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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 all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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 Stan-

dards Related to Rubber and Rubber Testing (Withdrawn 1987)³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,

³ The last approved version of this historical standard is referenced on www.astm.org.

¹ These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials 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.

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 mm²/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.

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.

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.

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 $40 \pm 5^\circ\text{C}$ ($104 \pm 9^\circ\text{F}$).	1.0	7.0
Allow the stock to rest for 1 to 24 h.	0.0	0.0

TABLE 3 Internal Mixer Method A—Final Mix

	Duration, min	Accumulative, min
Adjust the internal mixer to $40 \pm 5^{\circ}\text{C}$ ($104 \pm 9^{\circ}\text{F}$). 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.	0.0	0.0
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
Allow the batch to mix until a temperature of $110 \pm 5^{\circ}\text{C}$ ($230 \pm 9^{\circ}\text{F}$) or a total mixing time of 3 min is reached, whichever occurs first. Discharge the batch. Subtotal Time (min) max.	2.5	3.0
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
Set with the rolls of a standard laboratory mill maintained at $40 \pm 5^{\circ}\text{C}$ ($104 \pm 9^{\circ}\text{F}$) and at 0.8 mm (0.032 in.) opening. Pass the rolled stock endwise through the mill six times.	2.0	5.0
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

TABLE 4 Test Method B—Final Mix

	Duration, min	Accumulative, min
With the roll maintained at $40 \pm 5^{\circ}\text{C}$ ($104 \pm 9^{\circ}\text{F}$) and set at 1.5 mm (0.06 in.) opening, band, on the slow roll, the amount of masterbatch based on two times the formula.	0.0	0.0
Add slowly the required amount of sulfur and accelerator.	1.0	1.0
Make three $\frac{3}{4}$ cuts from each side.	2.0	3.0
Cut the batch from the mill, and set the mill-roll opening at 0.8 mm (0.032 in.) and pass the rolled stock endwise through the mill six times.	2.0	5.0
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

6.3.3.3 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.

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 After mixing according to [Table 5](#), measure and record the batch mass. If it differs from the theoretical value by more than 0.5 %, discard the batch.

6.4.2.2 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](#).

TABLE 5 Test Method C—Mixing Cycle

NOTE 1—Do not cut the batch while any free ingredient is evident in the bank or on the milling surface.

	Duration, min	Accumulative, min
With rolls temperature set at $35 \pm 5^{\circ}\text{C}$ and opening of 0.46 mm band the rubber on the front roll.	1.5	1.5
Add the preblended zinc oxide and stearic acid evenly across the rolls. Make two $\frac{3}{4}$ cuts from each side.	2.5	4.0
Add a small amount of dry carbon black evenly across the rolls, then using a spatula, wipe some of the oil-carbon black paste on to the mix. Repeat the sequence of dry black addition followed by the paste until all the paste has been added. Use a small piece of the mix to wipe out the paste container. Continue to add the dry black slowly until half the black is added. Increase the mill opening to 0.6 mm and continue to add the remaining black in small amounts. Clean the black out of the mill pan and add it to the mix.	10.0	14.0
Make three $\frac{3}{4}$ cuts from each side.	1.5	15.5
Add the TBBS and mix until no white spots are visible in the mix. Then slowly add the sulphur evenly across the rolls.	2.0	17.5
Make six successive $\frac{3}{4}$ cuts from each side.	2.0	19.5
Cut the batch from the mill. Set the mill opening at 0.8 mm and pass the rolled batch endwise through the the rolls six times.	2.0	21.5

6.4.2.3 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.

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

6.5.1 For general mixing procedure, refer to Practice D3182.

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.) thick

sheet. Cut the sheet into strips that are approximately 25 mm (1 in.) wide. Mix according to the procedure in Table 6.

6.5.3.1 After mixing according to Table 6, turn off the motor, raise the ram, remove the mixing chamber, and discharge the batch. Record the maximum batch temperature indicated, if desired.

6.5.3.2 Immediately pass the discharge from the mixer twice through a standard mill maintained at $40 \pm 5^{\circ}\text{C}$ ($104 \pm 9^{\circ}\text{F}$) with a roll separation of 0.5 mm (0.020 in.) once, then twice at a separation of 3 mm (0.12 in.) in order to dissipate heat. Pass the rolled batch endwise through the mill six times with an opening of 0.8 mm (0.31 in.) to enhance the dispersion.

TABLE 6 Test Method D—Miniature Internal Mixer Procedure

	Duration, min	Accumulative, min
Charge the mixing chamber with the rubber strips, lower the ram, and start the timer.	0.0	0.0
Masticate the rubber.	0.5	0.5
Raise the ram, add zinc oxide, sulfur, stearic acid, and TBBS that have previously been blended, taking care to avoid any loss.	1.0	1.5
Add portions of the carbon black and oil alternately, sweep the orifice, and lower the ram.	1.0	2.5
Allow the batch to mix, raising the ram momentarily to sweep down, if necessary.	6.5	9.0

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

6.5.3.4 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.

6.5.3.5 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.

7. Preparation and Testing of Vulcanizates

7.1 For stress-strain testing, prepare the test sheets and vulcanize them in accordance with Practice D3182.

7.1.1 The recommended standard vulcanization times for the mixes prepared by Test Methods A, B and C are 25, 35, and 50 min at 145°C (293°F). The recommended cure time for the miniature internal mixer compound is 35 min at 145°C (293°F).

7.1.2 Condition the cured sheets for 16 to 96 h at a temperature of 23 ± 2°C (73.4 ± 3.6°F).

NOTE 4—Quality control of rubber production may require testing within 1 to 6 h to provide close surveillance; however, slightly different results may be obtained.

7.1.3 Prepare the test specimens and obtain the modulus, tensile, and elongation parameters in accordance with Test Method D412.

7.2 An alternative to measuring vulcanization characteristics by means of tensile stress measurement on vulcanizates is the measurement of vulcanization characteristics in accordance with Test Method D2084 (Oscillating Disk Cure Meter Method) or Test Method D5289 (Rotorless Cure Meter Method). These methods will not produce equal results.

7.2.1 The recommended Test Method D2084 test conditions are 1.67 Hz (100 cpm) oscillation frequency, 1° oscillation amplitude, 160°C die temperature, 30-min test time, and no preheating. The recommended Test Method D5289 test conditions are 1.67 Hz (100 cpm) oscillation frequency, 0.5° oscillation amplitude, 160° C die temperature, 30-min test time, and no preheating. Test condition tolerances are specified by the test methods.

7.2.2 The recommended standard test parameters are M_L , M_H , t_{s1} , $t'50$, and $t'90$.

NOTE 5—It is recommended that M_H , if applicable, be taken as the torque value at 30 min.

8. Precision and Bias⁴

8.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to this practice for terminology and other statistical details.

8.2 The precision results in this precision and bias section give an estimate of the precision of the test method with the materials used in the particular interlaboratory program as described below. The precision parameters should not be used for acceptance/rejection testing of any group of materials

without documentation that they are applicable to those particular materials and the specific testing protocols that include these test methods.

8.3 A Type 2, Class III interlaboratory precision program was conducted using the Oscillating Disk Cure Meter. Both repeatability and reproducibility are short term. A period of a few days separates each of the two day test results. Four laboratories participated and five materials were used. Therefore, $p = 4$, $q = 5$, and $n = 2$. A test result is the value obtained from one determination.

8.4 The five rubbers used were homogeneous samples of different polybutadienes. Compounds were mixed in each laboratory banbury on each day using test method A. The test parameters used are described in 7.2.

8.5 The results of the precision calculations are given in Table 7 with the range of material mean values listed for each test property measured. The mean values were averaged while the standard deviations (s , S) were pooled.

8.6 The precision for these tests on a relative basis may be expressed as follows:

8.6.1 *Repeatability*—The repeatability (r) of these tests has been established as the appropriate value from the table. Two single measurements (determinations) that differ by more than the tabulated (r) (expressed as percentage of their mean value) must be considered suspect, that is, having arisen from different sample populations. Such a decision dictates that appropriate action be taken.

8.6.2 *Reproducibility*—The reproducibility (R) of these tests has been established as the appropriate value from the table. Two single measurements (determinations) that differ by more than the tabulated (R) (expressed as percentage of their mean value) must be considered suspect, that is, having arisen from different sample populations. Such a decision dictates that appropriate action be taken.

8.7 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for this test method since the value or level of the test property is exclusively defined by the test method. Bias, therefore, cannot be determined.

TABLE 7 Type 2 Precision—Vulcanization Parameters Using the Oscillating Disk Cure Meter^{A,B}

Property	Range of mean values	S_r	r	(r)	S_R	R	(R)
M_L	8.0–10.3	0.33	0.93	10.4	1.14	3.24	36.3
M_H	35.2–29.3	1.37	3.89	10.4	3.72	10.5	28.3
t_{s1}	4.4–5.9	0.11	0.31	5.9	0.81	2.3	44.6
$t'50$	9.6–10.6	0.35	1.00	9.8	0.91	2.57	25.3
$t'90$	13.7–15.5	0.35	0.98	6.8	1.44	4.08	28.2

^A Units: M_L and M_H = dN · m; t_{s1} , $t'50$, and $t'90$ = minutes.

^B This is short term precision with $p = 4$, $q = 5$, and $n = 2$.

S_r = Within laboratory standard deviation.

r = Repeatability in measured units.

(r) = Repeatability in percent.

S_R = Between laboratory standard deviation.

R = Reproducibility in measured units.

(R) = Reproducibility in percent.

Outliers have been rejected from the tabled data based on the Practice D4483 procedure.

⁴ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1067.

9. Keywords

9.1 BR; polybutadiene; polymerization; solution

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