Designation: D3848 – 03 (Reapproved 2013)

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

This standard is issued under the fixed designation D3848; 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]

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 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.

[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.

Current edition approved Nov. 1, 2013. Published January 2014. Originally approved in 1980. Last previous edition approved in 2009 as D3848 – 03 (2009). DOI: 10.1520/D3848-03R13.

[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.

[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 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 D3896.

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 D3182.

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 Methods D1646 or rheological properties in accordance with Test Method D6204, vulcanizing characteristics in accordance with Test Method D2084, or Test Method D5289, 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 D3182. 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:*

	Duration, min	Accumulative, min
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.		