



Standard Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets¹

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This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This practice provides a listing of reference compounding materials required to prepare the rubber test compounds listed in succeeding methods and contains procedures for weighing. It also specifies the mixing equipment, general mixing procedures, vulcanization equipment and procedures.

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.* For a specific warning statement, see 5.5.

2. Referenced Documents

2.1 *ASTM Standards:*²

D88 Test Method for Saybolt Viscosity

D1646 Test Methods for Rubber—Viscosity, Stress Relaxation, and Pre-Vulcanization Characteristics (Mooney Viscometer)

¹ This practice is under the jurisdiction of ASTM Committee D11 on Rubber and is the direct responsibility of Subcommittee D11.20 on Compounding Materials and Procedures.

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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.

D2084 Test Method for Rubber Property—Vulcanization Using Oscillating Disk Cure Meter

D2161 Practice for Conversion of Kinematic Viscosity to Saybolt Universal Viscosity or to Saybolt Furol Viscosity

D2226 Classification for Various Types of Petroleum Oils for Rubber Compounding Use

D2501 Test Method for Calculation of Viscosity-Gravity Constant (VGC) of Petroleum Oils

D4678 Practice for Rubber—Preparation, Testing, Acceptance, Documentation, and Use of Reference Materials

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

E145 Specification for Gravity-Convection and Forced-Ventilation Ovens

3. Significance and Use

3.1 This practice shall be used for specific procedures used in preparing rubber compounds for quality control of production, for research and development purposes, and for comparison of different materials.

4. Standard Materials

4.1 *Standard Reference Materials:*

4.1.1 The materials required for standard rubber test formulas shall be National Institute of Standards and Technology (NIST) Reference Materials or materials that are known to have properties similar to these standard materials. However, in case of dispute, the following actual standard materials from the NIST of the United States shall be used:

4.1.2 An Industry Reference Material (IRM) is a standard reference from a designated supplier and has been certified in accordance with Practice **D4678**.^{3,4}

NIST Standard Reference Material or IRM	Standard Reference Material No.	Industry Reference Material No.
Zinc oxide	370	
Sulfur	371	
Stearic acid	372	
Benzothiazyl disulfide		2
Tetramethylthiuram disulfide		1
Magnesium oxide	376	
Oil furnace carbon black—HAF	378	
Gas furnace carbon black—SRF	382	
Mercaptobenzothiazole	383	
<i>N-tert-butyl-2-benzothiazolesulfenamide</i>	384	
SBR-1500	386	
Natural rubber	201	

4.2 Other standard or industry reference materials are as follows: Industry Reference Black Current Lot in use at time of testing ASTM Oil Type 103 (defined by Classification **D2226**).⁵

5. Weighing of Materials

5.1 The standard batch mass (in grams) for the laboratory mill shall be three times the formula mass in parts per hundred grams of rubber, unless otherwise specified.

5.2 The batch mass (in grams) for an internal mixer shall be the nominal capacity ($1170 \pm 40 \text{ cm}^3$) multiplied by the density of the rubber compound.

5.3 The batch mass (in grams) for a miniature internal mixer (MIM) shall be 75 % of the nominal capacity ($85 \pm 1 \text{ cm}^3$) multiplied by the density of the rubber compound.

5.4 The rubber and carbon black shall be weighed to within a tolerance of $\pm 1 \text{ g}$ for mill and internal mixer compounds and with a tolerance of $\pm 0.1 \text{ g}$ for miniature internal mixer compounds. All other materials shall be weighed with a $\pm 0.1\text{-g}$ accuracy or less for mill and internal mixer compounds and with $\pm 0.01\text{-g}$ accuracy for the miniature internal mixer compounds.

5.5 Compounding materials other than rubber, carbon black, and oil may be added to miniature internal mixer batches more precisely and with greater ease if they are previously blended

³ NIST has discontinued supply of SRM 373, 374, and 385. A substitute material is available as IRM 2, 1, and 201. The sole source of supply of IRM 1 and 2 known to the committee at this time is Forcoven Products, Inc., P.O. Box 1556, Humble, TX 77338. The sole source of supply of IRM 201 known to the committee at this time is Akron Chemical Co., 255 Fountain St., Akron, OH 44304. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend. Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1034.

⁴ Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D11-1026.

⁵ The sole source of supply of a lot of oil conforming to the basic description in Classification **D2226** and, more specifically, to the values listed below known to the committee at this time is Sun Refining and Marketing Co., Process Materials Group, 10 Penn Center, 1801 Market St., Philadelphia, PA 19103 (available in 1 and 5-gal quantities): Kinematic Viscosity (Test Method **D88**) and (Practice **D2161**) $16.8 \pm 1.2 \text{ mm}^2/\text{s}$ at 100°F, Viscosity-Gravity Constant (Test Method **D2501**) 0.889 ± 0.002 . If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

in proportion to the mass required by the recipe. Such blend may be made in a mortar and pestle, by mixing for 10 min in a biconical blender with intensifier bar turning, or by mixing in a blender⁶ for five 3-s periods and scraping the inside of the mixer to dislodge materials stuck to the sides after each 3-s mix. (**Warning**—If mixed longer than 3 s, the stearic acid may melt and prevent good dispersion.)

5.6 Unless otherwise specified, carbon black shall be conditioned by heating for 1 h at $125 \pm 3^\circ\text{C}$ ($257 \pm 5^\circ\text{F}$) in a 1 B oven or equivalent, in accordance with Specification **E145**.

5.6.1 Place the carbon black in an open vessel of suitable dimensions so that the depth of the black is no more than 10 mm (0.4 in.) during conditioning. Store the conditioned carbon black in a closed moisture-proof container until cool and then use for weighing and mixing.

6. Equipment for Mixing

6.1 Standard Mill:

6.1.1 The standard mill shall have rolls between 150 and 155 mm (5.9 and 6.1 in.) in diameter. The mill shall be equipped with retaining guides, with a distance between the guides at the nip of 250 to 280 mm (10 to 11 in.).

NOTE 1—If mills of other sizes are used, adjustments to batch masses and mixing cycles may be required to obtain equivalent results.

6.1.2 The speed of the slow roll shall be $0.4 \pm 0.50 \text{ rad/s}$ ($24 \pm 0.5 \text{ rpm}$) and the ratio between slow and fast roll shall be 1:1.4. Other ratios may be used, but modifications in mixing procedure may be required to obtain equivalent results. The use of other than a standard mill shall be recorded with the reported data.

6.1.3 Means shall be provided for controlling the mill roll temperatures to the specified temperature $\pm 5^\circ\text{C}$ ($\pm 9^\circ\text{F}$).

6.1.4 The clearance between rolls shall be adjustable from 0.2 to 8.0 mm (0.008 to 0.31 in.) as a minimum range of adjustment. Roll clearance shall be determined by means of two lead strips $10 \pm 3 \text{ mm}$ ($0.4 \pm 0.1 \text{ in.}$) wide, at least 50 mm (2 in.) long, and 0.25 to 0.50 mm (0.01 to 0.02 in.) thicker than the roll clearance to be measured. The lead strips shall be inserted, one at each end of the rolls approximately 25 mm (1 in.) from the guides, while a piece of compounded rubber, with Mooney viscosity in excess of 50 ML 1 + 4 at 100°C (212°F), approximately 75 by 75 by 6 mm (3 by 3 by 0.25 in.) is passing through the center portion of the rolls. The rolls shall be at the temperature specified for mixing. After the lead strips have passed through the rolls, measure the thickness of the strips to the nearest 0.02 mm (0.001 in.). Tolerance on a roll clearance shall be $\pm 10 \%$ or 0.05 mm (0.002 in.), whichever is larger.

6.2 *Standard Internal Mixer*—The standard internal mixer shall have a chamber of $1575 \pm 50 \text{ cm}^3$ volume and two rotors with approximately 400-cm³ displacement volume, resulting in $1170 \pm 40\text{-cm}^3$ loading capacity. The slow rotor speed shall be

⁶ The sole source of supply of the apparatus known to the committee at this time is Waring Products, Inc. at www.waringproducts.com. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,¹ which you may attend.

8.16 rad/s (77 rpm) and the gear ratio shall be 1:1.125. The rotor wing tip to side clearance shall be $2.4 + 0.3, - 0.1$ mm ($0.094 + 0.010, - 0.005$ in.). The mixer shall be equipped with a thermocouple for measuring and recording batch mixing temperatures. The thermocouple shall be installed through the end frame and shall protrude into the mixing chamber 25 ± 2.5 mm (1 ± 0.1 in.) measured along the top side of the thermocouple probe. A ram that is 56 ± 3 mm by 140 ± 8 mm (2.2 ± 0.1 in. by 5.5 ± 0.3 in.) shall exert a force of 1.27 ± 0.06 kN (285 ± 14 lbf) on the batch in the chamber. The sides shall be hinged to swing open, made of cast stainless steel and jacketed for controlling temperature by means of a circulating liquid or steam. The end frames shall be of ductile iron that has a 0.20 ± 0.02 -mm (0.008 ± 0.001 -in.) thick chrome plating on the working surfaces. Rotors are of stainless steel, nitrided, drilled, and equipped with rotary unions for controlling the rotor temperature by means of a circulating liquid or steam.

NOTE 2—If internal mixers of other sizes are used, adjustments of batch masses and rotor speeds or mixing cycles will be required to obtain equivalent results.

6.3 Standard Miniature Internal Mixer (MIM):

6.3.1 The standard miniature internal mixer shall be equipped with a stainless steel mixer head having a bowl of 120-cm³ volume and stainless steel cam-style mixer rotors (removable or fixed) of 34 to 35-cm³ displacement, thus resulting in a 85 ± 1 -cm³ volume. The recommended loading is 75 % (64 cm³). The fast or driving rotor (left) shall turn at $6.28 + 3.14$ rad/s ($60 + 3, - 0$ rpm) and the gear ratio (drive to driven) shall be 1.5:1. The mixer shall be equipped with a thermocouple installed through the ridge in the bowl for measuring and recording the batch temperatures. The mixing chamber shall be closed during the mixing cycle by means of a lever or ram. The head and the backplate shall be maintained at the required temperature either electrically or by means of a thermal liquid medium.

NOTE 3—If miniature internal mixers equipped with Banbury-style mixer head and rotors or heads of other sizes are used, adjustments of batch masses, rotor speeds, or mixing cycles will be required to obtain equivalent results.

6.3.2 The miniature internal mixer may be equipped with a torque-measuring instrument and recorder, which are not essential for the mixing operation. If used, it must be calibrated occasionally and after each overhaul of the miniature internal mixer using the manufacturer's instructions.

7. General Mixing Procedures

7.1 Mill Mixing Procedure:

7.1.1 Mix compounds with the rubber banded on the slow roll, unless otherwise specified.

7.1.2 Measure the temperature of the surfaces of the rolls at a point approximately equidistant from the ends during the mixing operation either continuously on a recorder or with a manual device having an accuracy of at least $\pm 1^\circ\text{C}$ ($\pm 2^\circ\text{F}$), frequently enough to maintain the desired temperature. The batch may be removed momentarily from the mill to measure the surface temperature of the slow roll.

7.1.3 Whenever $\frac{3}{4}$ cuts are specified, cut the batch $\frac{3}{4}$ of the distance across the roll and hold the knife at this position until

the bank just disappears. Make successive $\frac{3}{4}$ cuts from alternate ends of the roll, allowing 20 s between each cut unless otherwise specified.

7.1.4 Do not cut any batch while free materials are evident in the bank or on the milling surface.

7.1.5 Add carbon black evenly across the mill at a uniform rate. Add all other dry materials slowly and evenly in prescribed times. Oil, if required, may be added alternately with carbon black. Carefully collect materials falling through the nip from the tray and return to the mix.

7.1.6 Conclude the mixing cycle by passing the rolled batch endwise through the mill six times with an opening of 0.8 mm (0.031 in.), to improve the dispersion.

7.1.7 Pass the batch four times through the mill at a setting of 6 mm (.25 in.), folding it back on itself each time.

7.1.8 Weigh the batch and record the mass. If required, cut samples from the batch to allow testing of compound viscosity and processability in accordance with Test Methods **D1646** or Test Method **D6204**, and vulcanization characteristics in accordance with Test Methods **D2084** or **D5289**. If tensile stress strain tests are required, sheet out the remainder of the batch to 2.2 to 2.4 mm (0.087 to 0.094 in.) thickness after shrinkage has taken place.

7.2 Internal Mixer Mixing Procedure:

7.2.1 The compound is usually prepared in two stages: the first one always in the internal mixer and the second either in the internal mixer or on the standard mill.

7.2.2 The initial internal mixer temperature will be either indicated or be such that the required discharge temperature is reached.

7.2.3 The rotor speed shall be 8.16 rad/s (77 rpm), unless otherwise specified.

7.2.4 Cut the rubber into pieces suitable for fast feeding.

7.2.5 Prepare the mix according to specified instructions regarding the order and time of material addition.

7.2.6 Consolidate the discharge on a standard mill, weigh, and allow to cool on a flat metal surface before proceeding with the second-stage mix.

7.2.7 If the second stage mix is prepared in the internal mixer, cut the batch from stage 1 into strips for easier feeding, and follow by the addition of the materials according to the specific instructions. The batch should then be discharged at the prescribed time or temperature. If the second-stage mix is prepared on the standard mill, add materials in prescribed order and time. The batch size may be reduced to better accommodate the mill and to result in better dispersion of compounding materials.

7.2.8 Conclude the mixing by passing the rolled batch endwise through the standard mill six times with an opening of 0.8 mm (0.031 in.), to improve the dispersion.

7.2.9 Pass the batch four times through the mill at a setting of 6 mm (0.24 in.), folding it back on itself each time.

7.2.10 Weigh the batch and record the mass. If required, cut samples from the batch to allow testing of compound viscosity and processability in accordance with Test Methods **D1646** or Test Method **D6204**, and vulcanization characteristics in accordance with Test Methods **D2084** or **D5289**. If tensile stress