IS 10016-6 (2000): Methods of Test for Polybutadiene Rubbers, Part 6: Evaluation of Vulcanization Characteristics of Polybutadiene Rubbers (BR : 2) [PCD 13: Rubber and Rubber Products]

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भारतीय मानक

पोलीब्यूटाडाइन रबड़ की परीक्षण विधियां

भाग 6 पोलीब्यूटाडाइन रबड़ के वल्कीकरण लक्षणों का मूल्यांकन (बी आर : 2)

Indian Standard

METHOD-SOFTESTFORPOLYBUTADIENERUBBERS

PART 6 EVALUATION OF VULCANIZATION CHARACTERISTICS OF POLYBUTADIENE RUBBERS (BR : 2)

ICS 83.060

BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

May 2000 Price Group 2

FOREWORD

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

In this series methods of test for polybutadiene rubbers are given. Various other methods of test for polybutadiene rubbers are covered in following parts:

Part1 Method of taking out test portions from sample bales

Part 2 Determination of ash

Part 4 Determination of CIS, Trans and Vinyl structure

Part5 Determination of gel content

The committee has decided to prepare common methods of test for synthetic rubber under SR (synthetic tubber) series and will be applicable to all types of synthetic rubbers being produced indigenously. After preparation of common methods of test, Part 1, Part 2 and Part 5 would be withdrawn.

In preparation of this standard assistance has been drawn from ISO 2476 : 1996 'Rubber, butadiene (BR) — Solution-polymerized types — Evaluation procedure' published by International Organization for Standardization (ISO) and ASTM D 3189 Rubber-Evaluation Solution BR (Polybutadiene Rubber), published by American Standards for Testing and Materials.

In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values *(revised)*'.

Indian Standard

METHODSOFTESTFORPOLYBUTADIENERUBBERS

PART 6 EVALUATION OF VULCANIZATION CHARACTERISTICS OF POLYBUTADIENE RUBBERS (BR : 2)

1 SCOPE

This standard prescribes standard materials, equipment and processing methods for evaluating vulcanization characteristics of solution-polymerized butadiene rubbers (BR), including oil-extended types (OEBR).

2 NORMATIVE REFERENCES

The following standards contain provisions which through reference in this 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*
1675: 1971	Stearic acid, technical *(first revision)*
3400 (Part 1) : 1987	Methods of test for vulcanized rubbers: Part 1 Tensile stress-strain properties (second *revision)*
3399 : 1993	Zinc oxide for rubber industry *(second revision)*
3660	Methods of test for natural rubber:
(Part 3) : 1988	Determination of ash (NR:3) *(second revision)*
(Part 4) : 1988	Determination of total copper (NR:4) *(second revision)*
(Part 5) : 1988	Determination of manganese (NR:5) (second *revision)*
(Part 6) : 1988	Determination of rubber hydrocarbon (NR:7) *(second revision)*
(Part 7) : 1988	Determination of Mooney viscosity (NR: 8)
(Part 8) : 1999	Mixing and vulcanizing of rubber in standard compound *(second revision)*
5599 : 1999	Rubber — Raw, natural and synthetic — Methods for sampling and sample preparation *(first revision)*
7069 : 1986	Benzothiazyl-2-cyclohexyl sulphenamide (first *revision)*
7497 : 1985	High abrasion furnace (HAP) carbon black (first *revision)*
8851 : 1994	Sulphur for rubber industry *(first revision)*
11720	Methods of test for synthetic rubber:
(Part 2) : 1989	Measurement of vulcanization characteristics with oscillating disc curemeter (SR:2)
(Part 3) : 1993	Determination of Mooney viscosity
(Part 4) : 1993	Determination of volatile matter
13867 : 1993	Rubber standard temperature, humidities and times for the conditioning and testing of test pieces

3 TEST RECIPE FOR EVALUATION OF VULCANIZATION CHARACTERISTICS

3.1 Standard Test Formula

The standard test formula is given below:

Sl No.	*Material*	*Parts by Mass*	
		Non-oil Extended	Oil Extended
i)	Butadiene rubber (BR)	100.00	100.00
ii)	Zinc oxide (see IS 3399)	3.00	3.00
iii)	Oil furnace black (HAP)" (No. 330) (see IS 7497)	60.00	60.00
iv)	Stearic acid (see IS 1675)	2.00	2.00
v)	Petroleum oil (naphthenic)[2] (see ASTM Type 103)	15.00	—
vi)	Sulphur (see IS 885 1)	1.50	1.50
vii)	CBS (see IS 7069) or TBBS[3] (for method D) (min)	0.90	0.90
viii)	Calculated density, mg/m^3	1.11	1.14 to 1.16 [4]

[1] The current Industry Reference Black may be used in place of HAF carbon black but this may give slightly different results.

[2] ASTM Type 103 oil has the following characteristics:

Kinematic viscosity at 100°C, 16.8 ± 1.2 mm^2/s

Viscosity gravity constant, 0.889 ± 0.002

The Viscosity Gravity Constant (VGC) is calculated from the Saybolt Universal viscosity at 37.8°C and the relative density at 15.5/15.5°C. Use the following equation to calculate the VGC from the measured properties:

$$VGC = \frac{10d - 1.075\,2\log_{10}\nu - 38}{10 - \log_{10}\nu - 38}$$

where

d = relative density at 15.5/15.5°C;

ν = Saybolt Universal Viscosity at 7.8°C.

[3] N-ter-butyl-2-benzothiazile sulphenamide as per Material Reference No. 384.

[4] Based on 37.5 percent oil-extended BR.

3.2 Procedure

3.2.1 *Equipment and Procedure*

Equipment and procedure for the preparation, mixing and vulcanization shall be in accordance with different parts of IS 3660 (Part 8) wherever applicable.

NOTE — Details of a suitable internal mixer are given in Annex A.

3.2.2 ***Mixing Procedure***

The following four mixing procedures are specified:

Method A	Internal mixer for initial and final mixing;
Method B	Internal mixer for initial and mill for final mixing;
Method C	Mill mixing; and
Method D	Miniature internal mixing.

NOTE — These procedures may give different results.

The mill handling of solution butadiene rubbers is more difficult than for other rubbers and mixing is best accomplished by using an internal mixer. Because of the difficulty of mill mixing butadiene rubber, it is recommended that one of the internal mixer procedures (methods A, B or D) be used where such equipment is available. With some types of butadiene rubber it is not possible to get a satisfactory mix using the mill mixing procedure.

3.2.2.1 Method A

Internal Mixer for initial and final mixing

Stage 1 — Initial mixing procedure

		Duration (min)	***Cumulative*** (min)
a)	Adjust the temperature, speed and ram pressure of the internal mixer to achieve the condition outlined in **3.2.2.1(e).** Close the discharge gate, start the rotor and raise the ram.	0	0
b)	Charge one-half of the tubber, the zinc oxide, the carbon black, the oil (omit from Formula 2 for OEBR), the stearic acid and the balance of the rubber. Lower the ram.	0.5	0.5
c)	Allow the batch to mix.	3.0	3.5
d)	Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	0.5	4.0
e)	Discharge the batch at a temperature of 170°C or after a total time of 6 min, whichever occurs first.	2.0	6.0
f)	Immediately pass the batch three times through a laboratory mill with a mill opening of 5.0 mm and a temperature of 50 ± 5°C. Check, weigh the batch. If the batch weight differs from the theoretical value by more than 0.5 percent, discard the batch.		
g)	Rest the batch for at least 30 minutes and up to 24 h.		

Stage 2 — Final mixing procedure

		Duration (min)	***Cumulative*** (min)
a)	Cool the internal mixer to a temperature of 40 ± 5°C with full cooling water on the rotors. Start the motor and raise the ram.	0	0
b)	Leave the cooling water on and the steam off. Roll all the sulphur and the CBS into one-half of the masterbatch and charge into the mixer. Add the remaining portion of the masterbatch. Lower the ram:	0.5	0.5
c)	Allow the batch to mix until a temperature of 110°C or a total time of 3 min is reached, whichever occurs first.	2.5	3.0
d)	Immediately pass the batch through a laboratory mill with a mill opening set at 0.3 mm and at a temperature of 50 ± 5°C.		
e)	Pass the rolled batch endwise through the rolls six times.		
f)	Sheet the batch to approximately 6 mm and check, weigh. Remove sufficient sample for curemeter testing, if required.		
g)	Sheet the batch to approximately 2.2 mm for preparing test slabs or to the appropriate thickness for preparing ring specimen.		

3.2.2.2 ***Method B***

Internal mixer for initial and mill for final mixing

Stage 1 — Initial mixing procedure

Prepare the initial mix in accordance with procedure outlined in **3.2.2.1.** (Stage 1)

Stage 2 — Final mill mixing procedure

Adjust the mass of all material (that is masterbatch, sulphur and CBS) to give a final batch mass of four times the formula mass,

NOTE — AU null openings should be adjusted to maintain a good rolling bank at the nip of the rolls mixing. If this is not obtained with the settings **specified** hereunder, small adjustments to **mill** openings may be **necessary:**

		Dura-tion (min)	*Cumu-lative* (min)
a)	Set and maintain the mill roll temperature at **40** ± 5°C and the mill opening at 1.5 mm. Band the masterbatch and band round the front roll.	1.0	1.0
b)	Add the sulphur and the CBS slowly to the batch.	1.0	2.0
c)	Make six **3/4** cuts from each side.	1.5	3.5
d)	Cut the batch from the mill. Set tbe **mill** opening to 0.8 **mm** and **pass** the **rolled** batch end-wise through the rolls six times.	**1.5**	5.0
e)	Sheet the batch to approximately 6 mm and check, weigh. Remove sufficient sample for **curemeter** testing, if **required.** If the weight differs **more** than 0.5 percent **from** the theoretical **value,** discard the batch.		
f)	Sheet the **batch** to approximately 2.2 mm for preparing test slabs or to the **appropriate** thickness for **preparing** specimens. Cool the compound on a flat **dry metal surface** at 23 ± 3°C for **16 h and RH ≤ 55 percent.**		

3.2.23 ***Method C*** — Mill mixing procedure

The standard laboratory batch mass, in grams, shah be based on four times the formula mass. Adjust the mill roll cooling conditions to maintain a temperature of 35 ± 5°C throughout the mixing operations.

NOTES

1 Methods A and B, which give better diipension of the ingredients. are to be preferred if an internal mixer is available.

2 All mill openings should be adjusted to maintain a good **rolling** bank at the nip of the **rolls** during mixing. If this is not obtained with the settings **specified hereunder,** small adjustments to mill openings may **be** necessary:

		Duration (min)	*Cumu-lative* (min)
a)	Band the rubber with the mill opening **set at** 1.3 mm.	1	1
b)	Add the **zinc** oxide and the stearic acid evenly **across** the **rolls.** Make **two 3/4** cuts **from each** side.	2	3
c)	Add the carbon black evenly across the rolls at a uniform rate. When about half the black has been incorporated. open the **rolls** to 1.8 mm and **then add** the remainder of the black. Make hvo **3/4** cuts **from** each side, allowing 30 s between each cut. Be certain to add **the** black that has dropped into the **mill** pan.	**15 to 18**	**18 to 21**
d)	Add the oil (omit **from** Formula 2 for OEBR) very slowly drop by drop.	8 to 10	26 to 31
e)	Add the sulphur and the CBS.	2	**28 to 33**
f)	Make six successive **3/4** cuts from each side.	2	**30 to 35**
g)	Cut the batch **from** the mill, set the mill opening to 0.8 mm and pass the rolled batch end-wise through the rolls six times.	2	**32 to 37**
h)	Sheet the **batch** to **approximately** 6 mm and check, weigh the batch. Remove sufficient sample for **curemeter** testing if **required**. The weight should not differ more than 0.5 percent **from** the theoretical value, if more discard the batch.		
j)	Sheet the batch to approximately 2.2 mm for preparing teat **slabs** or to the appropriate thickness for preparing ring specimens. **Cool** the compound on a flat dry metal surface at 23 ± **3°C** for l-24 h and RH ≤ 55 percent.		

NOTE — It is sometimes easier and mote practical to combine steps **3.2.2.3(c)** and **3.2.2.3(d)** above, either by premixing the oil and black *together-and* **then** *adding the oiled* black **directly** to the **rubber** on the **mill** as described in **4.2.2.3(c)** and thus omitting **4.2.2.3(d)** or by adding carbon black and oil **alternately.**

3.2.2.4 ***Method D*** — Miniature Internal Mixer **(MIM)** procedure

a) For general mixing procedure.

b) Mix with the head temperature of the miniature internal mixer maintained at **60** ± 3°C and the rotor speed set at 6.3 **to** 63 rpm.

c) Prepare the rubber by passing it through a mill one time with the temperature set at 40 ± **5°C** and an opening that would give approximately 5 mm thick sheet. Cut the sheet into strips that are approximately 25 mm wide.

		Dura-tion (min)	*Cumu-lative* (min)
a)	Charge the mixing chamber with the rubber strips lower the ram, and start timer.	0.0	***0.0***
b)	Masticate the rubber.	0.5	***0.5***
c)	Raise the ram, add zinc oxide, sulphur, stearic acid and TBBS that have previously been blended taking cam to avoid any loss.	1.0	***1.5***
d)	Add portions of carbon black and oil alternately sweep the orifice and lower **the** ram.	1	***2.5***
e)	Allow the batch to mix, raising the ram momentarily to sweep down if necessary.	6.5	***9.0***
f)	Turn off the motor, raise the ram, remove the mixing chamber, and discharge the batch. Record the maximum batch temperature indicated if **desired.**		

g) Pass the batch through a mill at 40 ± 5°C and 0.5 mm opening once then twice at 3 mm opening.

h) Check the batch mass and record. If it differs from the theoretical value by more than 0.5 percent, discard the batch.

4 CONDITIONING OF COMPOUNDS

Conditioning all batches produced by methods A, B, C or D at a standard laboratory temperature for 1 to 24 h after mixing and prior to vulcanizing as per IS 13867.

5 EVALUATION OF WLCANIZATION CHARACTERISTICS

5.1 Evaluation According to. Stress-Strain Properties

5.1.1 Vulcanize sheets at 145°C for 25, 35 and 50 min.

Alternatively, vulcanize sheet at 150°C for 20, 30 and 50 min. The recommended cure time for the miniature internal mixture compound if 35 min at 145°C.

5.1.2 Condition the vulcanized sheets for 16 to 96 h at a standard laboratory temperature and humidity defined in IS 13867.

5.13 Measure the stress-strain properties in accordance with IS 3400 (Part 1).

5.2 **Evaluation According to Oscillating Disc Curemeter Test**

Measure the following standard test parameters:

$$M_L, M_H, ts1, t'_c (50) \text{ and } t'_c = (90)$$

in accordance with IS 11720 (Part 2), using the following test conditions:

Oscillation frequency	: 1.7 Hz (100 cycles/ min)
Amplitude of oscillation	: 1°C arc
Selectivity.	: To be selected to give at least 75 percent full scale deflection
	NOTE — With some rubber, 75 percent may not be attainable.
Die temperature	: 160 ± 1°C
Pre-heat time	: None

ANNEX A

. (Clause 3.2.1)

INTERNAL MIXER

A-l The internal mixer should have a nominal capacity of approximately 1000 CC.

A-2 The rotor speed(s), ram pressure and coolant flow of the internal mixer should be such that the conditions specified in **3.2.2.1(e),** 3.2.2.2(c) and 3.233(e) will be accomplished.

A-3 The batch size should be the nominal capacity of the internal mixer measured in cubic centimetres, multiplied by the density (+ 0, – 10 percent).

NOTE — If an old or worn internal mixer is used. the batch muss should be increased accordingly.

Bureau of Indian Standards

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Review of Indian Standards

Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Handbook' and 'Standards: Monthly Additions'.

This Indian Standard has been developed from Doc: No. PCD 14 (1058).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Printed at Simco Printing Press, Delhi