



Designation: D 3958 – 05

Standard Test Methods for Rubber—Evaluation of BIIR and CIIR (Halogenated Isobutene—Isoprene Rubber)¹

This standard is issued under the fixed designation D 3958; 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 of halogenated isobutene-isoprene rubbers (BIIR and CIIR).

1.2 Both mill and miniature internal mixer procedures are given.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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 consult and establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*²

D 412 Test Methods for Vulcanized Rubber and Thermoplastic Elastomers—Tension

D 1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)

D 2084 Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter

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

D 3896 Practice for Rubber From Synthetic Sources—Sampling

D 4483 Practice for Evaluating Precision for Test Method

Standards in the Rubber and Carbon Black Manufacturing Industries

D 5289 Test Method for Rubber Property—Vulcanization Using Rotorless Cure Meters

D 6204 Test Method for Rubber—Measurement of Unvulcanized Rheological Properties Using Rotorless Shear Rheometers

3. Significance and Use

3.1 These tests are intended mainly 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 samples in a standard formula.

3.2 These tests may be used to obtain values for quality control acceptance of rubber.

4. Standard Test Formulas

4.1 *Standard Formulas*—See **Table 1**.

5. Sample Preparation

5.1 Obtain and prepare the test samples in accordance with Practice **D 3896**.

6. Mixing Procedures

6.1 For general mixing procedures, refer to Practice **D 3182**.

6.1.1 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.

6.2 *Mixing Cycles:*

6.2.1 *Method A—Mill Mix:*

6.2.1.1 Mix with the mill roll temperature maintained at $40 \pm 5^\circ\text{C}$ ($104 \pm 9^\circ\text{F}$). The indicated mill openings should be maintained insofar as possible to provide a standard for uniform breakdown of the rubber due to milling. Necessary adjustments may be made to maintain a good working bank at the nip of the rolls.

6.2.1.2 Use the carbon black in accordance with 4.4 of Practice **D 3182**. This is critical with halogenated IIR when the simple zinc oxide is used. (See **Table 2**.)

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

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 Formulas

Material	Quantity Parts by Mass Formula
BIIR or CIIR	100.00
Zinc oxide	5.00
Stearic acid	1.00
Current IRB	40.00
Total mass	146.00
Batch Factors:	
Mill	2.0 ^B
MIM	0.48 ^C

^A Use current IRM/SRM.

^B Weigh the rubber and the carbon black to the nearest 0.5 g, the zinc oxide to the nearest 0.1 g, and the stearic acid to the nearest 0.02 g.

^C Select a batch factor for the miniature internal mixer so that the mixing chamber volume will be 75 % filled with stock. A batch factor of 0.48 is suggested for the cam blade head with 85-cm³ mixing chamber capacity. Calculate all parts to the nearest 0.01 part. Weigh all materials to the nearest 0.01 g.

6.2.1.4 If required, cut samples from the batch to allow testing of compound viscosity and processability in accordance with Test Methods D 1646 or D 6204, and vulcanization characteristics in accordance with Test Methods D 2084 or D 5289. Condition the samples in accordance with Practice D 3182.

6.2.1.5 For routine laboratory testing, condition the sheeted compound for 0.5 to 6 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 0.5 to 6 h in a closed container to prevent absorption of moisture from the air, or in an area controlled at 35 ± 5 % relative humidity.

6.2.2 Method B—Miniature Internal Mixer (MIM) Mix:

6.2.2.1 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 empty chamber rotor rotational frequency at 1 to 1.05 r/s (60 to 63 rpm).

6.2.2.2 Condition the carbon black in accordance with 4.4 of Practice D 3182. This is critical with halogenated butyl rubber when the simple zinc oxide cure is used. (See Table 3.)

6.2.2.3 Turn off the motor, raise the ram, remove the head, and discharge the batch. Record the batch temperature.

6.2.2.4 Immediately pass the batch twice through a $40 \pm 5^\circ\text{C}$ ($104 \pm 9^\circ\text{F}$) mill set at 3 mm (0.12 in.).

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

6.2.2.6 If required, cut samples from the batch to allow testing of compound viscosity and processability in accordance with Test Methods D 1646 or D 6204, and vulcanization characteristics in accordance with Test Methods D 2084 or D 5289. Condition the samples in accordance with Practice D 3182.

6.2.2.7 After samples are cut for viscosity, processability, or vulcanization testing, pass the remaining stock endwise through the mill rolls six times, rolling up after each pass. The mill should be set at 0.8 mm opening and $40 \pm 5^\circ\text{C}$. After the last pass, the stock should be sheeted off the mill at a setting that will give a sheet approximately 2.2 mm thick after shrinkage. Make three to four passes always in the same direction to obtain grain effects. Allow to cool on a flat, dry metal surface.

6.2.2.8 For routine laboratory testing, condition the sheeted compound for 0.5 to 6 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 0.5 to 6 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 Internal Mixer Procedure:

6.3.1 For general mixing procedure refer to Method D 3182.

6.3.2 Mixing Cycle-Initial Mix—See Table 4.

6.3.2.1 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.

6.3.2.2 Determine and record the batch mass; if the mass differs by more than 0.5 % of the theoretical mass, discard the batch.

6.3.2.3 Pass the batch immediately through the standard laboratory mill three times, set at 6.0 mm (0.25 in.) and $40 \pm 5^\circ\text{C}$ ($104 \pm 9^\circ\text{F}$).

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

6.3.3 Final Mix—See Table 5.

6.3.3.1 Adjust the internal mixer temperature to $40 \pm 5^\circ\text{C}$ ($104 \pm 9^\circ\text{F}$), turn off steam and turn on full cooling water to the rotors, start the rotors at 8.1 rad/s (77 rpm), and raise the ram.

6.3.3.2 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.

6.3.3.3 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 ± 5 % relative humidity.

6.3.3.4 If required, cut samples from the batch to allow testing of compound viscosity and processability in accordance with Test Methods D 1646 or D 6204, and vulcanization characteristics in accordance with Test Methods D 2084 or D 5289. Condition the samples in accordance with Practice D 3182.

7. Preparation and Testing of Vulcanizates

7.1 For tension testing, prepare the test sheets and vulcanize them in accordance with Practice D 3182.

7.1.1 The recommended standard cure times for the mill mixes are 15, 30, and 45 min at 150°C (302°F). The recommended cure time for the miniature internal mixer compound is 30 min at 150°C .

7.1.2 Condition the cured sheets for 16 to 96 h at a temperature of $23 \pm 2^\circ\text{C}$ ($73 \pm 3.6^\circ\text{F}$) prior to making tension tests.

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

7.1.3 Prepare test specimens and obtain stress at 300 % elongation, tensile stress, and elongation parameters in accordance with Test Methods D 412.

