Designation: D3188 – 06 (Reapproved 2010)

Standard Test Methods for Rubber—Evaluation of IIR (Isobutene-Isoprene Rubber)[1]

This standard is issued under the fixed designation D3188; 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 formula, mixing procedures, and test methods for the evaluation and production control of non-halogenated isobutene-isoprene rubbers (IIR), commonly known as butyl rubber.

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 purpose 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 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 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.

6.1.1 *Method A*—Mill mix (6.2)

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

6.1.3 *Method C*—Lab Banbury (6.4)

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

6.2 *Method A—Mill Procedure:*

6.2.1 For general mixing procedures, refer to Practice D3182. Mix with the mill roll temperature maintained at 50 ± 5°C (122 ± 9°F). The indicated mill openings should be maintained as nearly as possible to provide a standard degree of breakdown for the rubber due to milling. Necessary adjustments may be made to maintain a good working bank at the nip of the rolls.

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

[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	NBS or IRM No.	Quantity, Parts by Mass
IIR	...	100.00
Zinc oxide	[A]	3.00
Sulfur	[A]	1.75
Stearic acid	[A]	1.00
Oil furnace black[B]	378	50.00
TMTD[C]	[A]	1.00
Total mass		156.75
Batch factor:		
Mill[D]		2.0
Miniature internal mixer[E]		
Cam Head		0.46
Banbury Head		0.40

[A] Use current IRM/SRM.
[B] The current industry reference black may be used in place of NBS 378, although slightly different results may be obtained.
[C] Tetramethylthiuram disulfide. NBS has discontinued supply of TMTD. A new source of supply material is available as IRM 1 from Forcoven Products Inc., P.O. Box 1536, Humble, TX 77338. A research report can be obtained from ASTM Headquarters. Request RR: D-11-1034.
[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.

with Test Methods D1646 or D6204, and vulcanization characteristics in accordance with Test Methods D2084 or D5289.

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 *Method B—Miniature Internal Mixer Mix:*

6.3.1 For general mixing procedure, refer to Practice D3182. 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).

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

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 head, and discharge the batch. Measure and record the maximum batch temperature 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 Method 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 vulcanization time is 40 min at 150°C (302°F).

7.1.2 Condition the cured sheets for 16 to 96 h at a temperature of 23 ± 2°C (73.4 ± 3.6°F) prior to making stress-strain tests.

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

7.1.3 Prepare test specimens and obtain the tensile stress, tension, and elongation in accordance with Test Methods D412.

8. Testing for Curing Characteristics using Cure Meters

8.1 An alternative to measuring vulcanization characteristics by means of tensile stress measurement on vulcanizates is the measurement of vulcanization characteristics in accordance with Test Method D2084 (Oscillating Disk Cure Meter Method) or Test Method D5289 (Rotorless Cure Meter Method). These methods will not produce equal results.

8.1.1 The recommended Test Method D2084 test conditions are 1.67 Hz (100 cpm) oscillation frequency, 1° oscillation amplitude, 160°C die temperature, 40-min test time, and no preheating. The recommended Test Method D5289 test conditions are 1.67 Hz (100 cpm) oscillation frequency, 0.5° oscillation amplitude, 160°C die temperature, 40-min test time, and no preheating. Test condition tolerances are specified by the test methods.

8.1.2 The recommended standard test parameters are: M_L, M_H, t_{s1}, t′50, and t′90.