~~Designation:D3191–06ε1~~ Designation: D 3191 – 09

Standard Test Methods for Carbon Black in SBR (Styrene-Butadiene Rubber)—Recipe and Evaluation Procedures[1]

This standard is issued under the fixed designation D 3191; 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.

~~ε[1] NOTE—Paragraphs 6.2.2.1 and 6.2.2.3 revised editorially in July 2008.~~

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:*[2]

D 412 Test Methods for Vulcanized Rubber and Thermoplastic ElastomersTension
D 1646 Test Methods for RubberViscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)
D 1799 Practice for Carbon BlackSampling Packaged Shipments
D 1900 Practice for Carbon BlackSampling Bulk Shipments
D 2084 Test Method for Rubber PropertyVulcanization Using Oscillating Disk Cure Meter
D 3182 Practice for RubberMaterials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets
D 3674 Test Method for Carbon Black Relative Extrucion Mass [3]
D 4483 Practice for Evaluating Precision for Test Method Standards in the Rubber and Carbon Black Manufacturing Industries
D 5289 Test Method for Rubber PropertyVulcanization Using Rotorless Cure Meters
E 145 Specification for Gravity-Convection and Forced-Ventilation Ovens

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.

---

[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.

~~Current edition approved July 1, 2006. Published July 2006. Originally approved in 1973. Last previous edition approved in 2002 as D3191–02.~~

Current edition approved May 1, 2009. Published May 2009. Originally approved in 1973. Last previous edition approved in 2006 as D 3191 – 06ε1.

[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.

[3] Withdrawn.

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.

[B] For 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.

[C] For 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 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.

[D] TBBS is *N-tert*-butyl-w-benzothiazolesulfenamide.

[E] For 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 ± 3°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 ± 5°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:

(1) Test Method A—Mill Mix,

(2) Test Method B—Internal Mixer, and

(3) Test Method C—Miniature Internal Mixer.

6.1.1.1 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 Methods 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.

6.2 *Mixing Cycle:*

6.2.1 *Test Method A—Mill Mix:*

	Duration, min	Accumulative, min
Set the mill opening at 1.1 mm (0.045 in.) and band the polymer on the front roll. Make ¾ cuts every ½ min from alternate sides.	2.0	2.0
Add the sulfur slowly and evenly across the mill at a uniform rate.	2.0	4.0
Add the stearic acid. Make one ¾ cut from each side after the stearic acid has been incorporated.	2.0	6.0
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 ¾ 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 ¾ cut from each side.	10.0	16.0
Note—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 and TBBS at the 1.8-mm (0.070-in.) setting.	3.0	19.0
Make three ¾ cuts from each side and cut the stock from the mill.	2.0	21.0
Set the rolls at 0.8 mm (0.032 in.). Pass the rolled stock endwise through the mill six times.	2.0	23.0
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.	1.0	24.0
Total Time	24.0	

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

6.2.1.2 To prevent absorption of moisture, condition the sheeted stock for 1 to 8 h at a temperature of 23 ± 3°C in a closed container after cooling unless the relative humidity is controlled at 35 ± 5 % in accordance with Practice D 3182.

6.2.2 *Test Method B—Internal Mixer:*

~~6.2.2.1BR~~6.2.2.1 BR Banbury: Water cooled (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 the table below. Close the gate.

	Duration, min	Accumulative, min
Raise ram, add SBR-1500 and zinc oxide, and lower ram.	0.75	0.75
Raise ram, add all other ingredients except TBBS, and lower ram.		

Raise ram, sweep, lower ram.	1.25	2.0
Dump at 3.5 min but not over 71°C.	1.5	3.5
Total Time	3.5	

~~6.2.2.3Mill~~6.2.2.3 Mill in accordance with Practice D 3182, 6-by-12 in. mill with water cooling. (Before using the mill warm up with a batch of rubber. Start mill operations when roll surface temperature is 32°C.)

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

	Duration, min	Accumulative, min
Return to mill, set at 1.8 mm (0.070 in.) between rolls, band on mill, add TBBS, and make five ¾ cuts from each side.	2.5	2.5
Total Time	2.5	

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

6.2.2.6 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.

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

~~6.2.2.8To prevent absorption of moisture, condition the sheeted stock for 1 to 8 h at a temperature of 23 ± 3°C in a closed container after cooling unless the relative humidity is controlled at 35 ± 5% in accordance with Practice D3182~~

6.2.2.8 Unless otherwise specified, condition the sheeted compound for 1 to 24 h at 23 ± 3°C (73.4 ± 5.4°F) at 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 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 ± 5°C and a mill opening at 0.51 mm (0.020 in.).

	Duration, min	Accumulative, min
With the head temperature of the miniature internal mixer maintained at 60 ± 3°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
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
When the ram positon 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
Allow the compound to mix.	6.0	9.0
Total time	9.0	

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

6.2.3.3 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.

6.2.3.4 Check the batch mass and record. Reject the batch if more than ±0.5 % differ from the theoretical mass.

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

6.2.3.6 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.).

6.2.3.7 To prevent absorption of moisture, condition the sheeted stock for 1 to 8 h at a temperature of 23 ± 3°C in a closed container after cooling unless the relative humidity is controlled at 35 ± 5 % in accordance with Practice D 3182. Vulcanize and test in accordance with Section 7.