IS 15462 (2004): Polymer and Rubber Modified Bitumen [PCD 6: Bitumen Tar and their Products]

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IS 15462 : 2004

भारतीय मानक

पॉलिमर और रबड़ परिवर्तित बिटुमन — विशिष्टि

Indian Standard

POLYMER AND RUBBER MODIFIED BITUMEN — SPECIFICATION

ICS 75.140

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

February 2004 **Price Group 6**

Bitumen, Tar and Their Products Sectional Committee, PCD 6

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Bitumen, Tar and Their Products Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

Polymer and rubber modified bitumen, often abbreviated as polymer modified bitumen is obtained by the incorporation of thermoplastics, crumb rubber powder (ordinary) or chemically treated crumb rubber and elastomers or a blend of polymeric and other additives compatible with bitumen or a short residue obtained after refining of crude oil. The modified bitumen is prepared at refineries or at suitable centrally located or mobile plants with high shear mixing facility. Recently, fully automated mobile plants with high shear mixing facility are also imported to provide high modified bitumen at the locations of hot mix plants. Mixing at site by simple stirrer is not advisable.

Over the years, different types of modifiers have been used to make modified bitumen. The most commonly used type of modifiers are rubbers and polymers. These are macro-molecules in which the same group of atoms is repeated very large number of times. These repeated groups can be formed from one or several different molecules (monomers). Agents other than synthetic polymers can also be used to modify bitumen, which are crumb rubber powder and natural rubber powder or in latex form. The table below lists the groups of principal modifiers which are used to modify bitumen for highway engineering applications and are also specified in Indian Road Congress (IRC) and Ministry of Road Transport & Highways (MoRTH) standards, specifications and codes of practice.

Principal chemical agents used to prepare rubber and polymer modified bitumen are :

Plastomeric Thermoplastic Polymers	
a) Polyethylene	PE
b) Ethylene-Vinyl Acetate Copolymer	EVA
c) Ethylene-Methyl Acrylate Copolymer	EMA
d) Ethylene-Butyl Acrylate Copolymer	EBA
Elastomeric Thermoplastic Polymers	
a) Ethylene Ter-Polymer	ETP
b) Styrene-Butadiene-Styrene Block Copolymer	SBS
c) Styrene-Isoprene-Styrene Copolymer	SIS
d) Styrene-Butadiene	SB
e) Latex and Other Rubbers	
f) Styrene Butadiene Rubber	SBR
Natural Rubber (Latex or Powder)	NR
Crumb Rubber or Treated Crumb Rubber	CR

While preparing this standard, considerable assistance has been derived from IRC SP : 53-2002 'Guidelines on use of polymer and rubber modified bitumen in road construction' prepared by Indian Road Congress and clause 520 of specification by Ministry of Road Transport & Highways, New Delhi.

The composition of the Committee responsible for formulation of this standard is given in Annex D.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 1 JULY 2005
TO
IS 15462 : 2004 POLYMER AND RUBBER MODIFIED BITUMEN

(*Page* 1, *clause* 2, *IS No. and Title*) — Substitute the following for the existing:

'IS 1206 (Part 2) : 1978 Methods of testing tar and bituminous materials, Determination of viscosity : Part 2 Absolute viscosity (*first revision*)'

[*Pages* 2, 3 *and* 4, *Tables* 1, 2, 3 *and* 4, *Sl No.* (vii), *col* 6] — Substitute '1206 (Part 2) : 1978' *for* '1206 (Part I) : 1978'.

[*Page* 2, *Table* 1, *Sl No.* (viii)(d), *col* 4] — Substitute '30' *for* '35'.

[*Page* 2, *Table* 1, *Sl No.* (viii)(d), *col* 5] — Substitute '20' *for* '35'.

(*Page* 6, *Annex* B, *clause* **B-3.1**, *line* 4) — Insert 'about 100 g of' after 'Pass' and 'of 200 mm diameter and' after 'IS Sieve'.

(*Page* 6, *Annex* B, *clause* **B-3.2**, *line* 3) — Substitute '48 ± 4 h' *for* '24 ± 4 h'.

(PCD 6)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 2 APRIL 2010
TO
IS 15462 : 2004 POLYMER AND RUBBER MODIFIED BITUMEN — SPECIFICATION

(*Page* 4, *Table* 4, *Title*) — Substitute '**Crumb Rubber**' *for* '**Polymer**'.

(*Page* 2, *Table* 1, *Note*) — Substitute the following for the existing:

'Requirements at *Sl No*. (v) and (viii)(d) are subject to agreement between the buyer and the seller.'

(PCD 6)

Reprography Unit, BIS, New Delhi, India

Indian Standard

POLYMER AND RUBBER MODIFIED BITUMEN — SPECIFICATION

1 SCOPE

This standard covers the requirements for physico-chemical properties of rubber and polymer modified bitumen binders for use in highways, airfield and other allied construction and maintenance works.

2 REFERENCES

The following standards contain provisions which, through reference in the text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

IS No.	*Title*
73 : 1992	Specification for paving bitumen (*second revision*)
334 : 2002	Glossary of terms relating to bitumen and tar (*third revision*)
1201 : 1978	Methods of testing tar and bituminous materials : Sampling (*first revision*)
1203 : 1978	Methods of testing tar and bituminous materials : Determination of penetration (*first revision*)
1205 : 1978	Methods of testing tar and bituminous materials : Determination of softening point (*first revision*)
1206 (Part 1) : 1978	Methods of testing tar and bituminous materials : Determination of viscosity : Part 1 Industrial viscosity (*first revision*)
1208 : 1978	Methods of testing tar and bituminous materials : Determination of ductility (*first revision*)
1209 : 1978	Methods of testing tar and bituminous materials : Determination of flash point and fire point (*first revision*)
9381 : 1979	Methods for testing tar and bituminous materials : Determination of FRAASS breaking point of bitumen
9382 : 1979	Methods for testing tar and bituminous materials : Determination of effect of heat and air by thin film oven test

3 DESCRIPTION

When used as bitumen modifier, selected polymer/ rubber or a blend of two or more modifiers shall have the following properties:

a) Compatible with bitumen,
b) Resist degradation at mixing temperature,
c) Capable of being processed by conventional mixing and laying machinery,
d) Produce required coating viscosity at application temperature, and
e) Maintain premium properties during storage, application and in-service.

NOTE — Homogeneity is very important for desired performance of polymer and rubber modified binders. Hence, these should be prepared at refinery or by appropriate industrial process and plant having high shear device. The use of higher shear mixer is essential.

3.1 Terminology

For the purpose of this standard, the definition given in IS 334 shall apply.

4 CLASSIFICATION

4.1 The polymer and rubber modified bitumen shall be classified into four types as given below :

a) Type A PMB(P) — Plastomeric thermoplastics based,
b) Type B PMB(E) — Elastomeric thermoplastics based,
c) Type C NRMB — Natural rubber and SBR latex based, and
d) Type D CRMB — Crumb rubber/treated crumb rubber based.

4.1.1 Type A, Type B and Type C shall be further classified into three grades according to their penetration value and Type D shall be further classified into three grades according to their softening point values as given below :

4.1.1.1 *Grades of Type A PMB(P)*

a) PMB (P)120
PMB(P)120 means that Type A PMB(P) corresponding to this grade has penetration value between 90 to 150.

b) PMB(P) 70
PMB(P) 70 means that Type A PMB(P) corresponding to this grade has penetration value between 50 to 90 .

c) PMB(P) 40
PMB(P) 40 means that Type A PMB(P) corresponding to this grade has penetration value between 30 to 50.

4.1.1.2 *Grades of Type B PMB(E)*
a) PMB(E) 120
b) PMB(E) 70
c) PMB(E) 40

4.1.1.3 *Grades of Type C NRMB*
a) NRMB 120
b) NRMB 70
c) NRMB 40

4.1.1.4 *Grades of Type D CRMB*
a) CRMB 50
b) CRMB 55
c) CRMB 60

NOTE — CRMB 50 means that Type D CRMB corresponding to this grade has softening point value 50 °C minimum.

5 REQUIREMENTS

5.1 Material and Manufacture

Over the years, different types of material have been investigated as additives for bitumen modifications. Some of them, which have been trial tested in India and countries abroad are polyethylene, ethylene vinyl acetate copolymers, ethylene methylacrylate, ethylene butylacrylate, styrene butadiene, styrene butadiene styrene block copolymer, natural rubber and crumb rubber from used truck tyres treated by gilsonite etc.

The PMB, shall be prepared by blending a suitable penetration grade bitumen or a feed stock (short residue) compatible with additives at refinery or any other plant having adequate (high shear) mixing and blending facilities. In case PMB is prepared using a penetration grade bitumen, the later shall conform to IS 73 and its revisions as applicable.

5.2 The material shall be homogenous and shall not foam, when heated at 170°C.

5.3 No mineral matter other than naturally present, in the ingredient materials, shall be used.

5.4 Modifier shall not de-mix on heating at 170°C or later during cooling.

5.5 The polymer modified bitumen of Type A, Type B, Type C and Type D shall also conform to the requirements given in Table 1, Table 2, Table 3 and Table 4 respectively.

Table 1 Requirements of Polymer Modified Bitumen PMB (P) (Plastomeric Thermoplastic Based) — Type A
(*Clause* 5.5)

Sl No.	Characteristics	Grade and Requirements			Method of Test, Ref to	
		PMB 120	PMB 70	PMB 40	IS No.	Annex
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Penetration at 25°C, 0.1 mm, 100g, 5s	90 to 150	50 to 90	30 to 50	1203	–
ii)	Softening point, (R&B), °C, *Min*	50	55	60	1205	–
iii)	FRAASS breaking point,[1] °C, *Max*	– 20	– 16	–12	9381	–
iv)	Flash point, COC, °C, *Min*	220	220	220	1209	–
v)	Elastic recovery of half thread in ductilometer at 15 °C, percent, *Min*	50	40	30		A
	Or					
	Complex modulus as ($G^*/\sin\delta$) as *Min* 1.0 kPa at 10 rad/s, at a temperature, °C	52	58	70		C
vi)	Separation, difference in softening point, R&B, °C, *Max*	3	3	3		B
vii)	Viscosity at 150 °C, Poise	1-3	2-6	3-9	1206 (Part 1)	–
viii)	Thin film oven tests and test on residue :					
	a) Loss in mass, percent, *Max*	1.0	1.0	1.0	9382	–
	b) Increase in softening point, °C, *Max*	7	6	5	1205	–
	c) Reduction in penetration of residue, at 25 °C, percent, *Max*	35	35	35	1203	–
	d) Elastic recovery of half thread in ductilometer at 25 °C, percent, *Min*	35	35	35	–	A
	Or					
	Complex modulus as ($G^*/\sin\delta$) as *Min* 2.2 kPa at 10 rad/s, at a temperature °C	52	58	70		C

NOTE — Requirement at Sl No. (v) is subject to agreement between the buyer and the seller.

[1] Relevant to snow bound cold climate areas.

Table 2 Requirements of Polymer Modified Bitumen PMB (E) (Elastomeric Thermoplastics Based) — Type B
(*Clause* 5.5)

Sl No.	Characteristics	Grade and Requirements			Method of Test, Ref to	
		PMB 120	PMB 70	PMB 40	IS No.	Annex
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Penetration at 25°C, 0.1 mm, 100 g, 5 s.	90 to 150	50 to 90	30 to 50	1203	-
ii)	Softening point (R&B), °C, *Min*	50	55	60	1205	-
iii)	FRAASS breaking point, [1] °C, *Max*	-20	-16	-12	9381	-
iv)	Flash point, COC, °C, Min	220	220	220	1209	-
v)	Elastic recovery of half thread in ductilometer at 15°C, percent, *Min*	70	70	70	-	A
vi)	Separation, difference in softening point (R&B), °C, *Max*	3	3	3	-	B
vii)	Viscosity at 150°C, Poise	1-3	2-6	3-9	1206 (Part 1)	-
viii)	Thin film oven test and tests on residue :					
	a) Loss in mass, percent, *Max*	1.0	1.0	1.0	9382	-
	b) Increase in softening point, °C, *Max*	7	6	5	1205	-
	c) Reduction in penetration of residue, at 25°C, percent, *Max*	35	35	35	1203	-
	d) Elastic recovery of half thread in ductilometer at 25°C, percent, *Min*	50	50	50	-	B

[1] Relevant to snow bound cold climate areas.

Table 3 Requirements of Polymer Modified Bitumen (NRMB) (Natural Rubber Based) — Type C
(*Clause* 5.5)

Sl No.	Characteristics	Grade and Requirements			Method of Test, Ref to	
		NRMB 120	NRMB 70	NRMB 40	IS No.	Annex
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Penetration at 25 °C, 0.1 mm, 100 g, 5 s	90-150	50-90	30-50	1203	
ii)	Softening point (R&B), °C, *Min*	45	50	55	1205	
iii)	FRAASS breaking point, [1], °C, *Max*	- 20	-16	-12	9381	-
iv)	Flash point, COC, °C, *Min*	220	220	220	1209	-
v)	Elastic recovery of half thread in ductilometer at 15°C, peercent, *Min*	50	40	30	-	A
vi)	Separation, difference in softening point (R&B) °C, *Max*	4	4	4	-	B
vii)	Viscosity at 150 °C, Poise	1-3	2-6	3-9	1206 (Part 1)	-
viii)	Thin film oven test and tests on residue :					
	a) Loss in mass, peercent, *Max*	1.0	1.0	1.0	9382	
	b) Increase in softening point °C, *Max*	7	6	5	1205	
	c) Reduction in penetration of residue, at 25°C, percent, *Max*	40	40	40	1203	-
	d) Elastic recovery of half thread in ductilometer at 25°C, percent, *Min*	35	25	20	-	A

[1] Relevant to snow bound cold climate area.

Table 4 Requirements of Polymer Modified Bitumen (CRMB) (Crumb and Modified Crumb Rubber Based)—Type D
(*Clause* 5.5)

Sl No.	Characteristics	Grade and Requirements			Method of Test, Ref to	
		CRMB 50	CRMB 55	CRMB 60	IS No.	Annex
(1)	(2)	(3)	(4)	(5)	(6)	(7)
i)	Penetration at 25°C, 0.1 mm, 100 g, 5 s.	<70	<60	<50	1203	–
ii)	Softening point (R&B), °C, *Min*	50	55	60	1205	–
iii)	Flash point, COC, °C, *Min*	220	220	220	1209	–
iv)	Elastic recovery of half thread in ductilometer at 15 °C, percent, *Min*	50	50	50	–	A
v)	Separation, difference in softening point (R&B), °C, *Max*	4	4	4	–	B
vi)	Viscosity at 150°C, Poise	1-3	2-6	3-9	1206 (Part 1)	–
vii)	Thin film oven test and tests on residue :					
	a) Loss in mass, percent, *Max*	1.0	1.0	1.0	9382	–
	b) Increase in softening point, °C, *Max*	7	6	5	1205	–
	c) Reduction in penetration of residue, at 25°C, percent, *Max*	40	40	40	1203	–
	d) Elastic recovery of half thread in ductilometer at 25 °C, percent, *Min*	35	35	35	–	A

6 SAMPLING AND CRITERIA FOR CONFORMITY

6.1 Lot

In any consignment, all the containers of PMB of same category and grade from the same batch of manufacture shall be grouped to constitute a lot.

6.2 The number of containers to be selected at random from the lot shall depend upon the size of the lot and shall be in accordance with Table 5.

Table 5 Number of Containers to be Selected
(*Clause* 6.2)

Sl No.	Lot Size (No. of Containers)	No. of Containers to be Selected
(1)	(2)	(3)
i)	Upto 50	2
ii)	51 to 100	3
iii)	101 to 200	4
iv)	201 to 300	5
v)	301 to 500	7
vi)	501 and above	10

6.3 From each of the containers selected as in **6.2** an average sample representative of the material in the container shall be drawn in accordance with the methods prescribed in IS 1201, taking all the precautions mentioned therein. The elastic recovery test and separation tests shall be conducted as per the methods prescribed in Annex A and Annex B. All these samples from individual containers shall be stored separately.

6.4 Number of Tests

6.4.1 All the individual samples shall be tested for separation, penetration, softening point and elastic recovery.

6.4.2 For the remaining characteristics, a composite sample prepared by mixing together equal quantities of PMB, as the case may be, from all individual samples taken from each sampled container, shall be tested.

6.5 Criteria for Conformity

6.5.1 The lot shall be considered as conforming to the requirements of this standard, if the conditions mentioned under **6.5.2** and **6.5.3** are satisfied.

6.5.2 From the test results of compatibility (separation test), penetration and elastic recovery, the mean (X) and the range (R) shall be calculated. The following conditions shall be satisfied :

a) $[X+0.6\,R]$ shall be greater than or equal to the minimum specification limit specified in Tables 1, 2, 3 and 4 ; and

b) $[X+0.6\,R]$ shall be less than or equal to the maximum specification limit specified in Tables 1, 2, 3 and 4.

6.5.3 The composite sample when tested for the characteristics mentioned in **6.4.2** shall satisfy the corresponding requirements of the characteristics given in Tables 1, 2, 3 and 4.

7 PACKING AND MARKING

7.1 Packing

Polymer and rubber modified bitumen of all types shall be suitably packed in a container as agreed to between the purchaser and the supplier.

7.2 Marking

Each container of PMB shall be legibly and indelibly marked with the following :

a) Manufacturer's name or trade-mark, if any;
b) Month and year of manufacture;
c) Type of the material and Grade; and
d) Batch number.

7.3 BIS Certification Marking

7.3 Each container may also be marked with the Standard Mark.

7.3.1 The use of the Standard Mark is governed by the provisions of the *Bureau of Indian Standards Act*, 1986 and the Rules and Regulations made there under. The details of conditions under which the license for the use of the Standard Mark may be granted to manufacturers or producers may be obtained from the Bureau of Indian Standards.

ANNEX A
(*Clause* 6.3 and *Tables* 1, 2, 3 *and* 4)

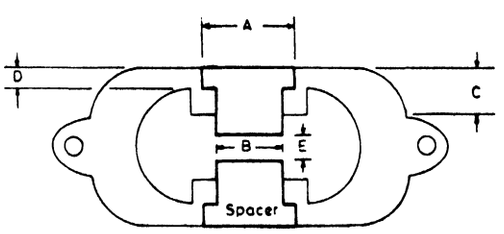
DETERMINATION OF ELASTIC RECOVERY

A-1 SIGNIFICANCE AND USE

This is a simple test intended to optimize dose of polymeric additive in bitumen and also help in assessing quality of PMB in laboratory.

A-2 PRINCIPLE

The elastic recovery of modified bitumen is evaluated by comparing recovery of thread after conditioning for 1 h at specified temperature and the specimen is elongated up to 10 cm deformation in a ductility machine. This is intended to assess degree of bitumen modification by Elastomeric additives. The cross-section of thread shall be as shown in Fig. 1.



A – 36.5 ± 0.1 mm
B – 30.0 ± 0.1 mm
C – 17.0 ± 0.1 mm
D – 10.0 ± 0.1 mm
E – 10.0 ± 0.1 mm

FIG. 1 DESIGN OF MOULD FOR ELASTIC RECOVERY

A-3 APPARATUS

A-3.1 Ductility Machine and Moulds — As per IS 1208 and Fig. 1.

A-3.2 Thermometer — Any standard thermometer (ASTM 63 °C) of equivalent range and accuracy shall be used.

A-3.3 Scissors — Any type of conventional scissors capable of cutting modified bitumen at the test temperature.

A 3.4 Scale — Any transparent scale of measuring up to 25 cm with ± 1 mm accuracy.

A-4 PROCEDURE

A-4.1 Prepare the test specimens in a set of three moulds as per dimensions given in Fig. 1 and condition as prescribed in test method of IS 1208. Elongate the test specimen at the specified rate to a deformation of 10 cm at a rate of 5 ± 0.25 cm/min. Immediately cut the test specimen into two halves at the mid-point using the scissors. Keep the test specimen in the water bath in an undisturbed condition for 1 h before testing.

A-4.2 After the 1 h time period, move the elongated half of the test specimen back into position near the fixed half of the test specimen so the two pieces of modified bitumen just touch. Record the length of the recombined specimen as *X*.

A-5 REPORT

Calculate the percent / elastic recovery by the following procedure :

$$\text{Elastic recovery (\%)} = \frac{10 - X}{10} \times 100$$

where

X = length of recombined specimen.

ANNEX B
(*Clause* 6.3 and *Tables* 1, 2, 3 *and* 4)

DETERMINATION OF SEPARATION

B-1 PRINCIPLE

The separation of modifier and bitumen during hot storage is evaluated by comparing the ring and ball softening point of the top and bottom samples taken from a conditioned, sealed tube of polymer modified bitumen. The conditioning consist of placing a sealed tube of modified bitumen in a vertical position at 163 ± 5°C in an oven for a period of 48 h. It provides a reference for determining the relative separation properties between different types of bitumen modifiers and their respective bitumens. Modified bitumen's relative stability to separation under storage in static conditions is determined in heated oven storage without agitation.

B-2 APPARATUS

B-2.1 Aluminum Tubes — 25.4 mm (1 inch) diameter and 136.7 mm (5.5 inch) length blind aluminum tubes (thickness of foil 1 mm), used to hold the test sample during the conditioning.

B-2.2 Oven, capable of maintaining 163 ± 5°C.

B-2.3 Freezer, capable of maintaining 6.7 ± 5°C.

B-2.4 Rack, capable of supporting the aluminum tubes in a vertical position in the oven and freezer.

B-2.5 Spatula and Hammer — The spatula must be rigid and sharp to allow cutting of the tube containing the sample when at a low temperature.

B-3 PROCEDURE

B-3.1 Place the empty tube, with sealed end down in the rack. Heat the sample carefully until sufficiently fluid to pour. Care should be taken to prevent localized overheating. Pass the molten sample through IS Sieve of 600 micron mesh size. After through stirring, pour 50.0 g into the vertically held tube. Fold the excess tube over two times, and crimp and seal.

B-3.2 Place the rack containing the sealed tubes in a 163 ± 5 °C oven. Allow the tubes to stand undisturbed in the oven for a period of 24 ± 4 h. At the end of the period, remove the rack from the oven, and place immediately in the freezer at 6.7 ± 5 °C, taking care to keep the tubes in a vertical position at all times. Leave the tubes in the freezer for a minimum of 4 h to solidify the sample completely.

B-3.3 Upon removing the tube from the freezer, place it on a flat surface. Cut the tube into three equal length portions with the spatula and hammer. Discard the centre section, and place the top and bottom portions of the tube into separate beakers,. Place the beakers into a 163 ± 5°C oven until the bitumen is sufficiently fluid to remove the pieces of aluminum tube.

B-3.4 After thoroughly stirring, pour the top bottom samples into appropriately marked rings for the ring and ball softening point test. Prepare the rings and apparatus according to details given in IS 1205. The top and bottom sample form the same tube should be tested at the same time in the softening point test.

B-4 REPORT

Report the difference, in °C, between the softening points of the respective top and bottom samples.

ANNEX C
(*Table* 1)

METHOD FOR DETERMINATION OF COMPLEX MODULUS

C-1 SCOPE

This method covers the determination of complex modulus (G*), Phase angle (sin δ) and G*/sin δ of modified bituminous binders. This standard is appropriate for unaged material and material aged in thin film oven or rolling thin film oven. Particulates materials in binder is limited to particles with longest dimensions less than 300 micrometer.

C-2 SIGNIFICANCE AND USE

The test temperature for this test is related to the temperature experienced by the pavement in the geographical area for which the use of binder is intended. The shear modulus is an indicator of stiffness or resistance of binder to deformation under load at specified temperature. The complex (G*) modulus and phase angle (sin δ) define the resistance to deformation of the binder in the visco-elastic region. The complex

modulus and phase angle are used to evaluate performance aspect of modified bitumen, where elastic recovery is insignificant.

C-3 SUMMARY OF TEST METHOD

This standard contains the procedure used to measure the complex modulus (G*), phase angle (sin δ) and shear modulus (G*/sin δ) of binders using a Dynamic Shear Rheometer and parallel plate test geometry. The standard is suitable for use when the complex modulus (G*) varies between 100 Pa and 10 MPa. The range of test temperature lies in between 35°C and 85°C depending upon grade, type and conditioning of the test sample. Test specimen of 1 mm thick, 25 mm diameter or 2 mm thick and 8 mm diameter are prepared between parallel metal plates. During the testing, one of the parallel plate is oscillated with respect to the other at pre-selected frequency and rotational deformation amplitudes. The required amplitudes depend upon the values of complex shear modulus of binders being tested. The test specimen is maintained at the test temperature within ±0.1°C by heating and cooling of upper and lower plates. The recommended frequency of testing is 10 rad/s. The complex modulus (G*) and phase angle (sin δ) are calculated as apart of the operation of the rheometer using software available with the equipment.

C-4 TEST EQUIPMENT

The test equipment comprises following items :

a) *Dynamic Shear Rheometer Test System* — A dynamic shear rheometer consisting of parallel metal plates, an environmental chamber, a loading device and a control and data acquisition system.

b) *Test Plates* — Metal test plates with polished surface, one 8 ± 0.5 mm in diameter and one 25 ± 0.05 mm in diameter. The base plate in some rheometer is a flat plate.

c) *Environmental Chamber* — A chamber for controlling the test specimen temperature by heating or cooling. The medium for heating and cooling the specimen in the environmental chamber is either a gas or liquid that will not affect binder properties. The temperature in the chamber may be controlled by the circulation of fluid or conditioned gas. When the air is used as medium a suitable drier must be included to prevent condensation of moisture on the plates and fixture.

d) *Temperature Controller* — A temperature controller capable of maintaining specimen temperature within ± 0.1°C for the test temperature ranging from 35 to 85°C is needed. A resistance thermal detector mounted inside the environmental chamber, in intimate with fixed plate with a range of 35 to 85°C readable to the nearest 0.1°C. The detector shall be used to control the temperature in the chamber and provide a continues read out of the temperature during the mounting, conditioning and testing of the specimens.

e) *Loading Device* — The loading device shall be capable to apply a sinusoidal oscillatory load to the specimen at the frequency of 10 rad/s. The loading device shall be capable of providing either a stress control or strain controlled load. If the load is strain controlled, the loading shall apply a cyclic torque sufficient to cause an angular rotational strain accurate to with in 100 micron radian of the strain specified. If the load is stress controlled, the loading device shall apply a cyclic torque accurate with to within 10 mN.m of the torque specified. Total system compliance to 100 N.m torque shall be < 2m. rad/N.m.

f) *Control and Data Acquisition System* — The control and data acquisition system shall provide a record of temperature, frequency, deflection angle and torque. The system shall be capable to record and calculate the shear stress, shear strain, complex shear modulus and phase angle of binder at specified test temperature.

g) *Specimen Mold* — A silicone rubber mold for preparation of test specimen.

h) *Specimen Trimmer* — A specimen trimmer with a straight edge at least 4 mm wide.

j) *Calibrated Temperature Detector* — A calibrated thermocouple, thermistor, or RTD with a thickness or diameter < 2.0 mm is suitable for measuring the temperature of a dummy specimen sample of binder. Thermocouples and thermistors are not reliable to ± 0.1°C unless calibrated to a standard traceable to the National Institute of Standard and Technology (NIST) and must be calibrated with associated meters or circuitry. Platinum RTDs are typically not suitable because they are too large to fit in the gaps between the plates in the DSR.

C-5 PREPARATION OF TEST SPECIMEN

A disk of binder with diameter equal to the oscillating plate (often called a spindle) of the DSR is needed for testing. There are two ways to prepare the sample for testing (1) Bitumen binder can be poured directly onto the spindle in sufficient quantity to provide the appropriate thickness of material, or (2) a mold can be used to form the disk of material to be tested. Then the disk can be placed between the spindle and fixed plate of DSR. In the first method, operator should have sufficient experience to apply exact quantity of binder. In the second method, binder is heated until fluid to pour. The heated binder is poured in to a rubber mold and allow to cool. The mold consisting binder may be

placed in a refrigerator until it attains solid consistency. Then the sample is removed from the mold and placed between the fixed plate and oscillating spindle of the DSR. The excess binder beyond the edge of the spindle should be trimmed. Regardless of the method used for preparation of the specimen, the final step in preparing the specimen is to slightly readjust the gap between the spindle and the lower plate so that a slight bulge is evident near the edge of the spindle. This step is normally occur immediately prior to the testing. The thickness of the bitumen binder disk sandwiched between the spindle and fixed plate must be carefully controlled. The proper specimen thickness is achieved by adjusting the gap between the spindle and fixed plate. This gap must be set before mounting the binder sample but while spindle and base plate are mounted in the rheometer at the test temperature. The gap is adjusted by means of a micrometer wheel. The micrometer wheel is graduated usually in units of micron. Turning the wheel allows precise positioning the spindle and base plate related to each other. On some rheometer, the micrometer wheel moves the spindle down. On other, it moves the base plate up. Thickness of the gap use depend on the test temperature. High test temperature of 46°C or greater require a small gap of 1 mm. High temperature measurement require a large spindle (25 mm) and low temperature a small spindle (8 mm). With the specimen mounted, the operator shall set the gap at the desired value of 1 000 or 2 000 micron. After the specimen is trimmed flush with upper plate, the extra 50 micron is dialed so that gap is exactly at the desired value and specimen bulges slightly.

C-6 TEST PROCEDURE

Bring the specimen to the test temperature ±0.1°C. After the sample is correctly in place and test temperature appear stable then allow the specimen for 10 min at the set temperature of the specimen to equilibrate. The actual temperature equilibration time is equipment dependent and should be checked using a dummy specimen with very accurate temperature sensing capabilities.

When operating in a strain control mode, testing consist of using the rheometer software to select appropriate strain value as under:

Material	*kPa*	*Target Strain,%*	*Strain Range, %*
Original binder	1.0(G*/sin δ)	12	9-15
TFOT residue	2.2(G*/sin δ)	10	8-12

When operating in a stress controlled mode, select an appropriate stress level using software as under :

Material	*kPa*	*Target Stress, kPa*	*Stress Range kPa*
Original binder	1.0(G*/sin δ)	0.12	0.09-0.15
TFOT residue	2.2(G*/sin δ)	0.22	0.18-0.26

Testing consist of using rheometer software to set the DSR to apply a constant oscillating stress and recording the resulting strain and time lag. The specification require oscillation speed to 10 rad/s, which is approximately 1.59 Hz. A computer is used with DSR to control test parameter and record test results. The operator need not worry about setting the value of applied stress. Instead, the operator should set the approximate value of shear strain. Shear strain values vary from 1-12 percent and depend on the stiffness of the binder being used . Relatively soft materials tested at high temperature are tested at strain values of approximately 10-12 percent . Hard materials are tested at strain value of about 1 percent. In the initial stage of the test, rheometer measures the stress required to achieve the set shear strain and then maintains this stress very precisely during the test. The shear strain can vary small amounts from the set value to achieve the constant stress. Variation in shear strain is normally control led by rheometer software . In the beginning of the test, the sample is first conditioned by loading the specimen for 10 cycles and then 10 additional cycles and then are applied to obtained test data. The rheometer software automatically compute and report values of complex modulus (G*) phase angle (sin δ).

C-7 INTERPRETATION OF RESULTS AND DATA PRESENTATION

The complex modulus (G*) and phase angle (sin δ) decrease with increasing shear strain. A linear region may be defined at small region where the modulus is relatively independent of shear strain. This region will vary with magnitude of complex modulus . The linear region is defined as range in strains where the complex modulus is 95 percent or more of the 0 strain value. The shear stress varies nearly from 0 at the centre of the plates to a maximum at the extremities of the plate perimeter. The shear stress is calculated from the applied or measured torque , measure or applied strain and the geometry of the test specimen. For the present specification only value of G* and sin δ are required. A complete report includes following parameters :

a) G* to the nearest three significant figures,

b) sin δ to the nearest 0.1 degrees,

c) test plate size to nearest 0.1 mm and gap to nearest 1 μm,

d) test temporary to the nearest 0.1°C,

e) test frequency to the nearest 0.1 rad/s, and

f) strain amplitude to the nearest 0.01 percent.

The test temperature as per requirement of specification for complex modulus value of 1 kPa(G*/sin δ) for original binder and 2.2 kPa (G*/sin δ) for residue of thin film oven test shall be calculated from the plot of (G*/sin δ) and temperature for compliance of specification.

ANNEX D
(Foreword)

COMMITTEE COMPOSITION

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