



Designation: D 3403 – 97 (Reapproved 2002)

Standard Test Methods for Rubber—Evaluation of IR (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 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:

- D 412 Test Methods for Vulcanized Rubber and Thermoplastic Rubbers and Thermoplastic Elastomers—Tension²
- 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 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
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method³

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.

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 Annual Book of ASTM Standards, Vol. 14.02.

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

4. Standard Test Formula

4.1 Standard Formula:

Material	Standard Testing Materials	NIST SRM No.	Quantity, Parts by Mass
Isoprene rubber (IR)	...	...	100.00
Zinc oxide ^A	...	370	5.00
Sulfur ^B	SRM371	371	2.25
Stearic acid ^B	SRM372	372	2.00
Oil furnace black ^C	IRB	378	35.00
TBBS ^{B,D}	RM8384	384	0.70
Total			144.95
Batch factor for mill mix ^E			3.00
Batch factor for internal mixer ^E			10.00
Batch factor for MIM mix ^F (Cam Head)			0.50
Batch factor for MIM mix ^F (Banbury Head)			0.43

^A An IRM is being prepared and will be included in these test methods at the December 1993 meeting.

^B Available from NIST.

^C The current Industry Reference Black (IRB) shall be used.

^D *N-tert-butyl-2-benzothiazolesulfenamide.*

^E For mill and internal mixer batches, weigh the rubber, carbon black and oil to the nearest 1.0 g, the sulfur and accelerator to the nearest 0.02 g, and the other compounding materials to the nearest 0.1 g.

^F For MIM batches, 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 accuracy in the weighing of these materials. The compounding material blend is prepared by blending a proportional mass of each material in a bioconical or 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 D 3896.

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 *Method A—Internal Mixer for Initial and Final Mix:*

6.2.1 For general mixing procedures refer to Practice D 3182.

6.2.2 *Mixing Cycle for Initial Mix:*

6.2.2.1

	Duration, min	Accumulative, min
Adjust the internal mixer temperature to achieve the discharge conditions outlined in 6.2.2.5. Close the discharge gate, start the rotor, and raise the ram.	0	0

6.2.2.2

	Duration, min	Accumulative, min
Charge $\frac{1}{2}$ of the rubber, all the zinc oxide, carbon black, stearic acid, and then the other $\frac{1}{2}$ of the rubber. Lower the ram.	0.5	0.5

6.2.2.3

	Duration, min	Accumulative, min
Allow the batch to mix.	3.0	3.5

6.2.2.4

	Duration, min	Accumulative, min
Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	0.5	4.0

6.2.2.5

	Duration, min	Accumulative, min
Allow the batch to mix until a temperature of 170°C (338°F) or a total mixing time of 6 min is reached, whichever occurs first. Discharge the batch.	2.0 max	6.0 max

6.2.2.6 Determine and record the batch mass. If the mass differs by more than $\pm 5\%$ of the theoretical mass, discard the batch.

6.2.2.7

	Duration, min	Accumulative, min
Pass the batch immediately through the standard laboratory mill three times, set at 6.0 mm (0.25 in.) and 70 $\pm$ 5°C (158 $\pm$ 9°F).	1.0	7.0 max

6.2.2.8 Rest the stock for 1 to 24 h.

6.2.3 *Mixing Cycle for Final Mix:*

6.2.3.1

	Duration, min	Accumulative, min
Cool the internal mixer with full cooling water on the rotor to 40 $\pm$ 5°C (104 $\pm$ 9°F), start the rotor, raise the ram.	0	0

6.2.3.2

	Duration, min	Accumulative, min
Leave the cooling water on and the steam off, charge $\frac{1}{2}$ of the masterbatch, with all the sulfur and accelerator rolled into this portion of the masterbatch before feeding to the mixer. Add the remaining portion of the masterbatch. Lower the ram.	0.5	0.5

6.2.3.3

Allow the batch to mix until a temperature of 110 $\pm$ 5°C (230 $\pm$ 9°F) or a total mixing time of 3 min is reached, whichever occurs first. Discharge the batch.

6.2.3.4 Determine and record the batch mass. If the mass differs by more than $\pm 5\%$ of the theoretical mass discard the batch.

6.2.3.5

	Duration, min	Accumulative, min
With the mill roll temperature set at 70 $\pm$ 5°C (158 $\pm$ 9°F) and opening at 0.3 mm (0.032 in.); pass the rolled batch endwise through the mill six times.	2.0	5.0

6.2.3.6

	Duration, min	Accumulative, min
Open the mill to give a minimum thickness of 6 mm (0.25 in.) and pass the stock through the mill four times, folding it back on itself each time.	1.0	6.0

6.2.3.7 From this batch cut enough sample to allow testing of compound viscosity in accordance with Test Method D 1646 or curing 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°C (73.4 $\pm$ 5.4°F) before testing.

6.2.3.8 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°C (122 $\pm$ 9°F) four times always in the same direction to obtain the effects of mill direction. Cool on a flat, dry metal surface.

6.2.3.9 For routine laboratory testing, condition the sheeted compound for 1 to 24 h at 23 $\pm$ 3°C (73.4 $\pm$ 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 $\pm$ 5 % relative humidity.

6.3 *Method B—Initial Internal Mix with Final Mill Mix:*

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

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

6.3.3 *Mixing Cycle for Final Mix:*

6.3.3.1

	Duration, min	Accumulative, min
With the temperature set at 70 $\pm$ 5°C (158 $\pm$ 9°F) and the opening at 1.90 mm (0.075 in.), band the masterbatch on the slow roll.	0	0

6.3.3.2

	Duration, min	Accumulative, min
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 $\frac{1}{4}$ cuts from each side.	3.0	3.0

6.3.3.3

	Duration, min	Accumulative, min
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Add the sulfur and that which falls into the mill pan. Make one $\frac{3}{4}$ cut from each side.

3.0

6.0

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 $\frac{3}{4}$ cut from each side, then add the remainder of the carbon black.

14

19

6.3.3.4

Duration,
min
2.0

Accumulative,
min
8.0

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.

6.3.3.5

Duration,
min
1.0

Accumulative,
min
9.0

Open the mill to give a minimum batch thickness of 6 mm (0.25 in.) and pass the batch through the rolls four times, folding it back on itself each time.

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

6.3.3.7 From this batch, cut enough sample to allow testing of compound viscosity in accordance with Test Method D 1646 or curing 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 Method C—Mill Mix:

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

6.4.2 Mixing Cycle:

6.4.2.1

Duration,
min
1

Accumulative,
min
1

With the mill roll temperature set at $70 \pm 5^\circ\text{C}$ ($158 \pm 9^\circ\text{F}$) and the opening at 0.20 mm (0.008 in.), pass the rubber through the rolls twice without banding.

6.4.2.2

Duration,
min
2

Accumulative,
min
3

Band the rubber on the slow roll with the mill opening at 1.40 mm (0.55 in.). Make two $\frac{3}{4}$ cuts from each side.

6.4.2.3

Duration,
min
2

Accumulative,
min
5

Set the mill opening at 1.70 mm (0.067 in.), add the zinc oxide. Make one $\frac{3}{4}$ cut from each side.

6.4.2.4

Duration,
min

Accumulative,
min

NOTE 1—Do not cut any stock while free carbon black is evident in the bank or on the milling surface. Be certain to return any materials that drop through the mill to the milling stock.

6.4.2.5

Duration,
min
3

Accumulative,
min
22

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 $\frac{3}{4}$ cuts from each side.

6.4.2.6

Duration,
min
3

Accumulative,
min
25

Add the sulfur and that which falls into the mill pan. Make one $\frac{3}{4}$ cut from each side.

6.4.2.7

Duration,
min
2

Accumulative,
min
27

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.

6.4.2.8

Duration,
min
1

Accumulative,
min
28

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.

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

6.4.2.10 From this stock, cut enough sample to allow testing of compound viscosity in accordance with Test Method D 1646, or curing 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.

6.4.2.11 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 $50 \pm 5^\circ\text{C}$ ($122 \pm 9^\circ\text{F}$) four times always in the same direction to obtain the effects of mill direction. Cool on a flat, dry metal surface.

6.4.2.12 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.

6.5 Method D—Miniature Internal Mix:

6.5.1 For general mixing procedures 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 set at 6.3 to 6.6 rad/s (60 to 63 rpm).

6.5.2 Prepare the rubber by passing it through a mill one time with the temperature set at $70 \pm 5^\circ\text{C}$ ($158 \pm 9^\circ\text{F}$) and the