

# Standard Test Methods for Rubber—Evaluation of IR (Isoprene Rubber)<sup>1</sup>

This standard is issued under the fixed designation D3403; 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 ( $\epsilon$ ) 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 isoprene rubber (IR).

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:<sup>2</sup>

- 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 Unvul-

nized Rheological Properties Using Rotorless Shear Rheometers

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

## 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.

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

## 4. Standard Test Formula

4.1 *Standard Formula*—See [Table 1](#).

## 5. Sample Preparation

5.1 Obtain and prepare the test samples in accordance with Practice [D3896](#).

## 6. Mixing Procedures

6.1 The compound may be prepared in an internal mixer, on a mill, or in a miniature internal mixer. It is not implied that comparable results will be obtained. The following mixing procedures are provided:

6.1.1 *Method A—Internal Mixer for Initial and Final Mix*—(6.2),

6.1.2 *Method B—Initial Internal Mix with Final Mill Mix*—(6.3),

6.1.3 *Method C—Mill Mix*, (6.4), and

6.1.4 *Method D—Miniature Internal Mix* (6.5).

6.2 *Internal Mixer for Initial and Final Mix (Method A):*

6.2.1 For general mixing procedures refer to Practice [D3182](#).

6.2.2 *Mixing Cycle for Internal Mixer Initial Mix (Methods A,B)*—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 Pass the batch immediately through the standard laboratory mill three times, with a mill opening of 6.0 mm (0.25 in.) and roll temperature of  $40 \pm 5^\circ\text{C}$  ( $104 \pm 9^\circ\text{F}$ ).

6.2.2.3 Allow the batch to rest for 1 to 24 h.

<sup>1</sup> 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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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto: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 IR Test Formula**

| Material                                             | IRM-SRM No. <sup>A</sup> | Quantity, Parts by Mass |
|------------------------------------------------------|--------------------------|-------------------------|
| Isoprene rubber (IR)                                 | ...                      | 100.00                  |
| Zinc oxide                                           | <sup>A</sup>             | 5.00                    |
| Sulfur                                               | <sup>A</sup>             | 2.25                    |
| Stearic acid                                         | <sup>A</sup>             | 2.00                    |
| Oil furnace black <sup>B</sup>                       | <sup>A</sup>             | 35.00                   |
| TBBS <sup>C</sup>                                    | <sup>A</sup>             | 0.70                    |
| Total                                                |                          | 144.95                  |
| Batch factor for mill mix <sup>D</sup>               |                          | 3.00                    |
| Batch factor for internal mixer <sup>D</sup>         |                          | 10.00                   |
| Batch factor for MIM mix <sup>E</sup> (Cam Head)     |                          | 0.50                    |
| Batch factor for MIM mix <sup>E</sup> (Banbury Head) |                          | 0.43                    |

<sup>A</sup> Use current IRM/SRM.

<sup>B</sup> The current Industry Reference Black (IRB) shall be used.

<sup>C</sup> TBBS is *N-tert-butyl-2-benzothiazolesulfenamide*.

<sup>D</sup> For mill and internal mixer batches, weigh the rubber, carbon black, and oil to the nearest 1.0 g, the sulfur and the TBBS accelerator to the nearest 0.02 g, and the other compounding materials to the nearest 0.1 g.

<sup>E</sup> For MIM mixes, weigh the rubber and carbon black 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, excluding carbon black, be prepared to improve the accuracy of the weighing of these materials. This material blend is prepared by blending a proportional mass of each material in a dry powder blender such as a biconical blender or vee blender. A mortar and pestle may be used for blending small quantities.

**TABLE 2 Internal Mixer - Initial Cycle**

|                                                                                                                                                                                  | Duration, min | Accumulative, min |
|----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------|-------------------|
| Adjust the internal mixer temperature to achieve the discharge conditions outlined below. Close the discharge gate, start the rotors at 8.1 rad/s (77 r/min), and raise the ram. | 0.0           | 0.0               |
| Charge one-half of the rubber, all of the zinc oxide, carbon black, stearic acid, and then the other one-half of the rubber. Lower the ram.                                      | 0.5           | 0.5               |
| Allow the batch to mix.                                                                                                                                                          | 3.0           | 3.5               |
| Raise the ram, and clean the mixer throat and the top of the ram. Lower the ram.                                                                                                 | 0.5           | 4.0               |
| Allow the batch to mix until a temperature of 170°C (338°F) or a total of 6 min is reached, whichever occurs first. Discharge the batch.                                         | 2.0           | 6.0               |

6.2.3 *Mixing Cycle for Internal Mixer Final Mix (Method A)*—See **Table 3**.

6.2.3.1 After mixing according to **Table 3**, measure and record the batch mass. If it differs from the theoretical value by more than 0.5 %, discard the batch.

6.2.3.2 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**.

6.2.3.3 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**.

6.3 *Internal Mixer Initial Mix with Final Mill Mix (Method B)*:

**TABLE 3 Internal Mixer - Final Cycle**

|                                                                                                                                                                                             | Duration, min | Accumulative, min |
|---------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------|-------------------|
| Adjust the internal mixer temperature to 40 ± 5°C (104 ± 9°F), turn off steam and turn on full cooling water to the rotors, start the rotors at 8.1 rad/s (77 r/min), and raise the ram.    | 0.0           | 0.0               |
| Charge one-half of 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. | 0.5           | 0.5               |
| 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               |
| With the rolls of a standard laboratory mill maintained at 40 ± 5°C (104 ± 9°F), and set at 0.8 mm (0.032 in.) opening, pass the rolled batch endwise through the mill six times.           | 2.0           | 5.0               |
| Open the rolls to give a minimum thickness of 6 mm (0.25 in.) and pass the compound through four times, folding it back on itself each time.                                                | 1.0           | 6.0               |

6.3.1 For general mixing procedures, refer to Practice **D3182**.

6.3.2 *Mixing Cycle for Initial Mix*—Follow the procedure outlined in **6.2.2**.

6.3.3 *Mixing Cycle for Mill Final Mix (Method B)*—See **Table 4**.

6.3.3.1 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.

6.3.3.2 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**.

**TABLE 4 Mill Final Mixing Cycle**

|                                                                                                                                                                  | Duration, min | Accumulative, min |
|------------------------------------------------------------------------------------------------------------------------------------------------------------------|---------------|-------------------|
| Set the mill opening at 1.90 mm (0.075 in.) and temperature at 70 ± 5°C (158 ± 9°F). Band the masterbatch on the slow roll.                                      | 0             | 0                 |
| Add the TBBS accelerator, taking care to avoid any loss. Sweep the mill pan and add until all the accelerator is in the batch. Make three ¾ cuts from each side. | 3             | 3                 |
| Add the sulfur and that which falls into the mill pan. Make one ¾ cut from each side.                                                                            | 3             | 6                 |
| Cut the batch from the mill and set the mill roll opening at 0.8 mm (0.032 in.). Pass the rolled stock endwise through the mill six times.                       | 2             | 8                 |
| Open the mill to give a minimum stock thickness of 6 mm (0.25 in.) and pass the compound through the mill four times, folding it back on itself each time.       | 1             | 9                 |