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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Rubber—Evaluation of Solution BR (Polybutadiene Rubber)[1]

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 Viscosity[2]

D 412 Test Methods for Vulcanized Rubber and Thermoplastic Rubbers and Thermoplastic Elastomers— Tension[3]

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 Meter[3]

D 2161 Practice for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity[4]

D 2501 Test Method for Calculation of Viscosity-Gravity Constant (VGC) of Petroleum Oils[4]

D 3040 Practice for Preparing Precision Statements for Standards Related to Rubber and Rubber Testing[5]

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets[3]

D 3896 Practice for Rubber from Synthetic Sources—Sampling[3]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[3]

D 5289 Test Method for Rubber Property—Vulcanization Using Rotorless Cure Meters[3]

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

[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 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.

5. Sample Preparation

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

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[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.

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:

	Duration, min	Accumulative, 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:*

	Duration, min	Accumulative, 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 *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 6.2.2.

6.3.3 *Final Mix:*

	Duration, min	Accumulative, min
6.3.3.1 With the roll maintained at 40 ± 5°C (104 ± 9°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
6.3.3.2 Add slowly the required amount of sulfur and accelerator.	1.0	1.0
6.3.3.3 Make three ¾cuts from each side.	2.0	3.0
6.3.3.4 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
6.3.3.5 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

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

6.3.3.7 Cut a specimen to allow testing of compound viscosity in accordance with Test Method D 1646 or vulcanizing characteristics with the oscillating disk cure meter in accordance with Test Method D 2084 as described in 8.2, or both, if these are desired.

6.3.3.8 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.

6.3.3.9 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.

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 ± 5°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*:

	Duration, min	Cumulative Time, min
6.4.2.1 With rolls temperature set at 35 ± 5°C and opening of 0.46 mm band the rubber on the front roll.	1.5	1.5
6.4.2.2 Add the preblended zinc oxide and stearic acid evenly across the rolls. Make two ¾cuts from each side.	2.5	4.0

NOTE 5—Do not cut the batch while any free ingredient is evident in the bank or on the milling surface.

	Duration, min	Cumulative Time, min
6.4.2.3 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
6.4.2.4 Make three ¾ cuts from each side.	1.5	15.5
6.4.2.5 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
6.4.2.6 Make six successive ¾ cuts from each side.	2.0	19.5
6.4.2.7 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

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

6.4.2.9 Cut a specimen 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.

6.4.2.10 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.

6.4.2.11 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.

6.5 *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 ± 3°C (140 ± 5°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 ± 5°C (109 ± 9°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.

		Duration, min	Accumulative, min
6.5.3.1	Charge the mixing chamber with the rubber strips, lower the ram, and start the timer.	0.0	0.0
6.5.3.2	Masticate the rubber.	0.5	0.5
6.5.3.3	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
6.5.3.4	Add portions of the carbon black and oil alternately, sweep the orifice, and lower the ram.	1	2.5
6.5.3.5	Allow the batch to mix, raising the ram momentarily to sweep down, if necessary.	6.5	9.0

NOTE 6—If only the measurement of cure meter parameters is required, a 5-min mixing cycle may be used reducing the step 6.5.3.5 to 2.5 min.

6.5.3.6 Turn off the motor, raise the ram, remove the mixing chamber, and discharge the batch. Record the maximum batch temperature indicated, if desired.

6.5.3.7 Pass the batch through a mill at 40 ± 5°C (104± 9°F) and 0.5 mm (0.020 in.) opening once, then twice at 3 mm (0.125 in.) opening.

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

6.5.3.9 Cut a specimen for testing vulcanization characteristics in accordance with Test Method D 2084, as described in 8.2, if required. Condition the specimens for 1 to 24 h at 23 ± 3°C (73.4 ± 5.4°F) before testing.

6.5.3.10 If either compound-viscosity or stress-strain testing is required, pass the rolled stock endwise through the mill 6 times with the mill temperature at 40 ± 5°C (104 ± 9°F) and the roll separation of 0.8 mm (0.032 in.).

6.5.3.11 Cut a specimen to allow testing of compound viscosity in accordance with Test Method D 1646, if required.

6.5.3.12 If tensile stress is required, sheet off the compound from the mill at a setting to give a finished gage of approximately 2.2 mm (0.085 in.) by passing the folded stock between the mill rolls set at 40 ± 5°C (104 ± 9°F) four times always in the same direction to obtain the effects of mill direction. Cool on a flat, dry metal surface.

6.5.3.13 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.

7. Preparation and Testing of Vulcanizates

7.1 For stress-strain testing, prepare the test sheets and vulcanize them in accordance with Practice D 3182.

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 7—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 D 412.

7.2 An alternative to measuring vulcanization characteristics by means of tensile stress measurements on vulcanizates is the measurement of vulcanization characteristics with an Oscillating Disk cure meter in accordance with Test Method D 2084, or a Rotorless Cure Meter in accordance with Test Method D 5289. These methods will not produce equal results.

7.2.1 The recommended standard Oscillating Disk test conditions are 1.7 Hz oscillation frequency; ±1° amplitude in oscillation, 160°C die temperature, 30 min test time, and no preheating. The recommended test conditions for the Rotorless Cure Meter are 1.7 Hz oscillation frequency, ±0.5° of arc for torsional shear cure meters and ±0.05 mm for linear shear cure meters, 160° C die temperature, 30 min test time, and no preheating. Tolerances for the listed conditions are included in the specified test methods.

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

NOTE 8—It is recommended that M_H, if applicable, be taken as the torque value at 30 min.

8. Precision and Bias [6]

8.1 This precision and bias section has been prepared in accordance with Practice D 4483. 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.

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[6] Supporting data are available from ASTM Headquarters. Request RR: D11-1067.

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 1 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.

TABLE 1 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	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 D 4483 procedure.

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.

9. Keywords

9.1 BR; polybutadiene; polymerization; solution