Designation: D3189 – 06 (Reapproved 2016)

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

This standard is issued under the fixed designation D3189; 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 cover 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]

D88 Test Method for Saybolt Viscosity

D412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

D1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)

D2084 Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter

D2161 Practice for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity

D2501 Test Method for Calculation of Viscosity-Gravity Constant (VGC) of Petroleum Oils

D3040 Practice for Preparing Precision Statements for Standards Related to Rubber and Rubber Testing (Withdrawn 1987)[3]

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D3896 Practice for Rubber From Synthetic Sources—Sampling

D4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries

D5289 Test Method for Rubber Property—Vulcanization Using Rotorless Cure Meters

D6204 Test Method for Rubber—Measurement of Unvulcanized Rheological Properties Using Rotorless Shear Rheometers

NOTE 1—The specific dated edition of Practice D3040 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 D3896.

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*—Internal Mixer Initial Mix 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

[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 Rubbers.

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[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] The last approved version of this historical standard is referenced on www.astm.org.

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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 ± 1.2 mm^2/s at 100°C in accordance with Test Methods D88 and Practice D2161. Viscosity Gravity Constant is 0.889 ± 0.002 in accordance with Test Method D2501. 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.

good carbon black dispersion is obtained.

6.2 *Test Method A—Internal Mixer for Initial and Final Mix*—See Table 2 and Table 3.

6.2.1 For general mixing procedure, refer to Practice D3182.

6.2.1.1 If required, cut samples from the batch to allow testing of compound viscosity and processability in accordance with Test Methods D1646 or D6204, and vulcanization characteristics in accordance with Test Methods D2084 or D5289.

6.2.1.2 If tensile stress strain tests are required, sheet off to a finished thickness of approximately 2.2 mm (0.087 in.) and condition the compound according to Practice D3182.

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

6.3.1 For general mixing procedure refer to Practice D3182.

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 After mixing according to Table 4, measure and record the batch mass. 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 D1646 or D6204, and vulcanization characteristics in accordance with Test Methods D2084 or D5289.

6.3.3.3 If tensile stress strain tests are required, sheet off to a finished thickness of approximately 2.2 mm (0.087 in.) and condition the compound according to Practice D3182.

6.4 *Test Method C—Mill Procedure:*

6.4.1 For general mixing procedure, refer to Practice D3182.

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

TABLE 2 Internal Mixer Method A—Initial Mix

	Duration, min	Accumulative, min
Adjust the internal mixer temperature to achieve the discharge conditions outlined below. Close the discharge gate, start the rotor, at 8.0 rads/s (77 r/min) and raise the ram.	0.0	0.0
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
Allow the batch to mix.	3.0	3.5
Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	0.5	4.0
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
Check the batch mass and record. If it differs from the theoretical value by more than 0.5 %, discard the batch.	0.0	0.0
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
Allow the stock to rest for 1 to 24 h.	0.0	0.0

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