Designation: D 3848 – 03

Standard Test Methods for Rubber—Evaluation of NBR (Acrylonitrile-Butadiene Copolymers) Mixed With Carbon Black[1]

This standard is issued under the fixed designation D 3848; 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 specify the standard materials, test formula, mixing procedures, and test methods for the evaluation of acrylonitrile-butadiene rubber (NBR) mixed with carbon black.

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]

D 412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

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

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

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

D 3896 Practice for Rubber from Synthetic Sources—Sampling

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

D 5289 Test Method for Rubber Property–Vulcanization Using Rotorless Cure Meters

D 6204 Test Method for Rubber–Measurement of Unvulcanized Rheological Properties Using Rotorless Shear Rheometers

3. Significance and Use

3.1 These test methods are mainly intended 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 samples in a standard test formula.

3.2 These test methods may also be used to obtain values for customer acceptance of rubber.

4. Standard Test Formula

4.1 *Standard Formula*:

Material	IRM/SRM SRM No.	Quantity, Parts By Mass
Masterbatch	...	100.00 + *X*[A]
Zinc oxide	[B]	3.00
Sulfur, coated[C]	...	1.50
Stearic acid	[B]	1.00
TBBS[D]	[B]	0.70 + *X*
Total mass		106.20 + *X*
Batch factors		
Mill[E]		
Miniature internal mixer[F]		

[A] *X* = parts carbon black per 100 parts base polymer.

[B] Use current IRM/SRM.

[C] The use of 2 % $MgCO_3$ coated sulfur is recommended. Standard 2 % $MgCO_3$ coated sulfur Lot No. M266573-P is available from C. P. Hall Co., 4460 Hudson Drive, Stow, OH 44224.

[D] *N-tert*-butyl-2-benzothiazolesulfenamide.

[E] For mill mixing, a batch factor should be selected to the nearest 0.5 to give as large as total mass as possible that will not exceed 525.0 g. Calculate all parts to the nearest 0.01 part. Weigh the masterbatch to the nearest 1 g, the sulfur and the accelerator to the nearest 0.02 g, and all the other compounding materials to the nearest 0.1 g.

[F] For MIM mix, select a batch factor to give a batch that will fill the mixing chamber volume to 75 % capacity. Calculate all parts to the nearest 0.1 g, the compounding material blend to the nearest 0.01 g, and the individual compounding materials, if used, to the nearest 0.001 g.

For the MIM procedure, it is recommended that a blend of compounding materials be prepared to improve accuracy in the weighing of the materials. The compound material blend is prepared by blending a proportional mass of each

[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.

material in a biconical or a vee blender. A mortar and pestle may be used for blending small quantities.

5. Sample Preparation

5.1 Obtain and prepare the test samples in accordance with Practice D 3896.

6. Mixing Procedures

6.1 Three mixing procedures are provided as follows:

6.1.1 *Method A—Mill Mix* (6.2) and

6.1.2 *Method B—Miniature Internal Mixer Mix* (6.3).

6.1.3 *Method C—Internal Mixer*

NOTE 1—It is not implied that comparable results will be obtained by these test methods.

6.2 *Method A—Mill Mix Procedure*:

6.2.1 For general mixing procedures, refer to Practice D 3182.

6.2.2 *Mixing Cycle—Initial Mix*:

	Duration, min	Accumulative, min
6.2.2.1 With the mill roll temperature set at 50 ± 5°C (122 ± 9°F) and the mill opening set at 1.40 mm (0.055 in.), band the masterbatch on the slow roll without cutting.	2	2
6.2.2.2 Add sulfur slowly and evenly across the mill at a uniform rate.	2	4
6.2.2.3 Add stearic acid. Make one ¾ cut from each side after the stearic acid has been incorporated.	2	6
6.2.2.4 Add the zinc oxide and the accelerator.	3	9
6.2.2.5 Make three ¾ cuts from each side and cut the batch from the mill.	2	11
6.2.2.6 Set the rolls at 0.8 mm (0.032 in.). Pass the rolled batch endwise through the mill six times.	2	13
6.2.2.7 Open the mill to give a minimum batch thickness of 6 mm (0.25 in.) and pass the stock through the mill four times, folding it back on itself each time.	1	14
6.2.2.8 Check the batch mass and record. If it differs from the theoretical value by more than 0.5 %, discard the batch.		
6.2.2.9 From this batch cut a sample for testing of compound viscosity in accordance with Test Method D 1646 or rheological properties in accordance with Test Method D 6204, vulcanizing characteristics in accordance with Test Method D 2084, or Test Method D 5289, or both, if these are desired. Condition the sample for 1 to 24 h at 23 ± 2°C (73.4 ± 3.6°F) before testing.		
6.2.2.10 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 effect of milling. Cool on a flat, dry metal surface.		
6.2.2.11 For routine laboratory testing, condition the sheeted compound for 1 to 24 h at 23 ± 2°C (73.4 ± 3.6°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 *Method B—Miniature Internal Mixer (MIM) Procedure*:

6.3.1 For general mixing procedures, refer to Practice D 3182. Mix with the MIM mixing chamber maintained at 60 ± 3°C (140 ± 5°F) and with an unloaded rotor speed of 6.3 to 6.6 rad/s (60 to 63 rpm).

6.3.2 Prepare the masterbatch by passing it through a mill one time with the temperature set at 50 ± 5°C (122 ± 9°F) and an opening of 0.5 mm (0.02 in.). Cut the sheet into strips that are approximately 25 mm (1 in.) wide, if desired.

6.3.3 *Mixing Cycle:*:

6.3.3.1 Feed the rubber strips into the mixing chamber and, when all are in, start the timer. Break down the rubber.	0.5	0.5
6.3.3.2 Add all the zinc oxide, sulfur, stearic acid, and TBBS which have previously been blended together, taking care to avoid any loss. Stop the mixer briefly and sweep loose pigments into the chamber with a brush.	0.5	1.0
6.3.3.3 Allow the compound to mix.		
6.3.3.4 Turn off the motor, raise the ram, remove the mixing chamber, and discharge the batch. Record the maximum batch temperature, if desired.	8.0	9.0
6.3.3.5		

Pass the batch between the rolls of a mill maintained at 50 ± 5°C (122 ± 9°F) and 0.5 mm (0.020 in.) opening once, then twice at 3.0 mm (0.122 in.) opening.

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 sample for testing of compound viscosity in accordance with Test Method D 1646 or rheological properties in accordance with Test Method D 6204, or vulcanizing characteristics in accordance with Test Method D 2084, or Test Method D 5289, if these are desired. Condition the sample 1 to 24 h at 23 ± 2°C (73.4 ± 3.6°F) before testing.

6.3.3.8

If stress strain testing is required, pass the rolled stock end-wise through the mill six times with the mill temperature at 50 ± 5°C (122± 9°F) and a roll separation of 0.8 mm (0.032 in.).

6.3.3.9

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 50 ± 5°C (122 ± 9°F) four times always in the same direction to obtain the effect of milling. Cool on a flat, dry metal surface.

6.3.3.10

For routine laboratory testing, condition the sheeted compound for 1 to 24 h at 23 ± 2°C (73.4 ± 3.6°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.

6.4 *Internal Mixer Procedure*:

6.4.1 For general mixing procedure refer to Practice D 3182.

6.4.2 *Mixing Cycle-Initial Mix*:

	Duration, min	Accumulative, min
6.4.2.1 Adjust the internal mixer temperature to achieve the discharge conditions outlined in 6.2.2. Close the discharge gate, start the rotor at 8.1 rad/s (77 rpm) and raise the ram.	0	0
6.4.2.2 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.	.5 3.0	.5 3.5
6.4.2.3 Allow the batch to mix.	.5	4.0
6.4.2.4 Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	2.0	6.0
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.	2.0	6.0
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.		
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).		
6.4.2.8 Allow the batch to rest for 1 to 24 h.		

6.4.3 Final Mix:

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, at 8.1 rad/s (77 rpm), and raise the ram.

	Duration, min	Accumulative, min
6.4.3.2 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.	5	5
6.4.3.3 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.	2.5	3.0
6.4.3.4 Determine the record the batch mass; if the mass differs by more than 0.5 % of the theoretical mass, discard it.		
6.4.3.5 With the rolls of a standard laboratory mill maintained at 40 ± 5°C (104 ± 9°F) and set at 0.8 mm (0.0032 in.) opening, pass the rolled batch endwise through the rolls six times.	2.0	5.0
6.4.3.6 Open the rolls to give a minimum thickness of 6 mm (0.25 in.) and pass the compound through four times, folding it back itself each time.	1.0	6.0
6.4.3.7 Cut enough samples for testing of compound viscosity in accordance with Test Method D 1646, or rheological properties in accordance with Test Method D 6204, or vulcanizing characteristics in accordance with Test Method D 2084, or Test Method D 5289, if these are desired. Condition the sample for 1 to 24 h at 23 ± 3°C (73.4 ± 5.4°F) before testing.		

6.4.3.8

If tensile stress is required, sheet off the compound from the mill at a setting to give a finished 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.

6.4.3.9

Four 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. Test for Cure Characteristics Using the Cure Meter

7.1 Measure 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.

7.2 The recommended standard Oscillating Disk test conditions are: 1.7 Hz oscillation frequency; ±1° amplitude of 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 ±0.05mm 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.

7.3 The recommended standard test parameters are: M_L, M_H, t_{sl}, t′50 and t′90.

8. Preparation and Testing of Vulcanizates

8.1 An alternative to measuring vulcanization parameters is the measurement of stress-strain properties of vulcanizates in accordance with Test Methods D 412. Prepare test sheets and vulcanize them in accordance with Practice D 3182.

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

NOTE 2—Vulcanization times of 25, 50, and 75 min at 145°C (293°F) for the mill-mixed compounds and 50 min at 145°C for the MIM-mixed compounds may also be used, but will not necessarily give the same results as the recommended standard vulcanization conditions.

8.1.2 Condition all vulcanizates for 16 to 96 h at a temperature of 23 ± 2°C (73.4 ± 3.6°F) prior to making stress-strain tests.

NOTE 3—Quality control of rubber production may require testing within 1 to 6 h to provide close surveillance of plant operations; however, slightly different results may be obtained.

8.1.3 Prepare test specimens and obtain tensile stress, tensile strength, and elongation in accordance with Test Methods D 412.

9. Precision and Bias [3]

9.1 This precision and bias section has been prepared in accordance with Practice D 4483. Please refer to this practice for terminology and other statistical calculation details.

9.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers, etc.) used in the particular interlaboratory program as described below. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that the parameters are applicable to the particular group of materials and the specific testing protocols of the test method.

9.3 A Type 2 interlaboratory test program was evaluated using the mill mixing technique. Both repeatability and reproducibility are short term; a period of a few days separates replicate test results. For testing via Test Method D 2084, a test result is the value, as specified by this method, obtained from one determination (measurement). Four laboratories participated in the program testing four different types of NBR, designated as DN127, DN120, 9020 and 9040. The program consisted of two replicates or $n = 2$; that is, one test result on each batch (compounded and mixed) and tested on each of the two test days.

9.4 The results of the precision evaluation are given in Table 1 for the four Test Method D 2084 measurement parameters, *ML*, *Ts*′1, *T*′90 and *MH*.

9.5 The precision of the mill mix test method (using Test Method D 2084) may be expressed in the format of the following statements that use an appropriate value of *r, R, (r)*, and (*R*) to be used in the decisions about the 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 test result for a material in routine testing operations. The general statements for repeatability and reproducibility apply to all four measurement parameters of Test Method D 2084.

9.5.1 *Repeatability*—The repeatability, *r*, of this test method 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 non-identical sample populations.

9.5.2 *Reproducibility*—The reproducibility, *R*, of this test method 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 than the tabulated *R* (for any given level) must be considered to have come from different or non-identical sample populations.

9.5.3 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 (in absolute units).

[3] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: D11–1064.

TABLE 1 Type 2 Precision for Curemeter Testing (Test Method D 2084)[A]

		Part 1: ML, dN·m[B]					
		Within Laboratory			Between Laboratory		
Material	Mean value	*Sr*	*r*	*(r)*	*SR*	*R*	(*R*)
9040	5.0	0.18	0.50	10.1	1.39	3.93	79.4
DN127	5.7	0.36	1.02	17.9	1.30	3.67	64.4
DN120	5.8	0.08	0.22	3.9	1.31	3.71	64.1
9020	9.4	0.09	0.25	2.6	2.66	7.52	80.4
		Part 2: T's1, minutes					
		Within Laboratory			Between Laboratory		
Material	Mean value	*Sr*	*r*	*(r)*	*SR*	*R*	(*R*)
9020	2.8	0.21	0.59	21.0	0.24	0.68	24.2
DN127	3.2	0.30	0.86	26.9	0.30	0.86	26.9
DN120	3.4	0.38	1.07	31.4	0.44	1.25	36.9
9040	3.5	0.09	0.25	7.4	0.46	1.31	38.1
		Part 3: T'90, minutes[C]					
		Within Laboratory			Between Laboratory		
Material	Mean value	*Sr*	*r*	*(r)*	*SR*	*R*	(*R*)
9020	13.4	0.76	2.15	16.0	0.96	2.71	20.2
DN127	14.4	0.18	0.51	3.5	3.40	9.63	66.7
DN120	14.5	0.19	0.53	3.6	3.56	10.1	69.7
9040	17.4	0.19	0.53	3.0	4.14	11.7	67.2
		Part 4: MH, dN·m[D]					
		Within Laboratory			Between Laboratory		
Material	Mean value	*Sr*	*r*	*(r)*	*SR*	*R*	(*R*)
9040	23.6	1.12	3.18	13.5	2.56	7.24	30.7
DN120	25.5	0.72	2.03	8.0	2.53	7.15	28.1
DN127	29.4	1.36	3.86	13.1	1.94	5.50	18.7
9020	32.5	1.31	3.71	11.4	3.07	8.69	26.8

[A] $p = 4$, $q = 4$, and $n = 2$.
Time period for precision is days.
Sr = within laboratory standard deviation, measurement units.
r = repeatability, in measurement units.
(r) = repeatability, percent.
SR = (total) between laboratory standard deviation, measurement units.
R = reproducibility, measurement units.
(R) = reproducibility, percent.
[B] Within cell standard deviation for all four materials rejected for one laboratory.
[C] One laboratory material three, cell average rejected; two laboratories one material each, cell standard deviation rejected.
[D] One laboratory material one, cell average rejected; one laboratory three materials, cell standard deviation rejected.

9.6 This precision evaluation program had an inadequate number of laboratories for a satisfactory evaluation of the Test Method D 2084 testing precision. In addition as Table 1 indicates, there were a number of rejections of cell values in the database (see Tables 2 and Tables 3 format of Practice D 4483). This further reduces the confidence of the final results for the evaluated precision.

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

10. Keywords

10.1 acrylonitrile-/butadiene copolymers; carbon black; NBR