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— Tension[2]

D 2084 Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter[2]

D 3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets[2]

D 3896 Practice for Rubber from Synthetic Sources—Sampling[2]

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries[2]

D 5289 Test Method for Rubber Property – Vulcanization using Rotorless Cure Meters[2]

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*:

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]

[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-cm ^{3} 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 ¾cut from each side.	10	11

[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.

	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 pigments that drop through the mill to the milling stock.		
Add the zinc oxide.	3	14
Make three ¾ 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 stock end-ways through the mill six times.	2	18
	Total time	18 min

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 ± 3°C (73.4 ± 5.4°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 ± 3°C (140 ± 5°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.

	Duration, min	Accumulative, min
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.	0	0
Allow the batch to mix, raising the ram momentarily to sweep down, if necessary.	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± 5°C (104 ± 9°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 ± 3°C (73± 5°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 ± 5°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 ± 3°C (73.4 ± 5.4°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 ± 5°C (104 ± 9°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 ± 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 rpm), and raise the ram.

	Duration, Min	Accumulative, Min
6.3.3.2		
Charge ½ 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		
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
6.3.3.4		

Determine and record the batch mass; if the mass differs by more than 0.5 % of the theoretical mass, discard it.		
6.3.3.5		
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 rolls six times.	2.0	5.0
6.3.3.6		
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

6.3.3.7 Cut enough sample for testing of compound viscosity in accordance with Test Method D 1646 or vulcanizing characteristics in accordance with Test Method D 2084, as described in 7.2, or both, if these are desired. Condition the specimen for 1 to 24 h at 23 ± 3°C (73.4 ± 5.4°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± 5°C (122 ± 9°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 ± 3°C (73.4 ± 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 ± 5 % relative humidity.

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± 2°C (73 ± 3.6°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.

7.2 An alternative to measuring vulcanization characteristics by means of tensile stress measurement on vulcanizates is the measurement of vulcanization characteristics with an Oscillating Disk cure meter in accordance with Test Method D 2084 or a Rotorless Cure Meter in accordance with Test Method D 5289. These methods will not produce equal results.

7.2.1 The recommended standard Oscillating Disk test conditions are: 1.7 Hz oscillation frequency; ±1° amplitude of oscillation, 160°C die temperature, 30-min test time, and no preheating. The recommended test conditions for the Rotorless Cure Meter are: 1.7 Hz oscillation frequency, ±0.5° of arc for torsional shear cure meters and ±0.05mm for linear shear cure meters, 160°C die temperature, 30 min. test time, and no preheating. Tolerances for the listed conditions are included in the specified test methods.

7.2.2 The recommended standard test parameters are: M_L, M_H, t_{sl}, t′50, and t′90.

NOTE 2—It is recommended that M_H be taken as the torque at 30 min. Where the torque plateaus or peaks before 30 min, M_{HF} or M_{HR}, according to the applicable test method, should be used.

8. Precision and Bias

8.1 This precision and bias section has been prepared in accordance with Practice D 4483. Refer to this practice for terminology and other statistical calculation details.

8.2 The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers) used in the particular interlaboratory program as described below. The precision parameters should not be used for acceptance/rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols that include this test method.

8.3 A Type 2 (interlaboratory) precision was evaluated. Both repeatability and reproducibility are short term, a period of a few days separates replicate test results. For Test Method D 2084 a test result is the value, as specified by this test method, obtained on one determination(s) or measurement(s). For Test Method D 412 a test result is the median of three measurements on separate test pieces.

8.4 Two different materials (rubbers BIIR, CIIR) were used in the interlaboratory program, these were tested in six laboratories on two different days for the internal mixer procedure and in five laboratories on two different days for the mill procedure. The results of the precision calculations for repeatability and reproducibility are given in Table 1.

8.5 The precision of this test method may be expressed in the format of the following statements that use an "appropriate value" of *r*, *R*, (*r*) or (*R*), to be used in decisions about test results. The appropriate value is that value of *r* or *R* associated with a mean level in Table 1 closest to the mean level under consideration at any given time for any test and for any material in routine testing operations.

8.6 *Repeatability*— The repeatability *r*, of this test method has been established as the appropriate value tabulated in Table 1. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated *r* (for any given level) must be considered as derived from different or non-identical sample populations.

TABLE 1 Type 2 Precision for MIM and Mill Procedures

NOTE 1—For both MIM and Mill procedures; precision values for MIM in parentheses; mid-point of range used for (*r*) and (*R*) calculations.
NOTE 2— Sr = repeatability standard deviation, in measurement units, r = repeatability, in measurement units, (r) = repeatability, (relative) percent, SR = reproducibility standard deviation, in measurement units, R = reproducibility, in measurement units, and (R) = reproducibility, (relative) percent.

Property	Units	Range of Values (See notes)	Within Laboratories			Between Laboratories		
			Sr	*r*	(*r*)	*SR*	*R*	(*R*)
For Test Method D 2084:								
M_L	dN·m	10.5 to 14.1	(0.50)	(1.42)	(11.5)	(1.68)	(4.25)	(38.6)
			0.21	0.59	4.8	1.02	2.89	(23.5)
M_H	dN·m	20.7 to 26.3	(0.51)	(1.44)	(6.1)	(1.73)	(4.90)	(20.9)
			0.33	0.93	4.0	1.07	3.03	12.9
t_{51}	min	2.3 to 3.9	(0.16)	(0.45)	(14.5)	(0.39)	(1.10)	(35.5)
			0.19	0.54	17.4	0.39	1.10	35.5
t_{90}	min	11.8 to 14.4	(0.62)	(1.75)	(13.4)	(2.29)	(6.48)	(49.5)
			0.38	1.08	8.2	1.72	4.87	37.2
For Test Methods D 412:								
Modulus 300 %,	MPa	6.0 to 7.2	(0.41)	(1.16)	(17.6)	(0.61)	(1.73)	(26.2)
			0.38	1.08	16.4	0.75	2.12	32.1
Tensile strength,	MPa	12.1 to 16.6	(0.57)	(1.60)	(11.1)	(2.15)	(6.08)	(42.2)
			0.62	1.75	12.2	1.58	4.47	31.0
Elongation,	%	477 to 513	(48)	(136)	(27.5)	(41.5)	(118)	(23.8)
			33	93	18.8	21.5	61	123

8.7 *Reproducibility*— The reproducibility *R*, of this test method has been established as the appropriate value tabulated in Table 1. Two single test results obtained in two different laboratories, under normal test method procedures, that differ by more than the tabulated *R* (for any given level) must be considered to have come from different or non-identical sample populations.

8.8 Repeatability and reproducibility expressed as a percentage of the mean level, (*r*) and (*R*), have equivalent application statements as above for *r* and *R*. For the (*r*) and (*R*) statements, the difference in the two single test results is expressed as a percentage of the arithmetic mean of the two test results.

8.9 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (or true) test property value. Reference values do not exist for this test method since the value (of the test property) is exclusively defined by the test method. Bias therefore cannot be determined.

9. Keywords

9.1 BIIR; CIIR; halogenated isobutene; isoprene rubber