



Designation: D 3191 – 02

Standard Test Methods for Carbon Black in SBR (Styrene-Butadiene Rubber)—Recipe and Evaluation Procedures¹

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1. Scope

1.1 These test methods cover the standard materials, test formula, mixing procedure, and test methods for the evaluation and production control of carbon blacks in styrene butadiene rubber (SBR).

1.2 The values stated in SI units are to be regarded as the standard. The values 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 Elastomers—Tension²
- D 1646 Test Method for Rubber —Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)²
- D 1799 Practice for Carbon Black—Sampling Packaged Shipments²
- D 1900 Practice for Carbon Black—Sampling Bulk Shipments²
- 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 3674 Test Method for Carbon Black—Relative Extrusion Mass²
- 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

¹ These test methods are under the jurisdiction of ASTM Committee D24 on Carbon Black and are the direct responsibility of Subcommittee D24.71 on Carbon Black Testing in Rubber.

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

E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens³

3. Significance and Use

3.1 The major portion of carbon black consumed by the rubber industry is used to improve the physical properties, life expectancy, and utility of rubber products. These test methods provide an SBR recipe and directions for evaluating all types of carbon black intended for use in rubber products. Other procedures are available elsewhere in the ASTM standards for the evaluation of carbon black itself.

3.2 These test methods may be used to characterize carbon black in terms of specific properties of the standard compound. These test methods are useful for the quality assurance of carbon black production. They may also be used for the preparation of reference compounds, to confirm the day-to-day reliability of testing operations used in the rubber industry, for the evaluation of experimental compounds, and quality control of production compounds.

4. Standard Test Formula

4.1 Standard Formula:

Material	IRM ^A No.	Quantity, parts by mass
SBR-1500	...	100.00
Zinc oxide ^{B,C}	91	3.00
Sulfur ^{B,C}	31	1.75
Stearic acid ^{B,C}	21	1.00
Carbon black	...	50.00
TBBS ^{C,D}	...	1.00
Total		156.75
Batch factor: ^E		

^A IRM 91 is available from R. E. Carroll, Inc., 1570 North Olden Ave., Trenton, NJ 08638; (800) 257-9365. IRM 21 and IRM 31 are available from Akron Rubber Development Lab, 2887 Gilchrist Road, Akron, OH 44305; (330) 794-6600.

^BFor the MIM procedure, it is recommended that a blend of compounding materials be prepared to improve accuracy of the weighing of these materials. This material blend is prepared by blending a proportional mass of each material in a dry powder blender such as a biconical blender or vee blender. A mortar and pestle may be used for blending small quantities.

^CFor mill mixes, weigh the rubber and carbon black to the nearest 1.0 g, the sulfur and the accelerator to the nearest 0.02 g, and all of the other compounding

³ *Annual Book of ASTM Standards*, Vol 14.04.

materials to the nearest 0.1 g. For MIM mixes, weigh the rubber and material blend to the nearest 0.01 g and individual pigments, if used, to the nearest 0.001 g.

^DTBBS is *N-tert-butyl-w-benzothiazolesulfenamide*.

^EFor the mill mixes, a batch factor should be selected to the nearest 0.5 to give as large a total mass as possible that will not exceed 525.0 g. Calculate all parts to the nearest 0.01 part. For MIM mixes, calculate a batch factor to the nearest 0.01 that will provide a 75% loading of the mixing chamber.

5. Sampling and Sample Preparation

5.1 Samples shall be taken in accordance with Practice D 1799 or Practice D 1900.

5.2 The carbon black shall be conditioned before weighing and mixing by heating in a Type 1B oven, as described in Specification E 145, for 1 h at $125 \pm 3^\circ\text{C}$. The black shall be placed in an open vessel of suitable dimensions so that the depth of black is no more than 10 mm during conditioning. The black conditioned as above shall be stored in a closed moisture-proof container until ready for mixing.

6. Mixing Procedure

6.1 For general mixing procedure refer to Practice D 3182.

6.1.1 Mixing shall be done with the mill roll temperature maintained at $50 \pm 5^\circ\text{C}$. The indicated mill openings are approximate and should be adjusted to maintain a good working bank at the nip of the rolls. The following three mixing procedures are offered:

6.1.1.1 *Test Method A—Mill Mix,*

6.1.1.2 *Test Method B—Internal Mixer, and*

6.1.1.3 *Test Method C—Miniature Internal Mixer.*

Check and record the stock mass. If it differs from the theoretical value by more than 0.5 %, reject the batch. From this stock, cut enough sample to allow testing of compound viscosity in accordance with Test Method D 1646, and curing characteristics in accordance with Test Method D 2084, or both, and extrudability of unvulcanized compounds in accordance with Test Method D 3674, if these are desired.

<https://standards.iteh.ai/catalog/standards/sist/na0e7>

6.2 Mixing Cycle:

6.2.1 *Test Method A—Mill Mix:*

6.2.1.1 Set the mill opening at 1.1 mm (0.045 in.) and band the polymer on the front roll. Make $\frac{3}{4}$ cuts every $\frac{1}{2}$ min from alternate sides.

6.2.1.2 Add the sulfur slowly and evenly across the mill at a uniform rate.

6.2.1.3 Add the stearic acid. Make one $\frac{3}{4}$ cut from each side after the stearic acid has been incorporated.

6.2.1.4 Add the carbon black evenly across the mill at a uniform rate. When one half the black is incorporated, open the mill to 1.4 mm (0.055 in.) and make one $\frac{3}{4}$ cut from each side. Add the remainder of the carbon black. When all the black has been incorporated, open the mill to 1.8 mm (0.070 in.) and make one $\frac{3}{4}$ cut from each side.

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.

6.2.1.5 Add the zinc oxide and TBBS at the 1.8-mm (0.070-in.) setting.

6.2.1.6 Make three $\frac{3}{4}$ cuts from each side and cut the stock from the mill.

6.2.1.7 Set the rolls at 0.8 mm (0.032 in.). Pass the rolled stock endwise through the mill six times.

6.2.1.8 Open the mill to give a minimum stock thickness of 6 mm (0.25 in.) and pass the stock through the rolls four times, folding it back on itself each time.

	Dura- tion, min	Accu- mula- tive, min
6.2.1.1	2.0	2.0
6.2.1.2	2.0	4.0
6.2.1.3	2.0	6.0
6.2.1.4	10.0	16.0
6.2.1.5	3.0	19.0
6.2.1.6	2.0	21.0
6.2.1.7	2.0	23.0
6.2.1.8	1.0	24.0
Total Time		24.0

Total Time 24.0

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

6.2.1.10 To prevent absorption of moisture, condition the sheeted stock for 1 to 8 h at a temperature of $23 \pm 3^\circ\text{C}$ in a closed container after cooling unless the relative humidity is controlled at $35 \pm 5\%$ in accordance with Practice D 3182.

6.2.2 *Test Method B—Internal Mixer:*

6.2.2.1 BR Banbury: Cool water (not over 16°C) rotors at 8.06 rad/s (77 r/min). Start loading when Banbury temperature recorder indicates 32°C .

6.2.2.2 Before mixing the first batch, adjust the internal mixer temperature to achieve the discharge conditions outlined in 6.2.2.6. Close the gate.

	Dura- tion, min	Accu- mula- tive, min
6.2.2.3 Raise ram, add SBR-1500 and zinc oxide, and lower ram.	0.75	0.75
6.2.2.4 Raise ram, add all other ingredients except TBBS, and lower ram.		
6.2.2.5 Raise ram, sweep, lower ram.	1.25	2.0
6.2.2.6 Dump at 3.5 min but not over 71°C .	1.5	3.5
Total Time		3.5

6.2.2.7 Mill; in accordance with Practice D 3182, 6 by 12 mill cold water. (Before using the mill warm up with a batch of rubber. Start mill operations when roll surface temperature is 32°C .)

6.2.2.8 Sheet out on the mill, weigh, and check batch mass. Discard if more than 0.5 % different from theoretical mass.

6.2.2.9 Return to mill, set at 1.8 mm (0.070 in.) between rolls, band on mill, add TBBS, and make five $\frac{3}{4}$ cuts from each side.	2.5	2.5
Total Time		2.5

6.2.2.10 Remove stock from the mill in a sheet and allow to rest 1 h on a flat, dry metal surface.

6.2.2.11 Weigh 650 g, roll, and pass endwise nine times, without banding through the mill set at 0.5 mm (0.020 in.) between rolls. Start with a surface temperature of 32°C .

6.2.2.12 Sheet out stock to a thickness of about 2.2 mm (0.085 in.) and cool on a flat, dry metal surface.

6.2.2.13 To prevent absorption of moisture, condition the sheeted stock for 1 to 8 h at a temperature of $23 \pm 3^\circ\text{C}$ in a closed container after cooling unless the relative humidity is controlled at $35 \pm 5\%$ in accordance with Practice D 3182. Vulcanize and test in accordance with Section 7.

6.2.3 *Test Method C—Miniature Internal Mixer:*

6.2.3.1 Prepare the rubber by passing it through a mill one time with the mill temperature at $50 \pm 5^\circ\text{C}$ and a mill opening at 0.51 mm (0.020 in.).

6.2.3.2 With the head temperature of the miniature internal mixer maintained at $60 \pm 3^\circ\text{C}$ and the unloaded slow rotor speed at 6.3 to 6.6 rad/s (60 to 63 r/min), feed the rubber into the mixing chamber and start the timer as soon as all the rubber is added. Break down the rubber. While the rubber is breaking down, set the powder chute in place.	1.0	1.0
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6.2.3.3 Add the sulfur, zinc oxide, stearic acid, and TBBS followed by the carbon black. Quickly insert the ram in the chute and place a 1-kg mass on the ram.	1.0	2.0
6.2.3.4 When the ram position indicates that the carbon black has been added, remove the chute and sweep the remaining carbon black from the ram and chute cavity into the mixing chamber.	1.0	3.0
6.2.3.5 Allow the compound to mix.	6.0	9.0
Total time	9.0	

6.2.3.6 Turn off the motor, raise the ram, remove the mixing chamber, and unload the batch. Record the batch temperature if desired.

6.2.3.7 With the mill at room temperature, pass the batch through the mill set at 0.80 mm (0.032 in.). Fold it on itself and feed it back through the mill five more times, always keeping the grain in the same direction and folding it on itself each time.

6.2.3.8 Check the batch mass and record. Reject the batch if more than $\pm 0.5\%$ differ from the theoretical mass.

6.2.3.9 For testing of stress-strain, pass the batch through the mill to produce a stock thickness of 2.2 mm (0.085 in.).

6.2.3.10 For testing of curing characteristics in accordance with Test Method D 2084, pass the batch through the mill to produce a minimum stock thickness of 6 mm (0.25 in.).

6.2.3.11 To prevent absorption of moisture, condition the sheeted stock for 1 to 8 h at a temperature of $23 \pm 3^\circ\text{C}$ in a closed container after cooling unless the relative humidity is controlled at $35 \pm 5\%$ in accordance with Practice D 3182. Vulcanize and test in accordance with Section 7.

7. Preparation and Testing of Vulcanizates

7.1 For stress-strain testing, prepare test slabs and vulcanize them in accordance with Practice D 3182.

7.1.1 The recommended standard cure is 50 min at 145°C .

7.1.2 Condition the cured sheets for 1 to 96 h at a temperature of $23 \pm 1^\circ\text{C}$.

7.1.3 Prepare the test specimens in accordance with Practice D 3182, and obtain modulus, tensile, and elongation parameters in accordance with Test Methods D 412. Typically, a test specimen is prepared using the current Industry Reference Black, for example IRB 7, with each set of mixes and the data obtained is reported as a difference from the IRB.

7.2 An alternative to measuring stress-strain properties of vulcanizates is the cure-meter measurement of vulcanization parameters with an Oscillating Disk cure meter in accordance with Test Method D 2084 or a Rotorless Cure Meter Test in accordance with Test Method D 5289.

7.2.1 The recommended standard Oscillating Disk test conditions are: 1.7 Hz oscillation frequency, $\pm 1^\circ$ 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, $\pm 0.5^\circ$ of arc for torsional shear cure meters, ± 0.05 mm 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_{s1} , $t'_c(50)$, and $t'_c(90)$.

8. Precision and Bias⁴

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

8.2 *Precision*—The precision results in this precision and bias section give an estimate of the precision of this test method with the materials (rubbers, carbon blacks, etc.) used in the particular interlaboratory program described in 8.3 through 8.4.2.3. The precision parameters should not be used for acceptance or rejection testing of any group of materials without documentation that they are applicable to those particular materials and the specific testing protocols of the test method.

8.3 *Mill Mix—Test Method A*—A Type 2 interlaboratory precision program was conducted in 1990. Both repeatability and reproducibility represent short-term testing conditions. Seven laboratories tested four carbon blacks (SRBs A-4, B-4, D-4, and F-4) once on each of two different days. Test results were obtained in accordance with Test Methods D 412 and are expressed as differences from IRB 6. A test result is the value obtained from a single determination. Acceptable difference values were not measured (see Table 1).

8.3.1 Repeatability:

8.3.1.1 *Tensile Stress at 300 % Elongation*—The pooled repeatability of Test Methods D 3191 Method A (using Test Methods D 412 Method A) tensile stress at 300 % elongation has been established as 0.87 MPa (125 psi). Two single test results (or determinations) that differ by more than 0.87 MPa (125 psi) must be considered suspect, that is, to have come from different sample populations. Such a decision dictates that some appropriate action be taken.

8.3.1.2 *Tensile Strength*—The pooled repeatability of Test Methods D 3191 Method A (using Test Methods D 412 Method A) tensile strength has been established as 2.02 MPa (293 psi). Two single test results (or determinations) that differ by more than 2.02 MPa (293 psi) must be considered suspect, that is, to have come from different sample populations. Such a decision dictates that some appropriate action be taken.

8.3.1.3 *Ultimate Elongation*—The pooled repeatability of Test Methods D 3191 Method A (using Test Methods D 412 Method A) ultimate elongation has been established as 47.7 %. Two single test results (or determinations) that differ by more than 47.7 % must be considered suspect, that is, to have come from different sample populations. Such a decision dictates that some appropriate action be taken.

8.3.2 Reproducibility:

8.3.2.1 *Tensile Stress at 300 % Elongation*—The pooled reproducibility of Test Methods D 3191 Method A (using Test Methods D 412 Method A) tensile stress at 300 % elongation has been established as 1.85 MPa (268 psi). Two single test results (or determinations) produced in separate laboratories that differ by more than 1.85 MPa (268 psi) must be considered suspect, that is, that they represent different sample populations. Such a decision dictates that appropriate investigative or technical or commercial actions, or both, be taken.

⁴ Supporting data for this precision evaluation are available from ASTM headquarters. Request Research Report: D24-1030.