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Standard Test Methods for Rubber—Evaluation of NBR (Acrylonitrile-Butadiene Rubber)[1]

This standard is issued under the fixed designation D3187; 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 formulas, mixing procedures and test methods for the evaluation of various types of acrylonitrile-butadiene rubber (NBR).

1.2 The values stated in SI units are to be regarded as the standard. The values given 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 and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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

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

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 test methods may be used to obtain values for customer control acceptance of rubber.

4. Standard Test Formula

4.1 *Standard Formula*—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 compound may be prepared either on a mill, in a miniature internal mixer, or a lab internal mixer, although slightly different results may be obtained.

6.2 *Mill Procedure:*

6.2.1 For general mixing procedure refer to Practice D3182.

6.2.1.1 Mix with the mill roll temperature maintained at 50 ± 5°C (122 ± 9°F). The indicated mill openings are desired and should be maintained in so far as possible to provide uniformity in the breakdown of the rubber due to milling. Necessary adjustments may be made to maintain a good working bank.

6.2.2 *Mixing Cycle*—See Table 2.

6.2.2.1 After mixing according to Table 2, measure and record the batch mass. If it differs from the theoretical value by more than 0.5 %, discard the batch.

6.2.2.2 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.

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[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.

TABLE 1 Standard Formula

Material	NIST SRM/IRM No.	Quantity, Parts by Mass
NBR	...	100.00
Zinc oxide	[A]	3.00
Sulfur, coated[B]	...	1.50
Stearic acid	[A]	1.00
Oil furnace black	SRB-B4	40.00
TBBS[C]	[A]	0.70
Total		146.20
Batch factor for mill mix[D]		4.0
Batch factor for MIM mix (Cam Head[E]		0.50
Batch factor for MIM mix (Banbury Head)[E]		0.43

[A] Use current IRM/SRM.

[B] The use of 2 % $MgCO_3$ coated sulfur is recommended. Standard 2 % $MgCO_3$ coated sulfur (Lot No. M-266573-P) is available from the C. P. Hall Co., 4460 Hudson Drive, Stow, OH 44224.

[C] *N-tert*-butyl-2-benzothiozolesulfenamide.

[D] For mill and lab BB mixes, weigh the rubber and carbon black to the nearest 1.0 g, the sulfur and accelerators to the nearest 0.02 g, and all other compounding materials to the nearest 0.1 g.

[E] For MIM batches, weigh the rubber and material, blend to the nearest 0.1 g. If individual compounding materials are used, blend to the nearest 0.001 g. For the MIM procedure, it is recommended that a blend of compounding materials, excluding black, 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.

6.2.2.3 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.

6.3 *Miniature Internal Mixer (MIM) Procedure:*

6.3.1 For general mixing procedure refer to Practice D3182.

6.3.1.1 Mix with the head temperature of the miniature internal mixer maintained at 60 ± 3°C (140 ± 5°F) and the rotor speed at 6.3 to 6.6 rad/s (60 to 63 r/min).

6.3.2 Prepare the rubber by passing it through a mill one time with the temperature set at 50 ± 5°C (122 ± 9°F) and an opening that will give an approximately 5 mm (0.2 in.) thick sheet. Cut the sheet into strips that are approximately 25 mm (1 in.) wide.

6.3.3 *Mixing Cycle*—See Table 3.

6.3.3.1 After mixing according to Table 3, turn off the motor, raise the ram, remove the mixing chamber and discharge the batch. Record the maximum batch temperature indicated, if desired.

6.3.3.2 Immediately pass the discharge from the mixer twice through a standard mill maintained at 50 ± 5°C (122 ± 9°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.

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

6.3.3.4 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.

6.3.3.5 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.

6.4 *Internal Mixer Procedure:*

6.4.1 For general mixing procedure refer to Practice D3182.

6.4.2 *Mixing Cycle-Initial Mix*—See Table 4.

6.4.2.1 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.

6.4.2.2 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).

6.4.2.3 Allow the batch to rest for 1 to 24 h.

6.4.3 *Final Mix*—See Table 5.

6.4.3.1 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.

6.4.3.2 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.

6.4.3.3 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.

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 cure times for the mill compound are 20, 40, and 60 min at 150°C (302°F). The recommended cure time for the miniature internal mixer compound is 40 min at 150°C (302°F).

Note 1—Vulcanization times of 25, 50, and 75 min at 145°C (293°F) for the mill compound and 50 min at 145°C (293°F) for the miniature internal mixer compound may also be used, but will not necessarily give the same results as the recommended standard vulcanization times.

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 2—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 test specimens and obtain modulus, tensile and elongation parameters in accordance with Test Methods 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 Methods D2084 or D5289. These methods will not produce equal results.

7.2.1 The recommended 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 D5289 test conditions are 1.67 Hz 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_{sl}, t'50, and t'90.

Note 3—It is recommended that M_H, if applicable, be taken as the torque value at 30 min.

TABLE 2 Mill Procedure: Mixing Cycle

NOTE 1—Do not cut the batch while free carbon black is evident in the bank or on the milling surface. Be certain to return to the batch any ingredients that drop through the mill.

	Duration, min	Accumulative, min
Set the mill opening at 1.4 mm (0.055 in.) and band the rubber on the slow roll.	2	2
Add stearic acid and zinc oxide together, followed by sulfur and accelerator together. Make the addition slowly and evenly across the mill, without cutting.	3	5
Make three ¾ cuts from each side.	2	7
Add half of the carbon black evenly across the mill at a uniform rate.	5	12
When this portion of the carbon black has been completely incorporated, open the rolls to 1.65 mm (0.065 in.) and make three ¾ cuts from each side.	2	14
Add the remaining carbon black evenly across the rolls at a uniform rate.	5	19
When all carbon black has been incorporated, make three ¾ cuts from each side	2	21
Set the rolls at 0.8 mm (0.032 in.), pass the rolled stock endwise between the rolls six times.	3	24
Open the mill to give a minimum batch thickness of 6 mm (0.25 in.) and pass the stock through the rolls four times, folding it back on itself each time.	1	25

TABLE 3 Miniature Internal Mixer—Mixing Cycle

	Duration, min	Accumulative, min
Charge the mixing chamber with the rubber strips, lower the ram, and start the timer.	0	0
Masticate the rubber.	1.0	1.0
Raise the ram and add zinc oxide, sulfur, stearic acid, and TBBS that have previously been blended, taking care to avoid any loss. Then add carbon black. Sweep the orifice and lower the ram.	1.0	2.0
Allow the batch to mix, raising the ram momentarily to sweep down materials if necessary.	7.0	9.0

TABLE 4 Internal Mixer—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 8.1 rad/s (77 rpm) and raise the ram.	0	0
Charge ½ the rubber, all of the zinc oxide, carbon black, stearic acid, and then the other one half of the rubber. Lower the ram.	0.5 3.0	0.5 3.0
Allow the batch to mix.	0.5	4.0
Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	2.0	6.0
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.	0	0

TABLE 5 Internal Mixer—Final Mix

	Duration, min	Accumulative, min
Adjust the internal mixer temperature to 40 ± 5°C (104 ± 9°F), turn off steam and turn on full cooling water to the rotors, start the rotors at 8.1 rad/s (77 rpm), and raise the ram.	0	0
Charge ½ the batch, with all the sulfur and accelerator rolled into this portion of the batch before feeding to the mixer. Add the remaining portion of the batch. Lower the ram.	0.5	0.5
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.	2.5	3.0
With the rolls of a standard laboratory mill maintained at 40 ± 5°C (104 ± 9°F) and set at 0.8 mm (0.032 in.) opening, pass the rolled batch endwise through the rolls six times.	2.0	5.0
Open the rolls to give a minimum thickness of 6 mm (0.25 in.) and pass the compound through four times, folding it back on itself each time.	1.0	6.0

8. Precision and Bias

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

8.2 The precision results in this precision and bias section give an estimate of the precision of these test methods with the materials (rubbers) 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 (interlaboratory) precision was evaluated. Both repeatability and reproducibility are short term; a period of a few days separates replicate test results. A test result for Test Methods D412 is the median value, as specified by these test methods, obtained on three determination(s) or measurement(s). For Test Methods D1646 and D2084, a test result is obtained from one determination.

8.4 Three different materials or rubbers were used in the interlaboratory program, these were tested in seven laboratories on two different days.

8.5 The results of the precision calculations for repeatability and reproducibility are given in Table 6.

8.6 The precision of these test methods may be expressed in the format of the following statements that use an "appropriate value" of *r*, *R*, (*r*), or (*R*), to be used in decisions about test results. The appropriate value is that value of *r* or *R* associated with a mean level in Table 6 closest to the mean level under consideration at any given time, for any given material in routine testing operations.

8.7 *Repeatability*—The repeatability, *r*, of these test methods has been established as the appropriate value tabulated in

TABLE 6 Type 2 Precision for Various Test Parameters

NOTE 1—
Sr = Repeatability standard deviation, in measurement units,
r = Repeatability, in measurement units,
(*r*) = Repeatability, (relative) percent,
SR = Reproducibility standard deviation, in measurement units,
R = Reproducibility, in measurement units, and
(*R*) = Reproducibility, (relative) percent.

Property	Units	Range of Values[A]	Within Laboratory			Between Laboratories		
			Sr	*r*	(*r*)	*SR*	*R*	(*R*)
For Test Method D2084:								
M_L	dN·m	5.4 to 12.4	0.28	0.79	8.9	0.53	1.50	16.9
M_H	dN·m	36.0 to 46.7	0.85	2.41	5.8	2.14	6.05	14.6
t_{s1}	min	2.8 to 3.9	0.10	0.28	8.2	0.49	1.39	40.9
t'_{90}	min	11.4 to 15.3	0.56	1.58	11.8	1.49	4.22	31.5
For Test Methods D412:								
300 % modulus, MPa		11.1 to 16.3	0.63	1.78	13.0	1.11	3.14	22.9
Tensile strength, MPa		26.7 to 31.4	0.77	2.18	7.5	1.28	3.62	12.4
Elongation, %		49.3 to 57.7	13.5	38.2	7.1	31.8	90.0	16.8
For Test Methods D1646:								
M_L (1 + 4)		54.4 to 104.3	1.30	3.68	4.63	7.8	22.1	27.8

[A] Midpoint of range used for (*r*), (*R*) calculations; For Test Method D2084—160°C, 1.7 Hz, 1° amplitude arc.

Table 6. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated *r* (for any given level) must be considered as derived from different or nonidentical sample populations.

8.8 *Reproducibility*—The reproducibility, *R*, of these test methods has been established as the appropriate value tabulated in Table 6. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated *R* (for any given level) must be considered to have come from different or nonidentical sample populations.

8.9 Repeatability and reproducibility expressed as a percentage of the mean level, (*r*) and (*R*), have equivalent application statements as above for *r* and *R*. For the (*r*) and (*R*) statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

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

9. Keywords

9.1 acrylonitrile-butadiene; NBR

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