



Designation: D 3188 – 00

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

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1. Scope

1.1 These test methods specify 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:

- D 412 Test Methods for Vulcanized Rubber and Thermoplastic Rubbers and Thermoplastic Elastomers—Tension²
- D 1646 Test Method 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

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 Rubber.

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

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:

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

^AUse current IRM/SRM.

^BThe current industry reference black may be used in place of NBS 378, although slightly different results may be obtained.

^CTetramethylthiuram 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.

^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.

^EFor 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 Practice D 3896.

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 D 3182. Mix with the mill roll temperature maintained at $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$). 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:

	Duration, min	Accumulative, min
Set the mill opening at 0.65 mm (0.025 in.) and band the rubber on the slow roll.	1	1
Mix the carbon black and the stearic acid and add evenly across the mill rolls at a uniform rate. Open the mill nip at intervals to maintain a constant rolling bank. When all the carbon black has been added, make a $\frac{1}{4}$ cut from each side. Note—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 materials that drop through the mill.	10	11
Add all the other materials.	3	14
Make three $\frac{1}{4}$ cuts from each side and cut the batch from the mill.	2	16
Set the mill opening at 0.8 mm (0.032 in.) and pass the rolled batch end-ways through the mill six times.	2	18

6.2.2.6 Determine and record the batch mass. If it differs from the theoretical value by more than 0.5 %, reject the batch. Remove enough sample from the batch to allow testing of compound viscosity in accordance with Test Method D 1646 or vulcanization characteristics in accordance with Test Method D 2084, or both, if these are desired. 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.

6.2.2.7 If tensile stress is required, sheet off the compound from the mill at a setting to give a finished gage of 2.2 mm (0.085 in.) by passing the folded stock between the 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.

6.2.2.8 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.

6.3 Method B—Miniature Internal Mixer Mix:

6.3.1 For general mixing procedure, refer to Practice D 3182. 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 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 $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$) 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:

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

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

6.3.3.6 Immediately pass the batch twice through a laboratory mill maintained at $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$) and with the roll separation of 3 mm (0.125 in.).

6.3.3.7 Determine and record the batch mass. If it differs from the theoretical value by more than 0.5 %, reject the batch. Remove enough sample from the batch to allow testing of compound viscosity in accordance with Test Method D 1646 or vulcanization characteristics in accordance with Test Method D 2084, or both, if these are desired. 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.

6.3.3.8 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 $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$) four times always in the same direction to obtain the effects of mill direction. Cool on a flat, dry metal surface.

6.3.3.9 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	Accumulative, Min
6.4.2.1		
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.	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		