



Designation: D 3189 – 05

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:²

D 88 Test Method for Saybolt Viscosity

D 412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

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

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

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

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

D 3040 Discontinued 1988; Practice for Preparing Precision Statements for Standards Related to Rubber and Rubber Testing; Replaced by D 4483³

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

D 3896 Practice for Rubber From Synthetic Sources—Sampling

D 4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

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

D 6204 Test Method for Rubber—Measurement of Unvulcanized Rheological Properties Using Rotorless Shear Rheometers

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*—See Table 1.

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*—See Table 2 and Table 3.

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

Current edition approved Dec. 1, 2005. Published January 2006. Originally approved in 1973. Last previous edition approved in 1999 as D 3189 – 99.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Withdrawn.



TABLE 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

^A Use the latest IRM/SRM.

^B N-*tert*-butyl-2-benzothiazolesulfenamide.

^C Viscosity 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.

^D For 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.

^E For 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.

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

6.2.1.1 If required, cut samples from the batch to allow testing of compound viscosity and processability in accordance with Test Methods D 1646 or D 6204, and vulcanization characteristics in accordance with Test Methods D 2084 or D 5289. Condition the samples in accordance with Practice D 3182.

6.2.1.2 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.

6.2.1.3 For routine laboratory testing, condition the sheeted compound for 1 to 24 h at $23 \pm 3^\circ\text{C}$ ($73.4 \pm 5.4^\circ\text{F}$) 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 $35 \pm 5\%$ relative humidity.

6.3 Test Method B—Internal Mixer for Initial Mix With Final Mill Mix:

6.3.1 For general mixing procedure refer to Practice D 3182.

6.3.2 Mixing Cycle—Initial Mix:

6.3.2.1 Prepare the initial mix in accordance with the procedure outlined in Table 2.

6.3.3 Final Mix—See Table 4.

6.3.3.1 Check the batch mass and record. If it differs from the theoretical value by more than 0.5 %, discard the batch.

6.3.3.2 If required, cut samples from the batch to allow testing of compound viscosity and processability in accordance with Test Methods D 1646 or D 6204, and vulcanization

characteristics in accordance with Test Methods D 2084 or D 5289. Condition the samples in accordance with Practice D 3182.

6.3.3.3 Set the mill opening to give a finished gage of 2.2 mm (0.085 in.). Sheet off the stock from the mill. Cool on a flat, dry metal surface.

6.3.3.4 For routine laboratory testing, condition the sheeted compound for 1 to 24 h at $23 \pm 3^\circ\text{C}$ ($73.4 \pm 5.4^\circ\text{F}$) 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 $35 \pm 5\%$ relative humidity.

NOTE 4—If the amount of one compound based on two times the formula is not enough for the test specimens, another final compound should be prepared.

6.4 Test Method C—Mill Procedure:

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

6.4.1.1 Pre-blend the zinc oxide and stearic acid in a single container.

6.4.1.2 To simplify the addition of the oil, prepare an oil-carbon black paste as follows: Add the oil to a 100 mL beaker. While stirring the oil with a spatula, slowly add 30 g of the carbon black until a paste is formed.

6.4.1.3 Adjust the mill cooling conditions to maintain a temperature of $35 \pm 5^\circ\text{C}$ throughout the mixing operations. Adjust the mill openings as necessary to maintain a good rolling bank at the nip of the rolls during the mixing.

6.4.2 Mixing Cycle—See Table 5.

6.4.2.1 Sheet the batch to 6 mm thickness and weigh it. If the weight differs by more than 0.5 % from the theoretical, discard it.

6.4.2.2 If required, cut samples from the batch to allow testing of compound viscosity and processability in accordance with Test Methods D 1646 or D 6204, and vulcanization characteristics in accordance with Test Methods D 2084 or D 5289. Condition the samples in accordance with Practice D 3182.

6.4.2.3 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.

6.4.2.4 For routine laboratory testing, condition the sheeted compound for 1 to 24 h at $23 \pm 3^\circ\text{C}$ ($73.4 \pm 5.4^\circ\text{F}$) 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 $35 \pm 5\%$ relative humidity.

6.5 Test Method D—Miniature Internal Mixer (MIM) Procedure:

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

6.5.2 Mix with the head temperature of the miniature internal mixer maintained at $60 \pm 3^\circ\text{C}$ ($140 \pm 5^\circ\text{F}$) and the rotor speed set at 6.3 to 6.6 rad/s (60 to 63 r/min).

6.5.3 Prepare the rubber by passing it through a mill one time with the temperature set at $40 \pm 5^\circ\text{C}$ ($109 \pm 9^\circ\text{F}$) and an opening that would give approximately 5 mm (0.2 in.) thickness.