Specimen height (mm)	95	300
Number of layers	2	5
Layer thickness (mm)	47,5	60

NOTE 5:

1. ***The number of layers may be increased to three in exceptional circumstances where de-bonding between layers occurs.***
2. ***If the required density cannot be achieved in any layer after compacting for 120 seconds with the vibratory hammer, stop compaction and increase the number of thinner layers to achieve the same overall height (e.g. increase the number of layers to 6, each 50mm thick).***

6. PROCEDURE

6.1 Equipment preparation

Ensure that the vibratory hammer frame is securely attached to the concrete support block and is installed vertically. Install the vibratory hammer and surcharge weight into the mounting frame and insert the tamping foot. Using the lifting system, raise and lower the hammer to ensure that there is no resistance to sliding.

Clean the mould and base plate. Lubricate the inside of the mould with a light application of lubricating grease or non-stick spray. Fix the mould and base plate to the concrete block. Check the alignment of the mould and vibratory hammer by lowering the tamping foot into the mould. Check that the lifting system provides sufficient slack for the tamping foot to rest on the base plate.

6.2 Compaction

6.2.1 Lower the vibratory hammer so that the tamping footrests on the base plate. Using a marking pen, mark the location of the slide on the frame. Raise the vibratory hammer using the lifting system and secure it at least 500 mm above the mould. Using a steel ruler, measure upwards from the mark on the frame and accurately mark the distance to the top of the first compacted layer (47.5 mm for ITS specimens and 60 mm for triaxial specimens); and subsequent layers (95 mm for ITS specimens; 120 mm, 180 mm, 240 mm, 300 mm for triaxial specimens).

When the vibratory hammer frame is fitted with an electronic system for controlling the compacted thickness of the layers, carefully follow the set up and operating procedures specified by the manufacturer.

6.2.2 Determine the mass of BSM required for each layer using the formula in 7.1.3.

6.2.3 From the sample prepared in the laboratory (see 5.1.1) or from the field mixed sample (see 5.1 2) measure out the mass of the BSM required for the first layer (accurate to ± 1 g) and carefully pour it into the mould without any spillage. Use the spatula to spread the material evenly in the mould avoiding segregation. Then use the Interlayer Roughening Device (IRD) to level the material inside the mould. Failure to level the material can cause the tamping foot to break due to eccentric loading. Retain the remainder of the sample in the sealed container to prevent moisture loss.

6.2.4 Lower the vibratory hammer until the tamping foot rests on the material. Ensure that the lifting system is slack, allowing the hammer to slide downwards as the material compacts. Turn on the vibratory hammer and start the timer. Allow the hammer to run until the mark on the sliding frame for the first layer is reached. Immediately turn the hammer off and stop the timer. Record the time taken to compact the layer. Raise the hammer using the lifting system and secure it at least 500 mm above the mould.

When the compaction time for any layer exceeds 120 seconds, terminate the manufacturing procedure, increase the number of layers and start the procedure again from Paragraph 6.2.1. In the unlikely event of the problem persisting with an increased number of layers, terminate the manufacturing procedure and seal all the material in airtight containers. Repeat the moisture / density relationship test on a new sample of the untreated material to determine the correct values for the MDD and OMC (see 5.1.1 or 5.1.2). Then start the procedure again using the revised MDD as the target density.

6.2.5 Prepare the surface of the compacted layer inside the mould using the interlayer roughening device (IRD). Place the IRD on top of the compacted material and apply sufficient pressure so that the teeth fully penetrate into the material. Maintaining the applied pressure, turn the IRD through 90° and then back again at least four times to loosen the material at the top of the layer. Lift the IRD out of the mould and inspect the roughened surface. If the material is not visibly loose, repeat the procedure described above as many times as necessary. When the surface has been sufficiently roughened, proceed immediately with the next layer.

6.2.6 Compact the second and any subsequent layers as described in 6.2.1 to 6.2.5. All layers are to be compacted in a continuous operation.

6.2.7 After the treated material has been placed in the mould for the second layer, transfer 500g to 1,000g of the remaining material in a suitable container for moisture content determination as described in SANS 3001-GR20.

6.2.8 After compaction is complete, remove the mould from the base plate, carefully place the mould and specimen on the carrying plate and following the handling and curing instructions in TG2 Test Method BSM4 (for ITS specimens) or TG2 Test Method BSM5 (triaxial specimens).

7. CALCULATIONS

<u>NOTE 6</u>: An example of the calculation procedure is given in Annexure A.

7.1 Calculations required in the procedure

7.1.1 Determine the mass of each BSM specimen at OMC to the nearest gram, using the following equation:

$$\mathrm{M_S} = \frac{(\pi \times \mathrm{d}^2)}{4 \times 10^6} \times \mathrm{h} \times \left(\mathrm{MDD} \times \left(1 + \frac{\mathrm{OMC}}{100}\right)\right)$$

Where:

MS	*mass of BSM specimen at OMC, expressed in grams (g)*
d	*diameter of the specimen, in millimetres (mm)*
h	*height of the specimen, in millimetres (mm)*
MDD	*MDD, in kilograms per cubic metre (kg/m^3)*
OMC	*OMC, as a percentage (%)*

7.1.2 Determine the total mass of BSM required for manufacturing a batch of specimens to the first decimal place, using the following equation:

$$\mathrm{M_B} = \frac{(\mathrm{N_S} \times \mathrm{M_S})}{1000} + 4$$

Where:

M_B	*mass of BSM required to manufacture a batch of specimens, in kilograms (kg)*
M_S	*number of specimens to be manufactured (see Table B.10)*

7.1.3 Determine the mass of material required per compaction layer (*of equal thickness*) to the nearest gram, using the following equation:

$$\mathrm{M_L} = \frac{\mathrm{M_S}}{\mathrm{n}}$$

Where:

ML	*is the mass of material in each layer, in grams (g)*
n	*is the number of layers (of equal thickness) to be compacted (see Table B.10)*

8. TEST REPORT

Report the MDD to the nearest kg/m^3 and the OMC and moulding moisture content to the nearest 0.1%.

The test report shall include the following general information:

a) Mould diameter
b) Details of aggregate and binder used in the mix including mix proportions
c) Date of manufacture

And for each specimen:

a) Identification marking
b) Number of layers and compaction time for each layer to the nearest 1 second

ANNEXURE A: Calculation Procedure

A.1 Use the following information given in Table B.11 to calculate A.2, A.3, and A.4.

Table B.11 Data for Example Calculations

1	2	3
Property	**ITS test**	**Triaxial test**
d (mm)	152	150
h (mm)	95	300
MDD (kg/m³)	2 145	
OMC (%)	5,6	
NS	6	10
N	2	5

A.2 Calculate the mass of each specimen at the OMC (see paragraph 7.1.1).

A.2.1 ITS test

$$\mathrm{M_S} = \frac{(\pi \times \mathrm{d}^2)}{4 \times 10^6} \times \mathrm{h} \times \left(\mathrm{MDD} \times \left(1 + \frac{\mathrm{OMC}}{100}\right)\right)$$

$$\mathrm{M_S} = \frac{(\pi \times 152^2)}{4 \times 10^6} \times 95 \times \left(2\ 145 \times \left(1 + \frac{5{,}6\mathrm{C}}{100}\right)\right) = 3\ 905\ \mathrm{g}$$

A.2.2 Triaxial test

$$\mathrm{M_S} = \frac{(\pi \times \mathrm{d}^2)}{4 \times 10^6} \times \mathrm{h} \times \left(\mathrm{MDD} \times \left(1 + \frac{\mathrm{OMC}}{100}\right)\right)$$

$$\mathrm{M_S} = \frac{(\pi \times 150^2)}{4 \times 10^6} \times 300 \times \left(2\ 145 \times \left(1 + \frac{5{,}6}{100}\right)\right) = 12\ 008\ \mathrm{g}$$

A.2 Calculate the mass of material required to manufacture a batch of specimens (see paragraph 7.1.2).

A.3.1 ITS test

$$\mathrm{M_B} = \frac{(\mathrm{N_S} \times \mathrm{M_S})}{1000} + 4 = \frac{(6 \times 3\ 905)}{1000} + 4 = 27{,}4\ \mathrm{kg}$$

A.3.2 Triaxial test

$$\mathrm{M_B} = \frac{(\mathrm{N_S} \times \mathrm{M_S})}{1000} + 4 = \frac{(6 \times 12\ 008)}{1000} + 4 = 124.1\ \mathrm{kg}$$

A.3 Calculate the mass of material required per layer (see 7.1.3).

A.4.1 ITS test

$$\mathrm{M_L} = \frac{\mathrm{M_S}}{\mathrm{n}} = \frac{3\ 905}{2} = 1\ 953\ \mathrm{g}$$

A.4.2 Triaxial test

$$\mathrm{M_L} = \frac{\mathrm{M_S}}{\mathrm{n}} = \frac{12\ 008}{5} = 2\ 401\ \mathrm{g}$$

BSM4: DETERMINATION OF THE INDIRECT TENSILE STRENGTH OF BITUMEN STABILISED MATERIAL

1. SCOPE

This test method concerns the determination of the indirect tensile strength of bitumen stabilised material (BSM) in the dry and soaked condition.

2. REFERENCES

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies. Information on currently valid national and international standards can be obtained from the SABS Standards Division.

- SANS 1649, Non-automatic self-indicating, semi-self-indicating and non-self-indicating weighing instruments with denominated verification scale intervals.
- SANS 3001-GR20, Civil engineering test methods Part GR20: Determination of the moisture content by oven-drying.
- SANS 3001-GR30, Civil engineering test methods Part GR30: Determination of the maximum dry density and optimum moisture content.
- TG2, Test Method BSM1, Determination of the foaming characteristics of bitumen.
- TG2, Test Method BSM2, Laboratory Mix Design of Bitumen Stabilised Materials.
- TG2, Test Method BSM3, Vibratory hammer compaction for test specimens of bitumen stabilised material.
- TG2, Test Method BSM5, Determination of the shear properties of bitumen stabilised material (Triaxial test).

3. DEFINITIONS

For the purpose of this document, the following definitions apply:

3.1 Bitumen stabilised material (BSM)
Granular, previously cement treated or reclaimed asphalt material blends, stabilised either using bitumen emulsion or foamed bitumen.

3.2 Indirect tensile strength (ITS)
The stress at failure generated by the load required to split a cylindrical specimen of bitumen stabilised material.

3.3 Maximum dry density (MDD)
The maximum dry density of the material determined from the peak of the dry density versus moisture content curve obtained as described in SANS 3001-GR30.

3.4 Optimum moisture content (OMC)
The moisture content at which the maximum dry density is achieved.

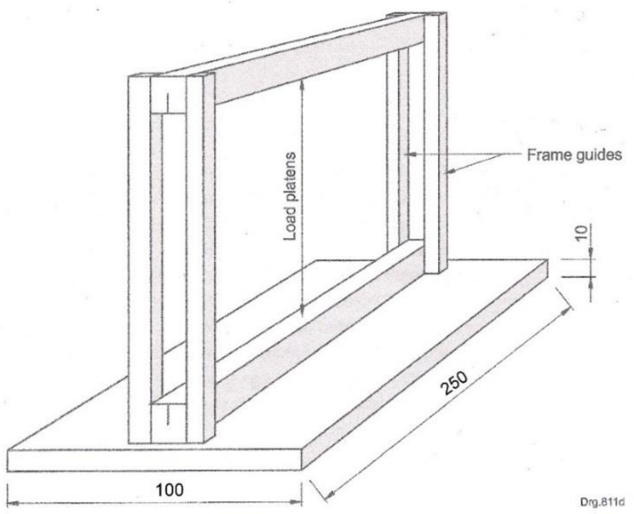
4. APPARATUS

4.1 Compression testing machine
Of capacity at least 30 kN total load, reading to the nearest 50 N and capable of penetrating at a constant rate of 50 ± 5 mm/min, equipped with a force indicating device.

***Warning**: Compression testing machines that are used to break specimens can apply a force in excess of 20 kN on the sample. Samples of BSM tend to deform in a plastic manner under the applied force. However, it is possible for particles to be expelled from the specimen during the test and it is recommended that the operator of the testing machine wears safety glasses for the duration of the test.*

4.2 Force indicating device
Capable of measuring a force of at least 30 kN reading to the nearest 0.05 kN and measuring displacement to the nearest 0.1 mm.

4.3 Indirect tensile strength (ITS) load frame
Consisting of two load platens, of hardened steel, 19 mm × 20 mm × 220 mm, with the 19 ± 0.1 mm face ground concave to a radius of 76 ± 1 mm, together with vertical frame guides, 10 mm × 10 mm × 175 mm, of mild steel attached to a base plate, of mild steel of suitable design to align the loading platens on the test specimen. The upper load platen shall be contained by the guides but shall slide freely between them and shall have a vertical centre-mark on each end face. The lower platen shall be fixed to the base plate. Other frame designs, which incorporate a load transfer plate, load cell and automatic data logger, may be used provided that the load platens comply with the dimensions given.



Description	**Dimensions** (mm)
Loading platen	19 x 20 x 220
Concave face of load platens	76 ± 1

Figure B3 Typical ITS Load Frame

4.4 Load transfer plate
Steel, round or square, or 19 mm diameter steel ball, to transfer the load from the compression testing machine to the top loading platen without any deformation. Its dimensions should be such that it is slightly longer than the specimen to be tested. The ball bearing is not to be used between the load bearing plate and the piston.

4.5 Drying oven
Capable of maintaining a temperature range of 39 °C to 41 °C with continuous forced-draft and of minimum capacity 240 litres.

4.6 Water bath
At least 150 mm deep to ensure that the specimens are covered by at least 25 mm of water, thermostatically controlled with a circulation mechanism to maintain a temperature of 25 °C ± 1 °C, with a perforated false bottom 25 mm high.

4.7 Electronic balance

Fine measurement type complying with SANS 1649 with a capacity of 5 kg and reading to 0.1 g.

4.8 Digital thermometer

Capable of measuring from 0 °C to 100 °C reading to ±0.1 °C.

4.9 Carrier plates

Plywood with a thickness of approximately 15 mm and diameter approximately 160 mm, one for each compacted specimen.

4.10 Callipers

Steel to measure the height and diameter of the test specimen reading to 0.1 mm.

5. SPECIMEN PREPARATION

5.1 Manufacture

Prepare and compact six ITS specimens as described in TG2, Test Methods BSM2 and BSM3. Specimens are manufactured at Optimum Moisture Content (OMC) for treatment with foamed bitumen and at Optimum Fluid Content (OFC) for treatment with bitumen emulsion.

Leave the specimens to stand for a minimum of 4 hours before removing them from their respective moulds and carefully placing each on a carrying plate.

5.2 Curing

5.2.1 Before curing, lift one of the specimens off its carrying plate and weigh. Record the mass (MW) to the nearest gram. Place the specimen back on its carrying plate.

5.2.2 Place the six specimens on their carrying plates in the oven at a temperature of 40 ± 1 °C for a period of at least 72 hours. Ensure that there is a minimum air space of 25 mm between specimens.

5.2.3 Remove one specimen per batch from the oven, weigh each one and record the mass to the nearest 1 g.

5.2.4 Return all the specimens to the oven for a further 4 hours.

5.2.5 Remove one specimen per batch from the oven, weigh each one and record the mass to the nearest 1 g.

5.2.6 Compare the masses obtained in the last two weighing cycles. When any specimen loses more than 10 g repeat 5.2.3 to 5.2.4 until no specimen loses more than 10 g.

5.2.7 Leave the specimens to cool to a temperature of 25 °C.

5.3 Measurements

5.3.1 Weigh each specimen and record the mass (MC) to the nearest 0.1 g.

5.3.2 Measure the height at three evenly spaced points around the circumference (h1, h2 and h3), calculate the average and record to the nearest 0.5 mm.

5.3.3 Measure the diameter at mid-height, at three evenly spaced points (d1, d2 and d3), calculate the average and record to the nearest 0.1 mm.

5.3.4 Calculate the bulk density of each specimen using the equation given in 7.1.1.

5.3.4.1 Calculate the mean (BD_{ave}) and standard deviation (s) of the bulk density results for the six specimens.

5.3.4.2 Check the set of bulk density results for an outlier using the mean and standard deviation values obtained in 5.3.4.1 and the equation given in 7.1.2.

5.3.4.3 When an outlier is obtained, exclude the specimen with that bulk density value and recalculate the mean and standard deviation for the remaining five values.

5.3.4.4 If a second outlier is obtained either discard all the specimens and repeat the procedure from 5.1 or abandon the test.

6. PROCEDURE

6.1 Unsoaked specimens

6.1.1 Leave the unsoaked specimens in a temperature controlled environment for at least 4 hours to achieve a temperature of 25 ± 1.5 °C.

6.2 Soaked specimens

6.2.1 Place three of the specimens in the water bath and soak for 24 hours at 25 ± 3 °C. Ensure that the specimens are covered by at least 25 mm of water.

6.2.2 Remove the specimens from the water bath, surface dry and test immediately.

6.3 ITS testing

6.3.1 Place the specimen on the bottom loading strip. Then place the top loading strip on top of the specimen, diametrically opposite the bottom strip. Ensure that the loading strips are parallel and centred on the vertical diametrical plane. Place the transfer plate (or steel ball) on the top bearing strip and position the assembly centrally under the loading ram of the compression testing device. Apply a load of 0.1 kN to the specimen to seat the loading strips. Inspect the assembly for symmetry.

6.3.2 Load the specimen at a steady rate of 50 ± 5 mm/min, until the maximum force is achieved. Record the maximum force, G_1, to the nearest 0.1 kN and record the displacement at peak load to the nearest 0.1 mm.

6.3.3 Unload the compression tester, remove the specimen.

6.3.4 Immediately after testing break each specimen in half. Record the temperature at the centre of the broken face to the nearest 0.1 °C.

6.3.5 Separate the broken unsoaked specimens from the soaked specimens. Break the half portions into small pieces and determine the moisture content of each respective group using SANS 3001 GR20 and record.

7. CALCULATIONS

<u>NOTE 1</u>: An example of the calculation procedure is given in Annexure A.

7.1 Calculations required in the procedure

7.1.1 Determine the bulk density of specimens in each set, unsoaked and soaked, to the nearest kilogram per cubic metre, using the following equation:

$$\mathrm{BD} = \frac{4 \times 10^6 \times \mathrm{M_C}}{(\pi \times \mathrm{d}^2) \times \mathrm{h}}$$

Where:

BD	bulk density of the treated specimen, in kilograms per cubic metre, (kg/m^3)
M_C	mass of cured treated specimen, expressed in grams (g)
d	diameter of the specimen, expressed in millimetres (mm)
h	is the height of the specimen, expressed in millimetres (mm)

7.1.2 Determine if there is an outlier in the set of bulk density results

7.1.2.1 Sort the BD set in ascending order.

$BD_{set} = \{BD_1; BD_2; …; BD_n\}$

7.1.2.2 When $(BD_2 - BD_1) \leq (BD_n - BD_{n-1})$ is true, BDn is the potential outlier, and when $(BD_2 - BD_1) > (BD_n - BD_{n-1})$ is true, BD_1 is the potential outlier.

7.1.2.3 Check for outlier, using the following equation:

When

$$\frac{|BD_{AVE} - BD_{Potential\ outlier}|}{s} > T_0$$

is true, the value is an outlier.

Where:

BD_{ave} mean of the bulk density set of values

s standard deviation of the bulk density set of values

Table B.12 Critical Outlier Value Versus Number of Samples

Number of specimens n	Critical outlier value T_o
5	1.672
6	1.822

***NOTE 2*: See SANS 3001-PR1, Annex B, Table B.6.**

7.1.3 When the potential value is an outlier, discard that value and repeat 7.1.2 to 7.1.3 using the amended data set and calculating new average and standard deviation values. If a second outlier is obtained, repeat or abandon the test.

7.2 Determine the Indirect Tensile Strength of the Treated Material using the following equation:

$$ITS = \frac{2 \times G}{\pi \times d \times h} \times 10^6$$

Where:

ITS indirect tensile strength, in kiloPascals, (kPa)

G applied force, in kiloNewtons, (kN)

d specimen diameter, millimetres (mm)

h specimen height, in millimetres (mm)

7.3 Determine the dry density of each specimen using the following formulae:

$$M_D = \frac{100 \times M_W}{100 + W}$$

Where:

M_D dry mass of the specimen, in grams (g)

MW mass of the moist specimen after compaction, in grams (g)

W moisture content of the specimen determined during compaction (see SANS 3001-BSM2), as a percentage (%)

$$D_D = \frac{4 \times 10^6 \times M_D}{(\pi \times d^2) \times h}$$

Where:

D_D *dry density of the specimen, in kilograms per cubic metre (kg/m^3)*

8. TEST REPORT

8.1 Report the BD to the nearest kg/m^3; and the ITS soaked and dry, to the nearest kPa, for:

a) individual specimens

b) mean values for the data set

8.2 The test report shall include the following general information:

a) average specimen height

b) details of aggregate and binder used in the mix including mix proportions

c) dates of:

    i) manufacture
    ii) start and end of curing (including times)
    iii) testing

d) identification of outliers

8.3 The test report shall include for each specimen:

a) identification marking

b) moisture content when tested (soaked or unsoaked)

c) dry density and moisture content

d) specimen height and diameter

e) deformation at failure

f) maximum load applied

g) temperature at centre of specimen

ANNEXURE A: Calculation Procedure

A.1 Use the following information given in Table B13 to calculate A2, A3 and A4.

Table B.13 Data for Example Calculations

Bulk density		Outlier	
Property	**Value**	**Property**	**Value (kg/m³)**
M_C	3 836 g	BD_1	2 244
d	152 mm	BD_2	2 230
h	94,2 mm	BD_3	2 251
		BD_4	2 220/
		BD_5	2 168
		BD_6	2 224

A.2 Calculate the bulk density of a specimen (see 7.1.1)

$$\mathrm{BD} = \frac{4 \times 10^6 \times \mathrm{M_C}}{(\pi \times \mathrm{d}^2) \times \mathrm{h}} = \frac{4 \times 10^6 \times 3\,836}{(\pi \times 152^2) \times 94.2} = 2\,244\ \mathrm{kg/m^3}$$

A.3 Calculate the mean and standard deviation of the BD data set

BD_{ave} = 2 222.8 kg/m³ and s = 29.4

A.4 Determine if an outlier exists (see paragraph 7.1.2).

A.4.1 Sort BD data set into ascending order (see 7.1.2.1)

BD_{set} = {2 168; 2 220; 2 240; 2 230; 2 244; 2 251}

A.4.2 Determine potential outlier (see 7.1.2.2)

$(BD_2 - BD_1) > (BD_n – Bd_{n-1})$ = (2 220 – 2 168) > (2 251 – 2 244)

= (52) > (7) is true and thus BD_1 is the potential outlier

A.4.3 Check for outlier (see 7.1.2.3)

T_o for n = 6 is 1.822 (see Table B.12)

$$\frac{|\mathrm{BD_{AVE}} - \mathrm{BD_{Potential\ outlier}}|}{\mathrm{s}} = \frac{|2\,222{,}8 - 2\,168|}{29.4} = 1{,}87$$

Thus,

$$\frac{|2\,222{,}8 - 2\,168|}{29.4} = 1.84 > T_0 = 1{,}822$$

And BD_1 is an outlier.

Discard BD_1 (2 168 kg/m³) and repeat calculations for the amended BD data set with n=5.

A.5 Use the following information given in Table B.14 to calculate A.6 and A.7.

Table B.14 Data for Example Calculations

1	2
Property	**Value**
G	5.2 kN
h	94.2 mm
d	152 mm
M_W	3 814 g
W *	5.1 %

<u>NOTE 1</u>: The value of W is obtained from SANS 3001-BSM2 compaction data.

Calculate the ITS (see paragraph 7.2)

$$\text{ITS} = \frac{2 \times \text{G}}{\pi \times \text{d} \times \text{h}} \times 10^6 = \frac{2 \times 5.2}{\pi \times 152 \times 94{,}2} \times 10^6 = 231\ \text{kPa}$$

A.6 Calculate the dry density of each specimen (see paragraph 7.3)

$$M_D = \frac{100 \times M_W}{100 + W} = \frac{100 \times 3{,}814}{100 + 5{,}1} = 3{,}629\ \text{g}$$

$$\text{D}_\text{D} = \frac{4 \times 10^6 \times \text{M}_\text{D}}{(\pi \times \text{d}^2) \times \text{h}} = \frac{4 \times 10^6 \times 3{,}629}{(\pi \times 152^2) \times 94{,}2} = 2\ 123\ \text{kg/m}^3$$

BSM5: DETERMINATION OF THE SHEAR PROPERTIES OF BITUMEN STABILISED MATERIAL (TRIAXIAL TEST)

1. SCOPE

This test method is concerned with the determination of the shear properties (cohesion (C) and angle of internal friction (ϕ)) of bitumen stabilised material (BSM) using a simple monotonic triaxial test. The specimens tested are 150 mm in diameter and 300 mm high.

2. REFERENCES

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies. Information on currently valid national and international standards can be obtained from the SABS Standards Division.

- SANS 1649, Non-automatic self-indicating, semi-self-indicating and non-self-indicating weighing instruments with denominated verification scale intervals.
- SANS 3001-GR20, Civil engineering test methods Part GR20: Determination of the moisture content by oven-drying.
- TG2, Test Method BSM1, Determination of the foaming characteristics of bitumen.
- TG2, Test Method BSM2, Laboratory Mix Design of Bitumen Stabilised Materials.
- TG2, Test Method BSM3, Vibratory hammer compaction for test specimens of bitumen stabilised material.
- TG2, Test Method BSM4, Determination of the indirect tensile strength of bitumen stabilised material.

3. DEFINITIONS

For the purpose of this document, the following definitions apply:

3.1 Bitumen stabilised material (BSM)
Granular, previously cement treated or reclaimed asphalt material, stabilised either using bitumen emulsion or foamed bitumen.

3.2 Shear properties
The values cohesion (c) and angle of internal friction (ϕ) determined from the Mohr-Coulomb circles obtained by plotting the vertical stress (σ_1) applied to the specimens in a simple monotonic triaxial test at a range of confining pressures (σ_3).

4. APPARATUS

4.1 Triaxial cell
Consisting of a confining cylinder and a base and top plate, capable of safely withstanding a confining pressure of at least 200 kPa. The internal dimensions of the cylinder are sufficient to accommodate a 150 mm diameter by 300 mm high specimen enclosed in an inflatable rubber bladder.

4.2 Rubber bladder
With an uninflated internal diameter of 160 ± 5 mm and height of 330 mm, fitted with an inflation valve.

Warning: *The rubber bladder is only to be inflated after the confining cylinder has been properly assembled with a specimen of the specified diameter and height. Improper assembly of the confining cylinder and/or attempting to use a smaller specimen can result in the bladder blowing out (exploding) when inflated, especially when high inflation pressures are applied. Such a blow-out has the potential to cause serious bodily harm.*

4.3 Compression testing machine

Capable of applying a force of at least 200 kN reading to the nearest 0.1 kN at a constant rate of 3 mm/min and measuring vertical displacement to the nearest 0.1 mm. The machine shall be capable of taking readings at 1 second intervals and shall have sufficient clearance to accommodate the assembled confining cylinder and top and plates. The moving actuator / loading ram is to be situated above the triaxial cell with a fixed reaction base below the cell.

4.4 Air compressor

With pressure gauge reading to 1 kPa and regulator capable of inflating the bladder and maintaining a maximum pressure of 200 ± 2.5 kPa.

4.5 Drying oven

Capable of maintaining a temperature of 40 ± 1 °C with continuous forced-draft and of minimum capacity 240 litres.

4.6 Water bath

At least 350 mm deep to ensure that the specimens are covered by at least 25 mm of water, thermostatically controlled with a circulation mechanism to maintain a temperature of 25 ± 1 °C, with a perforated false bottom 25 mm high.

4.7 Electronic balance

Fine measurement type that complies with SANS 1649 with a capacity of 20 kg and reading to ± 0.1 kg.

4.8 Digital thermometer

Capable of measuring from 0 °C to 100 °C reading to ± 0.1 °C.

4.9 Carrier plates

Plywood of thickness approximately 15 mm and diameter approximately 160 mm, one for each compacted specimen.

4.10 Plastic bags

Capacity 10 litres which can be sealed watertight.

5. SPECIMEN PREPARATION

5.1 Manufacture

Prepare and compact ten triaxial specimens as described in the TG2, Test Methods BSM2 and BSM3. Specimens are manufactured at Optimum Moisture Content (OMC) for treatment with foamed bitumen and at Optimum Fluid Content (OFC) for treatment with bitumen emulsion.

5.2 Measuring and conditioning the specimens

5.2.1 Leave all ten specimens overnight in their respective moulds covered with a moist hessian cloth.

5.2.2 Remove the specimens from their respective moulds the following morning. Mark each one with an appropriate identity number.

5.2.3 Place the specimens in the oven at a temperature of 40 ± 1 °C for 8 hours. Ensure that there is a minimum air space of 25 mm between specimens. To avoid damage to the large specimens, exercise care when moving them. Specimens are always moved on their respective carrier plates.

5.3 Curing

5.3.1 After 8 hours (i.e., when 5.2.3 is complete), remove all the specimens from the oven. Place each specimen in a loose-fitting plastic bag and seal the bag.

5.3.2 Place the specimens (in plastic bags) into the oven at a temperature of 40 ± 1 °C for 48 hours.

5.3.3 Remove the specimens from the oven after 48 hours and take them out of their respective plastic bags.

5.3.4 Weigh each specimen and record the mass (M_{PC}) to the nearest gram.

5.3.5 Measure the height at three evenly spaced points around the circumference (h_1, h_2 and h_3), calculate the average and record to the nearest 0.1 mm.

5.3.6 Measure the diameter at mid-height, at three evenly spaced points (d_1, d_2 and d_3), calculate the average and record to the nearest 0.1 mm.

5.3.7 Place two of the specimens under water for 24 hours in a soaking bath at a temperature of 25 °C. After 24 hours, remove the specimens from the water, surface dry and weigh before testing. Ensure that the specimens are covered by at least 25 mm of water.

5.3.8 Place the other eight specimens in fresh (dry) loose fitting plastic bags and leave to cool to 25 ± 2 °C for a minimum period of 12 hours. Each specimen is then removed from its plastic bag and tested.

5.4 Identify outliers

5.4.1 Calculate the bulk density (BD) of each specimen using the equation given in 7.1.1.

5.4.2 Calculate the mean (BD_{ave}) and standard deviation (s) of the bulk density results for the ten specimens.

5.4.3 Check the set of bulk density results for an outlier using the mean and standard deviation values obtained in 5.4.2 and the equation given in 7.1.2.

5.4.4 When an outlier is identified, exclude that bulk density value and recalculate the mean and standard deviation for the remaining nine values.

5.4.5 If a second outlier is identified, exclude that bulk density value and recalculate the mean and standard deviation for the remaining eight values.

5.4.6 If a third outlier is identified, abandon the test.

6. PROCEDURE

6.1 Preparation of test assembly

6.1.1 Fit the specimen carefully into the bladder inside the confining cylinder. Position the cylinder and specimen in the middle of the base plate taking care not to damage the edges of the specimen. Fasten the cylinder to the base plate. Carefully position the top plate on the specimen.

6.1.2 Check that there is sufficient space between the actuator / loading ram and the fixed reaction base of the compression testing machine. Place the assembly of specimen, cylinder and plates, on the reaction base of the compression testing machine and align correctly.

6.1.3 Lower the loading ram until it makes contact with the depression in the centre of the top plate. Monitor the load cell reading to ensure that the specimen is not loaded during this process.

6.1.4 Connect the air supply to the inflation valve protruding through the confining cylinder. Adjust the pressure regulator to inflate the bladder to the required pressure.

6.1.5 The monotonic triaxial testing is undertaken at four different confining pressures (σ_3): 0 kPa, 50 kPa, 100 kPa and 200 kPa.

Where sufficient specimens are available, two unsoaked (dry) specimens are tested at each confining pressure. When one specimen is identified as an outlier and excluded), test only one unsoaked specimen at a confining pressure of 50 kPa. If two specimens were excluded, test one unsoaked specimen at both the 50 kPa and 100 kPa confining pressures.

The two soaked specimens are tested at a confining pressure of 100 kPa.

6.2 Triaxial test

6.2.1 Set the compression testing machine in displacement control mode at a rate of 3 mm/min. Ensure that the load (f_i) and displacement (Δ_i) readings are measured and recorded every second.

6.2.2 Apply the vertical load up to a displacement of 18 mm (6% strain), or sooner if the load starts to reduce from the maximum.

6.2.3 Unload the specimen by returning the actuator to its start position and release the confining pressure. Move the actuator clear of the top plate and remove the confining cylinder assembly from the loading frame. Dismantle the confining cylinder and carefully remove the specimen from the bladder. Immediately break the specimen and record the temperature in the centre and middle to the nearest 0.1 °C.

6.2.4 Take a sample of approximately 1 000 g of material from the middle portion of the specimen and place in a sealed container. Determine the moisture content as described in SANS 3001-GR20.

6.2.5 Repeat the procedure until all the specimens (unsoaked and soaked) have been tested at the confining pressures given in 6.1.5.

7. CALCULATIONS

***NOTE 1**: An example of the calculation procedure is given in Annexure A.*

7.1 Calculations required in the procedure

7.1.1 Determine the bulk density of each specimen to the nearest kilogram per cubic metre, using the following equation:

$$BD = \frac{4 \times 10^6 \times M_{PC}}{(\pi \times d^2) \times h}$$

Where:

BD *bulk density of the treated specimen, in kilograms per cubic metre (kg/m^3)*
M_{PC} *mass of the treated specimen, expressed in grams (g)*
d *diameter of the specimen, expressed in millimetres (mm)*
h *height of the specimen, expressed in millimetres (mm)*

7.1.2 Determine if there is an outlier in the set of bulk density results

7.1.2.1 Sort the BD set in ascending order.

$$BD_{set} = \{BD_1; BD_2; \ldots; BD_n\}$$

7.1.2.2 When $(BD_2 - BD_1) \leq (BD_n - BD_{n-1})$ is true, BDn is the potential outlier, and when $(BD_2 - BD_1) > (BD_n - BD_{n-1})$ is true, BD_1 is the potential outlier.

7.1.2.3 Check for outlier, using the following equation:

When:

$$\frac{|BD_{AVE} - BD_{Potential\ outlier}|}{s} > T_0 \qquad \textit{the value is an outlier.}$$

Where:

BD_{ave} *mean of the bulk density set of values*
s *standard deviation of the bulk density set of values*

Table B.15. Critical Outlier Value Versus Number of Samples

Number of specimens n	Critical outlier value T_o
8	2.03
9	2.11
10	2.18

NOTE 2: See SANS 3001-PR1, Annex B, Table B.6.

7.1.3 When the potential value is an outlier, discard that value and repeat 7.1.2 using the amended data set (with only 9 values) to calculate new average and standard deviation values. If a second outlier is obtained, discard that value as well as repeat 7.1.2 using the amended data set (with only 8 values) to obtain new average and standard deviation values. If a third outlier is obtained, repeat or abandon the test.

7.2 Determine the triaxial shear properties cohesion (C) and angle of internal friction (ϕ)

7.2.1 Plot the applied vertical load (f_i) on the vertical axis versus displacement (Δ_i) on the horizontal axis for each specimen using the compression testing machine output. Read off and record the force at peak load or 18 mm displacement, (f_P), whichever is less, and the confining pressure (σ_3) for each specimen.

7.2.2 Calculate the total applied peak stress.

7.2.2.1 Determine the applied failure stress measured by the compression testing machine, using the following equation:

$$\sigma_{1P} = \frac{4 \times f_P \times 10^6}{(\pi \times d^2)}$$

Where:

σ_{1P} *applied peak stress measured by compression testing machine, in kilopascals (kPa)*

f_P *applied peak load, in kilonewtons (kN)*

d *specimen diameter at the start of the test, in milimetres (mm)*

7.2.2.2 Determine the total applied failure stress including the dead weight of the top plate and loading ram, using the following equation:

$$\sigma_{1TP} = \sigma_{1P} + \frac{4 \times M_{DW} \times 9{,}81}{(\pi \times d^2)}$$

Where:

σ_{1TP} *total applied stress including the weight of the top plate and loading ram, in kilopascals, (kPa)*

M_{DW} *mass of the top plate and loading ram, in grams (g)*

9,81 is the standard acceleration due to gravity, in metres per second per second (m/s^2)

7.2.3 Plot the total applied stress (σ_{1TP}) on the vertical axis versus the confining stress (σ_3) on the horizontal axis; for all the unsoaked specimens.

7.2.3.1 Select the best fit straight line through the points.

7.2.3.2 Determine the slope of the best fit line (A) and the intercept with the vertical **axis (B).**

NOTE 3: Commercial spreadsheets are available to plot the points, provide a best fit line and calculate a linear regression equation. A graphical interpretation should provide an answer within 2% of the calculated value.

7.3 Determine the shear properties cohesion (C) to the nearest kilopascal and angle of internal friction (ϕ) to the nearest first decimal place of a degree, using the following equations

$$\phi = sin^{-1}\left(\frac{A-1}{A+1}\right)$$

and

$$C = B \times \frac{1 - sin\,\phi}{2 \times cos\,\phi}$$

7.4 Determine the retained cohesion, expressed as the net applied stress after and before soaking, as a percentage, using the following equation

$$RetC = \frac{\left(\sigma_{1WET,F} - \sigma_3\right)}{\left(\sigma_{1EQUIL,F} - \sigma_3\right)} \times 100$$

Where:

RetC	*retained cohesion after soaking, expressed as a percentage;*
σ_3	*confining pressure in kilopascals (kPa)*
$\sigma_{1,WET,F}$	*soaked applied peak stress at confining pressure σ_3, in kilopascals (kPa)*
$\sigma_{1EQUIL,F}$	*unsoaked applied peak stress read from the best fit line at confining pressure (σ_3) in kilopascals (kPa).*

7.5 Determine the dry density and moisture content after curing and soaking using the following equation

$$D_T = \frac{100 \times M_{PS} \times 4 \times 10^6}{(100 + W_{PS}) \times (\pi \times h \times d^2)}$$

Where:

D_T	*dry density when tested, expressed in kilograms per cubic metre (kg/m^3)*
M_{PS}	*mass after soaking and just before testing, expressed in grams (g)*
W_{PS}	*moisture content measured just after testing, expressed as a percentage (%)*

8. TEST REPORT

8.1 Report

a) The slope (A) to two decimal places and intercept of the vertical axis (B) to the nearest kPa of the best fit σ_1 versus σ_3 line.
b) The cohesion (C) to the nearest kPa.
c) The internal angle of friction (ϕ) to the first decimal of a degree.
d) The retained cohesion (C_{RET}) to the nearest percentage point.

8.2 The test report shall include the following general information:

a) average specimen height

b) details of aggregate and binder used in the mix, including mix proportions and binder used

c) the following dates of:

   i) manufacture
   ii) start of moisture content adjustment (including time)
   iii) start and end of curing (including times)
   iv) testing

e) identification of outliers

8.3 <u>The test report shall include for each specimen:</u>

a) identification marking
b) condition when tested (soaked or unsoaked)
c) dry density and moisture content
d) specimen height and diameter
e) bulk density
f) deformation at failure
g) maximum load applied
h) temperature at centre of specimen
i) moisture content after testing, soaked and unsoaked specimens
j) dry density
k) confining pressure (σ_3)
l) peak load (f_P)
m) total applied stress (σ_{1TP})

ANNEXURE A: Calculation Procedure

A.1 Use the following information given in Table B.16 calculate A2, A3 and A4.

Table B.16 Data for Example Calculations to Check for Outlier

1	2	3	4
Bulk Density		**Outlier**	
Property	**Value**	**Property**	**Value (kg/m^3)**
M_{PC}	12 926 g	BD_1	2 407
d	150 mm	BD_2	2 449
h	302 mm	BD_3	2 422
		BD_4	2 434
		BD_5	2 404
		BD_6	2 418
		BD_7	2 412
		BD_8	2 407
		BD_9	2 403
		BD_{10}	2 415

A.2 Calculate the bulk density of a specimen (see 7.1.1).

$$\mathrm{BD} = \frac{4 \times 10^6 \times \mathrm{M_{PC}}}{(\pi \times \mathrm{d}^2) \times \mathrm{h}} = \frac{4 \times 10^6 \times 12\,929}{(\pi \times 150^2) \times 302} = 2\,422\ \mathrm{kg/m^3}$$

A.3 Calculate the mean and standard deviation of the BD data set.

BD_{ave} = 2 417.1 kg/m^3

s = 14.65

A.4 Determine if an outlier exists (see 7.1.2).

A.4.1 Sort BD data set into ascending order (see 7.1.2.1).

Bd_{set} = {2 403; 2 407; …… ; 2 434; 2 449}

A.4.2 Determine potential outlier (see 7.1.2.2).

$(BD_2 - BD_1) \leq (bd_n - Bd_{n-1}) = (2\,403 - 2\,407) \leq (2\,449 - 2\,434) = 4$

$(4) \leq (15)$ is true and thus BD_{10} is the potential outlier.

A.4.3 Check for outlier (see 7.1.2.3).

for n = 10 samples $T_0 = 2.176$ (see Table B.15)

$$\frac{|BD_{AVE} - BD_{Potential\ outlier}|}{s} = \frac{|2\,417{,}1 - 2\,449|}{14.65} = 2{,}178$$

Thus,

$$\frac{|BD_{AVE} - BD_{Potential\ outlier}|}{s} = 2{,}178 > T_0 = 2{,}176$$

And, BD_{10} is an outlier.

Discard BD_{10} (2 449 kg/m^3) and repeat calculations for the amended BD data set.

A.5 Use the following information given in Table B.17 to calculate A.6 and A.7

Table B.17 Data for Example Calculations for Test Values

1	2
Property	**Value**
M_M	12 926 g
M_{Ci}	12 496 g
OMC or OFC	5.5%
d	150 mm
f_P	49.7 kN
M_{DW}	5 100 g
M_{PS}	12 875 g
W_{PS}	3.5%

A.6 Calculate the dry density at time of testing

$$D_T = \frac{100 \times M_{PS} \times 4 \times 10^6}{(100 + W_{PS}) \times (\pi \times h \times d^2)} = \frac{100 \times 12{,}875 \times 4 \times 10^6}{(100 + 3{,}5) \times (\pi \times 302 \times 150^2)} = 2\ 331\ \text{kg/m}^3$$

A.7 Calculate the total applied peak stress

$$\sigma_{1P} = \frac{4 \times f_P \times 10^6}{(\pi \times d^2)} = \frac{4 \times 49{,}7 \times 10^6}{(\pi \times 150^2)} = 2\ 812\ \text{kPa}$$

and

$$\sigma_{1TP} = \sigma_{1P} + \frac{4 \times M_{DW} \times 9{,}81}{(\pi \times d^2)} = 2\ 812 + \frac{4 \times 5\ 100 \times 9{,}81}{(\pi \times 150^2)} = 2\ 815\ \text{kPa}$$

A.8 Use the following information given in Table B.18 to calculate A.9 to A11:

Table B.18 Data for Example Calculations

Measured Stresses (kPa)				
σ_3	σ_1			
	Unsoaked		**Soaked**	
0	1 270	1 262		
50	1 436	1 515		
100	1 821	1 772	1 381	1397
200	2 255	2 315		

A.9 Calculate the best fit line, intercept value and slope

Using the data in Table B.18 and a commercial spreadsheet, determine the best fit equation for the σ_3 versus σ_1.

f(x) = 5.18457 x + 1 252.1

and thus,

A the slope of the line = 5.18

B the intercept with the y axis = 1 252 kPa

A.10 Calculate the shear stresses c and ϕ

$$\phi = \sin^{-1}\left(\frac{A-1}{A+1}\right) = \sin^{-1}\left(\frac{5.18-1}{5.18+1}\right) = 42.6$$

and

$$\mathrm{C} = \mathrm{B} \times \frac{1-\sin\phi}{2\times\cos\phi} = 1\ 252 \times \frac{1-\sin 42{,}6\phi}{2\times\cos 42{,}6} = 275\ \mathrm{kPa}$$

A.11 Calculate the retained cohesion

Using the data in Table B.18:

Mean soaked applied peak stress at 100 kPa confining pressure

= 0.5 x (1 381 + 1 397) = 1 389 kPa

Mean unsoaked applied peak stress at 100 kPa confining pressure

= 0.5 x (1 821 + 1 772) = 1 796.5 kPa

$$\mathrm{RetC} = \frac{(\sigma_{1\mathrm{S,F}} - \sigma_3)}{(\sigma_{1\mathrm{US,F}} - \sigma_3)} \times 100 = \frac{(1\ 389 - 100)}{(1\ 796.5 - 100)} \times 100 = 76\%$$