



Designation: D 3958 – 00

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.

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:

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

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.

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.

Current edition approved May 10, 2000. Published June 2000. Originally published as D 3958 – 80. Last previous edition D 3958 – 95.

² Annual Book of ASTM Standards, Vol 09.01.

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

4. Standard Test Formulas

4.1 Standard Formulas:

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

^AUse current IRM/SRM.

^BWeigh 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.

^CSelect 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.

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 ± 5°C (104 ± 9°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.

	Duration, min	Accumulative, min
Set the mill opening at 0.65 mm (0.025 in.) and band the rubber on the slower roll.	1	0

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.

10	11
Duration, min	Accumulative, min
3	14
2	16
2	18
Total time	18 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 pigments that drop through the mill to the milling stock.

Add the zinc oxide.
Make three $\frac{1}{4}$ cuts from each side and cut the batch from the mill.

Set the mill opening at 0.8 mm (0.032 in.) and pass the rolled stock end-ways through the mill six times.

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. Cut enough sample from the batch to allow testing of vulcanization characteristics in accordance with Test Method D 2084.

6.2.1.4 Sheet off the stock from the mill at a setting to give approximately a finish gage of 2.2 mm (0.085 in.). Cool on a flat, dry metal surface.

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.

Charge the pigments and carbon black first, followed by the rubber cut into approximately 20 mm (0.75 in.) wide strips, lower the ram, and start the timing.	Duration, min	Accumulative, min
Allow the batch to mix, raising the ram momentarily to sweep down, if necessary.	0	0
	5.0	5.0
Total time		5.0 min

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 From the stock cut enough sample to allow testing of vulcanizing characteristics in accordance with Test Method D 2084. Condition the specimen 45 min to 6 h at $23 \pm 3^\circ\text{C}$ ($73 \pm 5^\circ\text{F}$) before testing.

6.2.2.7 After the sample is cut for cure meter testing, pass the remaining stock endwise through the mill rolls 6 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:

	Duration, Min	Accumulative, Min
6.3.2.1		
Adjust the internal mixer temperature to achieve the discharge conditions outlined in 6.3.2.5. Close the discharge gate, start the rotor at 8.1 rad/s (77 rpm) and raise the ram.	0	0
6.3.2.2		
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
6.3.2.3		
Allow the batch to mix.	.5	4.0
6.3.2.4		
Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	2.0	6.0

6.3.2.5 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.

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

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

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

6.3.3 Final Mix:

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.

	Duration, Min	Accumulative, Min
6.3.3.2		
Charge $\frac{1}{2}$ 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	.5	.5

6.3.3.3