



Designation: D 3189 – 99

Standard Test Methods for Rubber—Evaluation of Solution BR (Polybutadiene Rubber)¹

This standard is issued under the fixed designation D 3189; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods specify the standard materials, test formula, mixing procedures, and test methods for evaluation of butadiene rubber (BR) made by polymerization in solution.

1.2 The values stated in SI units are to be regarded as the standard. The values in parentheses are for information only.

1.3 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

D 88 Test Method for Saybolt Viscosity²

D 412 Test Methods for Vulcanized Rubber and Thermoplastic Rubbers and Thermoplastic Elastomers—Tension³

D 1646 Test Method for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)³

D 2084 Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter³

D 2161 Practice for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity⁴

D 2501 Test Method for Calculation of Viscosity-Gravity Constant (VGC) of Petroleum Oils⁴

D 3040 Practice for Preparing Precision Statements for Standards Related to Rubber and Rubber Testing⁵

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets³

D 3896 Practice for Rubber from Synthetic Sources—Sampling³

D 4483 Practice for Determining Precision for Test Method

Standards in the Rubber and Carbon Black Industries³

D 5289 Test Method for Rubber Property—Vulcanization Using Rotorless Cure Meters³

NOTE 1—The specific dated edition of Practice D 3040 that prevails in these test methods is referenced in the precision section.

3. Significance and Use

3.1 These test methods are intended mainly for referee purposes but may be used for quality control of rubber production. They may also be used in research and development work and for comparison of different rubber samples in a standard formula.

3.2 These tests may be used to obtain values for customer acceptance of rubber.

4. Standard Test Formula

4.1 Standard Formulas:

Material	IRM-SRM No.	Quantity, Parts by Mass
BR	...	100.00
Zinc oxide	A	3.00
Sulfur	A	1.50
Stearic acid	A	2.00
Current IRB	A	60.00
TBBS ^B	A	0.90
ASTM Type 103 petroleum oil ^C	A	15.00
Total		182.40
Batch Factor for mill mix ^D		4.0
Batch Factor for internal mixer ^D		7.13
Batch Factor for MIM Mix (Cam Head) ^{D,E}		0.40
Batch Factor for MIM Mix (Banbury Head) ^{D,E}		0.34

^AUse the latest IRM/SRM.

^BN-tert-butyl-2-benzothiazolesulfenamide.

^CViscosity is $16.8 \pm 1.2 \text{ mm}^2/\text{s}$ at 100°C in accordance with Test Methods D 88 and Practice D 2161. Viscosity Gravity Constant is 0.889 ± 0.002 in accordance with Test Method D 2501. Available in 1 and 5-gal (3.8 and 19-L) quantities from Sun Oil, Industrial Products Dept., 1608 Walnut St., Philadelphia, PA 19103.

^DFor mill and internal mixer batches, weigh the rubber, carbon black and oil to the nearest 1.0 g, the sulfur and accelerators to the nearest 0.02 g, and the other compounding materials to the nearest 0.1 g. For MIM batches, weigh the rubber and material, blend to the nearest 0.01 g and the individual compounding materials, if used, to the nearest 0.001 g.

^EFor the MIM procedure, it is recommended that a blend of compounding materials, excluding carbon black and oil, be prepared to improve accuracy in the weighing of these materials. The material blend is prepared by blending a proportional mass of each material in a dry powder blender such as a biconical blender or vee blender. A mortar and pestle may be used for blending small quantities.

¹ These test methods are under the jurisdiction of ASTM Committee D-11 on Rubber and are the direct responsibility of Subcommittee D11.23 on Synthetic Rubber.

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² Annual Book of ASTM Standards, Vol 04.04.

³ Annual Book of ASTM Standards, Vol 09.01.

⁴ Annual Book of ASTM Standards, Vol 05.01.

⁵ Discontinued—see 1986 Annual Book of ASTM Standards, Vols 09.01 and 09.02.

5. Sample Preparation

5.1 For tests intended for referee purposes obtain and prepare the test samples in accordance with Practice D 3896.

6. Mixing Procedures

6.1 The following four mixing procedures are offered:

6.1.1 *Test Method A*— Internal Mixer for Initial and Final Mix,

6.1.2 *Test Method B*— Initial Mixer with Final Mill Mix,

6.1.3 *Test Method C*— Mill Mix, and

6.1.4 *Test Method D*— Miniature Internal Mixer Mix.

NOTE 2—The compound may be prepared either on a mill or in an internal mixer or in a miniature internal mixer although slightly different results may be obtained.

NOTE 3—Since the mill handling characteristics of the solution polybutadiene rubber are somewhat more difficult than for other polymers; the use of one of the internal mixer procedures is recommended (Test Method A, B, or D). The mill procedure (Test Method C) may be used provided good carbon black dispersion is obtained.

6.2 *Test Method A—Internal Mixer for Initial and Final Mix:*

6.2.1 For general mixing procedure, refer to Practice D 3182.

6.2.2 *Mixing Cycle*— Initial Mix:

	Dura- tion, min	Accu- mula- tive, min
6.2.2.1		
Adjust the internal mixer temperature to achieve the discharge conditions outlined in 6.2.2.5. Close the discharge gate, start the rotor, at 8.0 rads/s (77 r/min) and raise the ram.	0	0
6.2.2.2		
Charge one half the rubber, all of the zinc oxide, carbon black, oil, stearic acid, and then the other one half of the rubber. Lower the ram.	0.5	0.5
6.2.2.3		
Allow the batch to mix.	3.0	3.5
6.2.2.4		
Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	0.5	4.0
6.2.2.5		
Allow the batch to mix until a temperature of 170°C (338°F) or a total mixing time of 6 min is reached, whichever occurs first. Discharge the batch. Subtotal time (min) max.	2.0	6.0
6.2.2.6		
Check the batch mass and record. If it differs from the theoretical value by more than 0.5 %, discard the batch.		
6.2.2.7		
Pass the batch immediately through the standard laboratory mill three times, set at 6.0 mm (0.25 in.) and 40 ± 5°C (104 ± 9°F).	1.0	7.0

6.2.2.8

Allow the stock to rest for 1 to 24 h.

6.2.3 *Final Mix:*

	Dura- tion, min	Accu- mula- tive, min
6.2.3.1		
Adjust the internal mixer to 40 ± 5°C (104 ± 9°F). Turn off steam and turn on full cooling water to the rotors, start the motor at 8.0 rads/s (77 r/min) and raise the ram.		0
6.2.3.2		
Charge one half the masterbatch, with all the sulfur and accelerator rolled into this portion of the masterbatch before feeding to the mixer. Add the remaining portion of the masterbatch. Lower the ram.	0.5	0.5
6.2.3.3		
Allow the batch to mix until a temperature of 110 ± 5°C (230 ± 9°F) or a total mixing time of 3 min is reached, whichever occurs first. Discharge the batch. Subtotal Time (min) max.	2.5	3.0
6.2.3.4		
Check the batch mass and record. If it differs from the theoretical value by more than 0.5 %, discard the batch.		
6.2.3.5		
Set with the rolls of a standard laboratory mill maintained at 40 ± 5°C (104 ± 9°F) and at 0.8 mm (0.032 in.) opening. Pass the rolled stock endwise through the mill six times.	2.0	5.0
6.2.3.6		
Open the mill to give a minimum stock thickness of 6 mm (0.25 in.) and pass the stock through the rolls four times, folding it back on itself each time.	1.0	6.0
6.2.3.7		
Cut enough sample to allow testing of compound viscosity in accordance with Test Method D 1646 or vulcanizing characteristics in accordance with Test Method D 2084, as described in 8.2, or both, if these are desired.		
6.2.3.8		
Set the mill opening to give a finished gage of 2.2 mm (0.085 in.). Sheet off the compound from the mill. Cool on a flat, dry metal surface.		
6.2.3.9		
For routine laboratory testing, condition the sheeted compound for 1 to 24 h at 23 ± 3°C (73.4 ± 5.4°F) and a relative humidity not greater than 55 %. For maximum precision, condition for 1 to 24 h in a closed container to prevent absorption of moisture from the air or in an area controlled at 35 ± 5 % relative humidity.		
6.3 <i>Test Method B—Internal Mixer for Initial Mix With Final Mill Mix:</i>		
6.3.1 For general mixing procedure refer to Practice D 3182.		
6.3.2 <i>Mixing Cycle—Initial Mix:</i>		
6.3.2.1 Prepare the initial mix in accordance with the procedure outlined in 6.2.2.		