



Designation: D8582 – 24

Standard Practice for Recovered Carbon Black—Reference Compound¹

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1. Scope

1.1 This practice covers the standard raw materials, formulation, and mixing procedure for the evaluation and quality control of recovered carbon black in a standardized rubber compound.

1.2 *Units*—The values stated in SI units are to be regarded as the standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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

D3182 Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets

D3191 Test Methods for Carbon Black in SBR (Styrene-Butadiene Rubber)—Recipe and Evaluation Procedures

D5900 Specification for Physical and Chemical Properties of Industry Reference Materials (IRM)

3. Summary of Practice

3.1 This practice shall be used for in-rubber testing of recovered carbon black. A reference compound is required for

¹ This practice is under the jurisdiction of ASTM Committee D36 on Recovered Carbon Black (rCB) and is the direct responsibility of Subcommittee D36.70 on rCB Testing in Rubber.

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² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at www.astm.org/contact. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

quality control of production, for research and development purposes, and for comparison of different raw materials.

3.2 This practice is not suitable for the determination of the rCB performance in a specific product application, for example, the rCB performance in a tire compound.

3.3 Comparisons between different types of filler, for example, carbon black versus recovered carbon black or different types of rCB, or both, must be performed using the same ASTM standard, and therefore, the same reference compound.

4. Significance and Use

4.1 This standard practice provides an SBR Reference Compound for evaluating different types of recovered carbon black intended for use in rubber products. Test methods are available in the ASTM Book of Standards, Vol. 09.01, for the evaluation of recovered carbon black itself.

4.2 This practice is useful for the quality assurance of rCB production. It may also be used for the preparation of reference compounds for compounding experiments, to confirm the day-to-day reliability of testing operations used in the rubber industry, for the evaluation of experimental compounds, and for quality control of production compounds.

5. Standard Test Formula

5.1 The standard reference compound has been formulated to give enhanced discrimination between samples in dynamic tests as well as enhanced dispersion compared to the carbon black reference formulation in Test Methods D3191.

5.2 Standard Formula:

Material	Quantity, parts by mass ^B
SBR 1502	100.00
Zinc Oxide	3.00
Stearic Acid	1.00
Recovered Carbon Black	70.00
Sulfur	1.75
TBBS ^A	1.00
Total:	176.75

^A N-tert-butyl-w-benzothiazolesulfenamide.

^B Weigh rubber and rCB to the nearest 1 g, sulfur and accelerator to the nearest 0.02 g, and all other compounding materials to the nearest 0.1 g.

5.3 The use of Industry Reference Materials (IRM) as specified in Specification D5900 is recommended for standard

rubber test formulas. The current list of Industry Reference Materials for rubber can be found on the webpage of the ASTM Committee D11.

6. Mixing Procedures

6.1 *Procedure A*—The mixing procedure for a standard internal mixer according to Practice D3182 is described in Table 1 (times in seconds). The procedure involves two different steps: master batch and final mix.

6.2 *Procedure B*:

6.2.1 This mixing procedure uses a standard internal mixer for the master batch (1st step) and a standard mill for the final mix (2nd step). The procedure is described in Table 2 (times in seconds).

6.2.2 Continue to the 2nd step of the mixing process with a standard 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. Total time: 150 s.

6.2.3 Use a standard mill in accordance with Practice D3182 with water cooling. If mills of other sizes are used, adjustments to batch masses and mixing cycles may be required to obtain equivalent results.

6.2.4 Before using the mill, warm up with a batch of rubber. Start mill operations when roll surface temperature is 32°C.

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

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

6.2.7 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.

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

6.3 If internal mixers of other sizes are used, adjustments of batch masses, ram pressure, rotor speeds or mixing times will be required to obtain equivalent results. The use of other mixer sizes shall be recorded with the reported data. For general information regarding mixing procedures see Practice D3182.

7. Keywords

7.1 formulation; mixing procedure; recipe; recovered carbon black, rCB

TABLE 1 Mixing Procedure A

1st step	Duration, s	Accumulation, s
Fill factor 0.62 to 0.72, rotation speed 6.3 to 8.1 rad/s (60 to 77 rpm)		
Lower Ram pressure 0.5 to 0.6 MPa	0	0
Raise ram, add SBR-1500 and zinc oxide, and lower ram	45	45
Raise ram, add rCB and stearic acid, and lower ram	75	120
Mix and adjust speed to target temperature 120 to 140 °C	90	210
Continue mixing at target temperature		
Discharge and record dump temperature		
Store compound for 2 to 12 h @ RT before 2nd step		
2nd step		
Fill factor 0.58 to 0.68, rotation speed 5.2 to 6.8 rad/s (50 to 65 rpm)		
Lower Ram pressure 0.5 to 0.6 MPa	0	0
Raise ram, add 1st mixing step, and lower ram	20	20
Raise ram, add sulfur and TBBS, and lower ram	25	45
Mix and adjust speed to target temperature 90 to 110 °C	100	145
Discharge and record dump temperature	5	150
Store compound for 12 to 24 h @ RT before curing		