Designation: D 3188 – 00

Standard Test Methods for Rubber—Evaluation of IIR (Isobutene-Isoprene Rubber)[1]

This standard is issued under the fixed designation D 3188; 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 specify the standard materials, test formula, mixing procedures, and test methods for the evaluation and production control of non-halogenated isobutene-isoprene rubbers (IIR), commonly known as butyl rubber.

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— Tension[2]

D 1646 Test Method for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)[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 test methods are mainly intended 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 rubber samples in a standard formula.

3.2 These test methods may be used to obtain values for acceptance of rubber.

4. Standard Test Formula

4.1 *Standard Formula:*

Material	NBS or IRM No.	Quantity, Parts by Mass
IIR	...	100.00
Zinc oxide	[A]	3.00
Sulfur	[A]	1.75
Stearic acid	[A]	1.00
Oil furnace black[B]	378	50.00
TMTD[C]	[A]	1.00
Total mass		156.75
Batch factor:		
Mill[D]		2.0
Miniature internal mixer[E]		
Cam Head		0.46
Banbury Head		0.40

[A] Use current IRM/SRM.

[B] The current industry reference black may be used in place of NBS 378, although slightly different results may be obtained.

[C] Tetramethylthiuram disulfide. NBS has discontinued supply of TMTD. A new source of supply material is available as IRM 1 from Forcoven Products Inc., P.O. Box 1536, Humble, TX 77338. A research report can be obtained from ASTM Headquarters. Request RR: D-11-1034.

[D] For mill mixes, weigh the rubber and carbon black to the nearest 1.0 g, the sulfur and accelerators to the nearest 0.02 g, and all other compounding materials to the nearest 0.1 g.

[E] For MIM batches weigh the rubber carbon black to the nearest 0.1 g, the compounding material blend to the nearest 0.01 g, and individual compounding materials, if used, to the nearest 0.001 g. For the MIM procedure, it is recommended that a blend of compounding materials, including black, be prepared to improve accuracy in the weighing of these materials. This material blend is prepared by blending a proportional mass of each material in a dry powder such as a biconical blender or vee blender. A mortar and pestle may be used for blending small quantities.

5. Sample Preparation

5.1 For tests intended for referee purposes obtain and prepare the samples in accordance with Practice D 3896.

6. Mixing Procedures

6.1 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.

6.1.1 *Method A*—Mill mix (6.2)

6.1.2 *Method B*—Miniature Internal Mixer (MIM) Mix (6.3)

6.1.3 *Method C*—Lab Banbury (6.4)

NOTE 1—It is not implied that comparable results will be obtained by these test methods.

---

[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.

Current edition approved May 10, 2000. Published July 2000. Originally published as D 3188 – 73. Last previous edition D 3188 – 95.

[2] *Annual Book of ASTM Standards*, Vol 09.01.

6.2 *Method A—Mill Procedure*:

6.2.1 For general mixing procedures, refer to Practice D 3182. Mix with the mill roll temperature maintained at 50 ± 5°C (122 ± 9°F). The indicated mill openings should be maintained as nearly as possible to provide a standard degree of breakdown for the rubber due to milling. Necessary adjustments may be made to maintain a good working bank at the nip of the rolls.

6.2.2 *Mixing Cycle*:

	Duraation, min	Accumulative, min
Set the mill opening at 0.65 mm (0.025 in.) and band the rubber on the slow roll.	1	1
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
Note—Do not cut the batch while free carbon black is evident in the bank or on the milling surface. Be certain to return to the batch any materials that drop through the mill.		
Add all the other materials.	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 batch end-ways through the mill six times.	2	18

6.2.2.6 Determine and record the batch mass. If it differs from the theoretical value by more than 0.5 %, reject the batch. Remove enough sample from the batch to allow testing of compound viscosity in accordance with Test Method D 1646 or vulcanization characteristics in accordance with Test Method D 2084, 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.

6.2.2.7 If tensile stress is required, sheet off the compound from the mill at a setting to give a finished gage of 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.

6.2.2.8 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.

6.3 *Method B—Miniature Internal Mixer Mix*:

6.3.1 For general mixing procedure, refer to Practice D 3182. Mix with the head temperature of the miniature internal mixer maintained at 60 ± 3°C (140 ± 5°F) and the unloaded rotor speed at 6.3 to 6.6 rad/s (60 to 63 rpm).

6.3.2 Prepare the rubber by passing it through a mill one time with the temperature set at 50 ± 5°C (122 ± 9°F) and an opening of 0.5 mm (0.02 in.) thick. Cut the sheet into strips that are approximately 25 mm (1 in.) wide, if desired.

6.3.3 *Mixing Cycle*:

	Duration, min	Accumulative, min
Charge the mixing chamber with the rubber strips and the blended materials, lower the ram, and start the timer.	0	0
Allow to mix.	1	1
Raise the ram, add carbon black, sweep the orifice, and lower the ram.	1	2
Allow the batch to mix, raising the ram momentarily to sweep down the materials, if necessary.	3	5

6.3.3.5 Turn off the motor, raise the ram, remove the head and discharge the batch. Measure and record the maximum batch temperature if desired.

6.3.3.6 Immediately pass the batch twice through a laboratory mill maintained at 50 ± 5°C (122 ± 9°F) and with the roll separation of 3 mm (0.125 in.).

6.3.3.7 Determine and record the batch mass. If it differs from the theoretical value by more than 0.5 %, reject the batch. Remove enough sample from the batch to allow testing of compound viscosity in accordance with Test Method D 1646 or vulcanization characteristics in accordance with Test Method D 2084, 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.

6.4 *Internal Mixer Procedure*:

6.4.1 For general mixing procedure refer to Method D 3182.

6.4.2 *Mixing Cycle-Initial Mix*:

	Duration, Min	Accumulative, Min
6.4.2.1		
Adjust the internal mixer temperature to achieve the discharge conditions outlined in 6.4.2.5. Close the discharge gate, start the rotor at 8.1 rad/s (77 rpm) and raise the ram.	0	0
6.4.2.2		
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
6.4.2.3		
Allow the batch to mix.	.5	4.0
6.4.2.4		
Raise the ram and clean the mixer throat and the top of the ram. Lower the ram.	2.0	6.0

6.4.2.5 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.

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

6.4.2.7 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).

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

6.4.3 *Final Mix*:

6.4.3.1 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
6.4.3.2 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
6.4.3.3 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
6.4.3.4 Determine and record the batch mass; if the mass differs by more than 0.5 % of the theoretical mass, discard it.		
6.4.3.5 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
6.4.3.6 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

6.4.3.7 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.

6.4.3.8 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.

6.4.3.9 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 stress-strain testing, prepare the test sheets and vulcanize them in accordance with Practice D 3182.

7.1.1 The recommended standard vulcanization time is 40 min at 150°C (302°F).

7.1.2 Condition the cured sheets for 16 to 96 h at a temperature of 23 ± 2°C (73.4 ± 3.6°F) prior to making stress-strain tests.

NOTE 2—Quality control of rubber production may require testing within 1 to 6 h to provide surveillance of the plant operations; however, slightly different results may be obtained.

7.1.3 Prepare test specimens and obtain the tensile stress, tension, and elongation in accordance with Test Methods D 412.

8. Testing for Curing Characteristics using Cure Meters

8.1 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.

8.1.1 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.

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

NOTE 3—Where the effect of surface contamination is not a problem, a ±3° angle of oscillation may be used in order to obtain greater sensitivity. In this case, the parameter t_s2 is to be taken instead of t_{s1}.

8.1.3 The recommended standard test parameters are M_L, M_H(Note 4), t_{s1}, t' 50, and t' 90.

NOTE 4—It is recommended that M_H be taken as the torque value at 40 min.

9. Precision and Bias [3]

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

9.2 The precision results in this precision and bias section give an estimate of the precision of the test method with the materials used in the particular interlaboratory program as described in the following paragraphs. 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.

9.3 A Type 2, Class III interlaboratory precision program was conducted. Materials were tested for M_L, M_H, $t_{s\ 2}$, t'50, and t'90 using an oscillating disc cure meter. Test Method D 2084 was followed. Test conditions were as follows: temperature—160°C; preheat—none; arc—±3°; M_H taken at 40 min; oscillation—1.7 Hz. Both repeatability and reproducibility are short-term. A period of a few days separates test

---

[3] Supporting data are available from ASTM Headquarters. Request RR: D11–1068.

results, which were repeated on three separate days. Four laboratories participated and three materials were used. Therefore, $p = 4$, $q = 3$, and $n = 3$. A test result is the value obtained from one determination.

9.4 The materials used in the test program were isobuteneisoprene rubbers as follows: Polymer A = low Mooney/low unsaturation; Polymer B = high Mooney/high unsaturation; and Polymer C = low Mooney/high unsaturation. Both rubber samples and chemicals necessary for the test recipe were distributed to the participating laboratories.

9.5 The results of the precision calculations for each of the elevated parameters are given in Table 1 with the materials arranged in increasing mean value within each test type.

9.6 The precision of these test methods may be expressed in the format of the following statements that use what is called an appropriate value of *r, R,* (*r*), or (*R*), that is, that value to be used in decisions about test results (obtained with the test method). 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 given material in routine testing operations.

TABLE 1 Precision[A]

Material	Mean Level	Within Laboratories			Between Laboratories		
		s_r	r	(r)	S_R	R	(R)
	M_L:						
A	12.30	0.17	0.47	3.80	0.23	0.64	5.22
C	12.60	0.21	0.58	4.63	0.68	1.92	15.27
B	17.60	0.27	0.76	4.31	0.52	1.47	8.35
Average/Pool[B]	14.20	0.22	0.61	4.33	0.51	1.45	10.18
	M_H:						
A	60.30	0.96	2.71	4.49	2.23	6.32	10.48
B	71.60	0.92	2.61	3.65	1.76	4.97	6.94
C	81.60	1.30	3.68	4.51	3.29	9.32	11.42
Average/Pool[B]	71.17	1.07	3.04	4.27	2.51	7.11	9.98
	t_{s2}:						
B	1.30	0.14	0.40	30.69	0.21	0.59	45.72
A	1.70	0.09	0.25	14.48	0.22	0.63	36.96
C	3.70	0.10	0.28	7.65	0.19	0.52	14.15
Average/Pool[B]	2.20	0.11	0.32	14.37	0.21	0.58	26.53
	$t'50$:						
C	8.20	0.11	0.30	3.62	0.34	0.95	11.56
B	9.20	0.13	0.35	3.91	0.22	0.63	6.89
A	10.60	0.15	0.42	3.98	0.29	0.81	7.66
Average/Pool[B]	9.30	0.13	0.36	3.90	0.29	0.81	8.69
	$t'90$:						
C	24.00	1.42	4.02	16.74	1.99	5.63	23.48
B	27.00	0.40	1.13	4.18	1.19	3.37	12.49
A	28.10	0.74	2.09	7.44	0.74	2.09	7.44
Average/Pool[B]	26.40	0.95	2.70	10.21	1.41	3.98	15.07

[A] This is short term precision with $p = 4$, $q = 3$, and $n = 3$.
s_r = Within laboratory standard deviation,
r = Repeatability in measured units ($s_r \times 2.83$),
(r) = Repeatability in % ((r/mean) × 100),
S_R = Between laboratories standard deviation,
R = Reproducibility in measured units ($S_R \times 2.83$), and
(R) = Reproducibility in % ((R/mean) × 100).

[B] Mean levels are averages; standard deviations are pooled. Units—M_L and M_H are dN·m; t_{s2}, $t'50$, and $t'90$ are minutes.

9.6.1 *Repeatability*— The repeatability, *r*, of these test methods has been established as the appropriate value given in Table 1. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated *r* (expressed in actual test units) must be considered as suspect, that is, having been derived from different or nonidentical sample populations. If this is the case, appropriate corrective action should be taken.

9.6.2 *Repeatability*— The repeatability, (*r*), of this test method has been established as the appropriate value given in Table 1. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated (*r*) (expressed as a percentage of the mean value) must be considered as suspect, that is, having been derived from different or nonidentical sample populations. If this is the case, appropriate corrective action should be taken.

9.6.3 *Reproducibility*— The reproducibility, *R*, of this test method has been established as the appropriate value given in Table 1. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated *R* (expressed in actual test units) must be considered as suspect, that is, having been derived from different or nonidentical sample populations. If this is the case, appropriate corrective action should be taken.

9.6.4 *Reproducibility*— The reproducibility, (*R*), of this test method has been established as the appropriate value given in Table 1. Two single test results, obtained under normal test method procedures, that differ by more than this tabulated (*R*) (expressed as a percentage of the mean value) must be considered as suspect, that is, having been derived from different or nonidentical sample populations. If this is the case, appropriate corrective action should be taken.

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

10. Keywords

10.1 IIR; isobutene–isoprene rubber