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

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^{ε1} ~~Note~~ Section 4 was corrected editorially in March 1996.

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1. Scope

1.1 These test methods specify 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:*

D 412 Test Methods for Vulcanized Rubber and Thermoplastic Rubbers and Thermoplastic Elastomers— Tension²

D 1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)²

D 2084 Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter²

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets²

D 3896 Practice for Rubber from Synthetic Sources—Sampling²

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries²

D 5289 Test Method for Rubber Property – Vulcanization using Rotorless Cure Meters²

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:*

Material	NIST SRM/IRM No.	Quantity, Parts by Mass
NBR	...	100.00
— Zinc oxide	— IRM-91	— 3.00
— Zinc oxide	— A	— 3.00
— Sulfur, coated ^A	— ...	— 1.50
— Sulfur, coated ^B	— ...	— 1.50
— Stearic acid	— 372	— 1.00
— Stearic acid	— A	— 1.00
— Oil furnace black	— SRB-B4	— 40.00
— TBBS ^B	— 384	— 0.70
— TBBS ^C	— A	— 0.70

² Annual Book of ASTM Standards, Vol 09.01.

Material	NIST SRM/IRM No.	Quantity, Parts by Mass
Total		146.20
Batch factor for mill mix ^C		—4.0
Batch factor for mill mix ^D		4.0
Batch factor for MIM mix (Cam Head) ^D		—0.50
Batch factor for MIM mix (Cam Head) ^E		0.50
Batch factor for MIM mix (Banbury Head) ^D		—0.43
Batch factor for MIM mix (Banbury Head) ^E		0.43

^AUse current IRM/SRM.

^BThe use of 2 % MgCO₃ coated sulfur is recommended. Standard 2 % MgCO₃ 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.*

^DFor mill 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.

^DFor 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.

^EFor 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.

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 compound may be prepared either on a ~~mill or 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 D 3182.

6.2.1.1 Mix with the mill roll temperature maintained at $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$). 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:

	Duration, min	Accumula- tive, 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 $\frac{3}{4}$ 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 $\frac{3}{4}$ cuts from each side.	2	14

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	Accumula- tive, min
Add the remaining carbon black evenly across the rolls at a uniform rate.	5	19
When all carbon black has been incorporated, make three $\frac{3}{4}$ 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

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

6.2.2.2 Cut a specimen to allow testing of compound viscosity in accordance with Test Method D 1646 or vulcanization

characteristics in accordance with Test Method D 2084 as described in 8.2, or both, if these are desired.

6.2.2.3 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.

6.2.2.4 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.

6.3 Miniature Internal Mixer (MIM) Procedure:

6.3.1 For general mixing procedure refer to Practice D 3182.

6.3.1.1 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 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 $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$) 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:

	Duration, min	Accumula- tive, 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

NOTE 2—If only the measurement of cure meter parameters is required, a 5-min mixing cycle may be used, reducing step 6.3.3.4 to 3 min.

6.3.3.1 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 Pass the batch through a mill set at $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$) and 0.5-mm (0.020-in.) opening once, then twice at 3-mm (0.125-in.) opening.

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

6.3.3.4 From the compound, cut a specimen for testing vulcanization characteristics in accordance with Test Method D 2084, as described in 8.2, if required. Condition the specimen for 1 to 24 h at $23 \pm 3^\circ\text{C}$ ($73.4 \pm 5.4^\circ\text{F}$) before testing.

6.3.3.5 If either compound-viscosity or stress-strain testing is required, pass the rolled stock endwise through the mill six times with the mill rolls set at $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$) and 0.8 mm (0.032 in.).

6.3.3.6 Cut a specimen to allow testing for compound viscosity in accordance with Test Methods D 1646, if required.

6.3.3.7 If tensile stress is required, sheet off the stock 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 $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$) four times, always in the same direction to obtain the effects of mill direction. Cool on a flat, dry, metal surface.

6.3.3.8 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.

6.4 Internal Mixer Procedure:

6.4.1 For general mixing procedure refer to Method D 3182.

6.4.2 Mixing Cycle-Initial Mix:

	Duration, Min	Accumula- tive, Min
6.4.2.1 <u>Adjust the internal mixer temperature to achieve the discharge conditions outlined in 6.4.2.5. Close the discharge gate, start the rotor at 8.1 rad/s (77 rpm) and raise the ram.</u>	<u>0</u>	<u>0</u>
6.4.2.2 <u>Charge one half the rubber, all of the zinc oxide, carbon black, stearic acid, and then the other one half of the rubber. Lower the ram.</u>	<u>.5</u> <u>3.0</u>	<u>.5</u> <u>3.5</u>

6.4.2.3

<u>6.4.2.4</u>	<u>Allow the batch to mix.</u>	<u>.5</u>	<u>4.0</u>
<u>6.4.2.5</u>	<u>Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.</u>	<u>2.0</u>	<u>6.0</u>
<u>6.4.2.5 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.</u>			
<u>6.4.2.6 Determine and record the batch mass; if the mass differs by more than 0.5 % of the theoretical mass, discard the batch.</u>			
<u>6.4.2.7 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).</u>			
<u>6.4.2.8 Allow the batch to rest for 1 to 24 h.</u>			
<u>6.4.3 Final Mix:</u>			
<u>6.4.3.1 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.</u>			
		<u>Duration,</u> <u>Min</u>	<u>Accumula-</u> <u>tive, Min</u>
<u>6.4.3.2</u>	<u>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</u>	<u>.5</u>	<u>.5</u>
<u>6.4.3.3</u>	<u>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.</u>	<u>2.5</u>	<u>3.0</u>
<u>6.4.3.4</u>	<u>Determine and record the batch mass; if the mass differs by more than 0.5 % of the theoretical mass, discard it.</u>		
<u>6.4.3.5</u>	<u>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.</u>	<u>2.0</u>	<u>5.0</u>
<u>6.4.3.6</u>	<u>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.</u>	<u>1.0</u>	<u>6.0</u>
<u>6.4.3.7 Cut enough sample for testing of compound viscosity in accordance with Test Method D 1646 or vulcanizing characteristics in accordance with Test Method D 2084, as described in 7.2, or both, if these are desired. Condition the specimen for 1 to 24 h at 23 ± 3°C (73.4 ± 5.4°F) before testing.</u>			
<u>6.4.3.8 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 rolls set at 50 ± 5°C (122 ± 9°F) four times always in the same direction to obtain the effects of mill direction. Cool on a flat, dry metal surface.</u>			
<u>6.4.3.9 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.</u>			

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 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 3—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 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 test specimens and obtain modulus, tensile and elongation parameters in accordance with Test Method D 412.

7.2 An alternative to measuring vulcanization characteristics by means of ~~modulus~~ tensile stress measurement on vulcanizates is the measurement of vulcanization ~~parameters~~ 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 ~~(100 cpm)~~ oscillation frequency, $1 \pm 0.3^\circ$ frequency; $\pm 1^\circ$ amplitude of oscillation, $160 \pm 0.3^\circ\text{C}$ ($320 \pm 0.5^\circ\text{F}$) 160°C die temperature, 30-min test time, and no preheating. The recommended test conditions for the Rotorless Cure Meter are: 1.76 Hz oscillation frequency, $\pm 0.5^\circ$ 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_H , M_{HF} , M_H , ~~or~~ M_H , R , *as appropriate*, M_L , t_{s1} , t'_{50} , 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 Bias

8.1 This precision and bias section has been prepared in accordance with Practice D 4483. Refer to Practice D 4483 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 Method D 412 is the median value, as specified by these test methods, obtained on three determination(s) or measurement(s). For Test Methods D 1646 and D 2084, 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 1.

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 1 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 1. 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 1. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more

TABLE 1 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 D 2084:								
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 Method D 412:								
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 Method D 1646:								
$M_L(1 + 4)$		54.4 to 104.3	1.30	3.68	4.63	7.8	22.1	27.8

^AMidpoint of range used for (r) , (R) calculations; For Test Method D 2084—160°C, 1.7 Hz, 1° amplitude arc.

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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