This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

Designation: D3958 – 06 (Reapproved 2021)

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

This standard is issued under the fixed designation D3958; 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 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 establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

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 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 D3896.

6. Mixing Procedures

6.1 For general mixing procedures, refer to Practice D3182.

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*—See Table 2.

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.

[1] These test methods are under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials 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.

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TABLE 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.

6.2.1.2 Condition the carbon black in accordance with 5.6 of Practice D3182. This is critical with halogenated IIR when the simple zinc oxide cure is used.

6.2.1.3 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.

6.2.1.4 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.

6.2.1.5 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.

6.2.2 *Method B: Miniature Internal Mixer (MIM) Mix*—See Table 3.

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 5.6 of Practice D3182. This is critical with halogenated butyl rubber when the simple zinc oxide cure is used.

6.2.2.3 After mixing according to Table 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 discharge from the mixer twice through a standard mill maintained at 40 ± 5°C (104 ± 9°F) with a roll separation of 0.5 mm (0.020 in.) once, then twice at a separation of 3 mm (0.12 in.) in order to dissipate heat. Pass the rolled batch endwise through the mill six times with an opening of 0.8 mm (0.31 in.) to enhance the dispersion.

6.2.2.5 Measure and record the batch mass. If it differs from the theoretical value by more than 0.5 %, discard 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 D1646 or D6204, and vulcanization characteristics in accordance with Test Methods D2084 or D5289.

6.2.2.7 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 Procedure:*

6.3.1 For general mixing procedure refer to Method D3182.

6.3.2 *Mixing Cycle-Initial Mix*—See Table 4.

6.3.2.1 After mixing according to Table 4, determine and record the batch mass; if the mass differs by more than 0.5 % of the theoretical mass, discard the batch.

6.3.2.2 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).

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

6.3.3 *Final Mix*—See Table 5.

6.3.3.1 After mixing according to Table 5, 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.

6.3.3.3 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.

7. Preparation and Testing of Vulcanizates

7.1 For tension testing, prepare the test sheets and vulcanize them in accordance with Practice D3182.

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 D412.

7.2 An alternative to measuring vulcanization characteristics by means of tensile stress measurement on vulcanizates is the measurement of vulcanization characteristics in accordance with Test Method D2084 (Oscillating Disk Cure Meter Method) or Test Method D5289 (Rotorless Cure Meter Method). These methods will not produce equal results.

7.2.1 The recommended Test Method D2084 test conditions are 1.67 Hz (100 cpm) oscillation frequency, 1° oscillation amplitude, 160°C die temperature, 40-min test time, and no preheating. The recommended Test Method D5289 test conditions are 1.67 Hz (100 cpm) oscillation frequency, 0.5° oscillation amplitude, 160°C die temperature, 40-min test time, and no preheating. Test condition tolerances are specified by the 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.

TABLE 2 Method A: Mill Mix

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.

	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 ¾ cut from each side.	10	11
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

TABLE 3 Method B: Miniature Internal Mixer Mix

	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

TABLE 4 Mixing Cycle—Initial Mix

	Duration, min	Accumulative, min
Adjust the internal mixer temperature to achieve the discharge conditions outlined below. Close the discharge gate, start the rotor at 8.1 rad/s (77 rpm) and raise the ram.	0	0
Charge ½ the rubber, all of the carbon black and stearic acid, and then the other one half of the rubber. Lower the ram.	0.5 3.0	0.5 3.5
Allow the batch to mix.	0.5	4.0
Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	2.0	6.0
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.		

8. Precision and Bias

8.1 This precision and bias section has been prepared in accordance with Practice D4483. Refer to this practice for terminology and other statistical calculation details.

8.1.1 The recommended standard test parameters are M_L, M_H, t_{sl}, t'50, and t'90.

8.1.2 Alternate test conditions include use of 3° oscillation amplitude for Test Method D2084 and the use of 1° oscillation amplitude for Test Method D5289. When 3° oscillation amplitude is used for Test Method D2084 tests, replace test parameter t_{s1} with t_{s2}.

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

TABLE 5 Final Mix

	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 rpm), and raise the ram.		
Charge ½ the batch with all of the zinc oxide rolled into this portion of the batch before feeding into 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 rolls 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

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 D2084 a test result is the value, as specified by this test method, obtained on one determination(s) or measurement(s). For Test Method D412 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 6.

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 6 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 6. 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.

8.7 *Reproducibility*—The reproducibility *R*, of this test method has been established as the appropriate value tabulated in Table 6. Two single test results obtained in two different

TABLE 6 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 *Sr*	*r*	(*r*)	Between Laboratories *SR*	*R*	(*R*)
For Test Method D2084:								
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 D412:								
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

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

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