Designation: D 3184 – 89 (Reapproved 2001)

Standard Test Methods for Rubber—Evaluation of NR (Natural Rubber)[1]

This standard is issued under the fixed designation D 3184; 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 formulas, mixing procedures, and test methods for the evaluation and production control of natural rubber (NR).

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— Tension[2]

D 1485 Test Methods for Rubber from Natural Sources—Sampling and Sample Preparation[2]

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 Meter[2]

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets[2]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]

3. Significance and Use

3.1 These tests 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 rubber samples in a standard formula.

4. Standard Test Formulas

4.1 *Standard Formulas*:

		Quantity, Parts by Mass, and Formula No.		
Material	NIST SRM No.	1A Gum	2A Black Filled	3A[A] Gum
Natural rubber	...	100.00	100.00	100.00
Zinc oxide	370	6.00	5.00	6.00
Sulfur	371	3.50	2.25	3.50
Stearic acid	372	0.50	2.00	0.50
Oil furnace black[B]	378	...	35.00	...
Mercaptobenzothiazole	383	0.50	...	...
TBBS[C]	384	...	0. 70	0.70
Total		110.50	144.95	110.70
Batch factor:[D]				
Mill		3.0	3.0	3.0
Miniature internal mixer:[E]				
Cam Head		0.57	0.48	0.57
Banbury Head		0.50	0.42	0.50

[A] Recommended formula for Test Method D 2084 vulcanization characteristics using oscillating disk cure meter.

[B] The current Industry Reference Black may be used in place of NIST 378, although slightly different results may be obtained.

[C] *N-tert*-butyl-2-benzothiazole sulfenamide.

[D] For 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.

[E] For MIM batches weigh the rubber carbon black to the nearest 0.1 g, the compounding material blend to the nearest 0.01 g, and individual compounding materials, if used, to the nearest 0.001 g. For the MIM procedure, it is recommended that a blend of compounding materials, including black, be prepared to improve accuracy in the weighing of these materials. This material blend is prepared by blending a proportional mass of each material in a dry powder 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 samples in accordance with Test Methods D 1485.

6. Mixing Procedures

6.1 The following three mixing procedures are offered as follows:

6.1.1 *Mill Method A*—For gum formulas 1A and 3A.

6.1.2 *Mill Method B*—For black formula 2A.

6.1.3 *MIM Method C*—For formula 1A, 2A, and 3A.

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

6.2 *Mill Method A*:

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

6.2.2 Mixing shall be done with the mill roll temperature maintained at 70 ± 5°C (158 ± 9°F). The indicated mill openings are desired and should be maintained insofar as

[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and are the direct responsibility of Subcommittee D 11.22 on Natural Rubber.

Current edition approved March 31, 1989. Published May 1989. Originally published as D 3184 – 73. Last previous edition D 3184 – 88.

[2] *Annual Book of ASTM Standards*, Vol 09.01.

possible to provide a standard for uniform breakdown of the rubber due to milling.

6.2.3 *Mixing Cycle—Gum Compound*:

	Duration, min	Accumulative, min
Set the mill opening at 0.20 mm (0.008 in.) and pass the rubber through the rolls twice without banding.	1	1
Band with the mill opening at 1.40 mm (0.055 in.) and break down, opening the mill to 1.90 mm (0.075 in.) as the band becomes smooth.	4	5
Add the stearic acid.	2	7
Add the zinc oxide, sulfur, and accelerator.	4	11
Make three ¾ cuts from each side.	2	13
Cut the stock from the mill. Set the opening at 0.80 mm (0.032 in.) and pass the rolled stock endwise through the mill six times.	2	15
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.	3	18
Total Time	18	—

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

6.2.3.9 From this stock, cut enough sample to allow testing of compounded viscosity in accordance with Test Methods D 1646 or curing characteristics in accordance with Test Method D 2084, or both, if these are desired. Sheet off the stock from the mill at a setting to give an approximate finish gage of 2.2 mm (0.085 in.). Cool on a flat, dry metal surface.

6.2.3.10 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.

6.3 *Mill Method B*:

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

6.3.2 Mixing shall be done with the mill roll temperature maintained at 70 ± 5°C (158 ± 9°F). The indicated mill openings are desired and should be maintained insofar as possible to provide a standard for uniform breakdown of the rubber due to milling.

6.3.3 *Mixing Cycle—Black-Filled Compound*:

	Duration, min	Accumulative, min
6.3.3.1 Set the mill opening at 0.20 mm (0.008 in.) and pass the rubber through the rolls twice without banding.	1	1
6.3.3.2 Band the rubber on the front roll with the mill opening at 1.40 mm (0.055 in.). Make two ¾ cuts from each side.	2	3
6.3.3.3 Set the mill opening at 1.70 mm (0.067 in.). Add zinc oxide. Make one ¾ cut from each side.	2	5
6.3.3.4 Add the carbon black evenly across the mill at a uniform rate. When about half the black is incorporated, add the stearic acid and open the mill to 1.90 mm (0.075 in.). Make one ¾ cut from each side then add the remainder of the carbon black.	14	19
Note—Do not cut any stock while free carbon black is evident in the bank or on the milling surface. Be certain to run any pigments that drop through the mill to the milling stock.		
6.3.3.5 Add the accelerator, taking care to avoid any loss. Sweep the mill pan and add until all the pigment is in the batch. Make three ¾cuts from each side.	2	21
6.3.3.6 Add the sulfur and that which falls into the mill pan. Make one ¾ cut from each side.	3	24
6.3.3.7 Cut the stock from the mill. Set the opening at 0.80 mm (0.032 in.) and pass the rolled stock endwise through the mill six times.	2	26
6.3.3.8 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.	3	29
Total Time	29	—

6.3.3.9 Check the batch mass and record. If it differs from the theoretical value by more than 0.5 % reject the batch.

6.3.3.10 From this stock, cut enough sample to allow testing of compounded viscosity in accordance with Test Methods D 1646, or curing characteristics in accordance with Test Method D 2084, or both, if these are desired. Sheet off the stock from the mill at a setting to give an approximate finish gage of 2.20 mm (0.085 in.). Cool on a flat, dry metal surface.

6.3.3.11 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.

6.4 *Method C—Miniature Internal Mixer Mix*:

6.4.1 For general mixing procedure, refer to Practice D 3182. Mix with the head temperature of the miniature internal mixer maintained at 60 ± 3°C (140 ± 5°F) and the unloaded rotor speed at 6.3 to 6.6 rad/s (60 to 63 rpm).

6.4.2 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 of 0.5 mm (0.02 in.) thick. Cut the sheet into strips that are approximately 25 mm (1 in.) wide if desired.

6.4.3 *Mixing Cycle*:

	Duration, min	Accumulative, min
6.4.3.1 Charge the mixing chamber with the rubber strips and the blended materials, lower the ram, and start the timer.	0	0
6.4.3.2 Allow to mix.	1	1
6.4.3.3 Raise the ram, add carbon black, sweep the orifice, and lower the ram.	1	2
6.4.3.4 Allow the batch to mix, raising the ram momentarily to sweep down the materials, if necessary.	3	5

6.4.3.5 Turn off the motor, raise the ram, remove the head and discharge the batch. Measure and record the maximum batch temperature if desired.

6.4.3.6 Immediately pass the batch twice through a laboratory mill maintained at 50 ± 5°C (122 ± 9°F) and with the roll separation of 3 mm (0.125 in.).

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

6.4.3.8 Cut a specimen for testing vulcanization characteristics in accordance with Test Method D 2084, if required.

Condition the specimen for 1 to 24 h at 23 ± 3°C (73.4 ± 5.4°F) before testing.

6.4.3.9 If either compound viscosity or stress-strain testing is required, pass the rolled compound end-wise through the mill six times with the mill rolls set at 50 ± 5°C (122 ± 9°F) and 0.8 mm (0.032 in.).

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

6.4.3.11 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.

6.4.3.12 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 test slabs and vulcanize them in accordance with Practice D 3182.

7.1.1 The recommended standard cure times are 10, 20, 40, and 80 min at 140°C (284°F).

7.1.2 Condition the cure sheets for 16 to 96 h at a temperature of 23 ± 2°C (73.4 ± 3.6°F).

NOTE 2—Quality control of production may require testing within 1 to 6 h, to provide close surveillance of the plant operations; however, slightly different results may be obtained.

7.1.3 Prepare test specimens in accordance with Practice D 3182 and obtain modulus, tensile, and elongation parameters in accordance with Test Methods D 412.

7.2 An alternative to measuring stress-strain properties of vulcanizates is the cure meter measurement of vulcanization parameters in accordance with Test Method D 2084.

7.2.1 The recommended standard test conditions are 1.7 Hz (100 cpm) oscillation frequency, 1 ± 0.03° amplitude of oscillation, and 160 ± 0.3°C (320 ± 0.5°F) die temperature.

7.2.2 The recommended standard test parameters are M_L, M_H, t_{S1}, t' (50), and t' (90). For formula 3A, use M_{HF} instead of M_H.

8. Precision and Bias

8.1 Precision and bias statements are in the process of being prepared as prescribed in Practice D 4483. They will be added to this test method when they are completed.

9. Keywords

9.1 mill method; miniature mixer; mixing procedures; natural rubber; sample preparation; test methods