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

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 appro-
priate safety and health practices and determine the applica-
bility of regulatory limitations prior to use. For specific
precaution statements, see 5.5.

2. Referenced Documents
2.1 ASTM Standards:
D 88 Test Method for Saybolt Viscosity2
D 1646 Test Method for Rubber—Viscosity, Stress Relax-
ation, and Pre-Vulcanization Characteristics (Mooney Vis-
cometer)3
D 2084 Test Method for Rubber Property—Vulcanization
Using Oscillating Disk Cure Meter4
D 2161 Practice for Conversion of Kinematic Viscosity to
Saybolt Universal Viscosity or to Saybolt Furol Viscosity4
D 2226 Classification for Various Types of Petroleum Oils
for Rubber Compounding Use3
D 2501 Test Method for Calculation of Viscosity-Gravity
Constant (VGC) of Petroleum Oils4
D 4678 Practice for Rubber—Preparation, Testing, Accep-
tance, Documentation, and Use of Reference Materials5
E 145 Specification for Gravity-Convection and Forced-
Ventilation Ovens5

3. Significance and Use
3.1 This practice shall be used for specific procedures used
in preparing rubber compounds for quality control of produc-
tion, for research and development purposes, and for compar-
ison of different materials.

4. Standard Materials
4.1 Standard Reference Materials:
4.1.1 The materials required for standard rubber test formu-
las 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 D 4678.6,7

<table>
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<tbody>
<tr>
<td>Zinc oxide</td>
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<tr>
<td>Sulfur</td>
<td>371</td>
<td></td>
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<tr>
<td>Stearic acid</td>
<td>372</td>
<td></td>
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<tr>
<td>Benzothiazyl disulfide 8</td>
<td>2</td>
<td></td>
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<tr>
<td>Tetramethylthiuram disulfide 6,7</td>
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<td>1</td>
</tr>
<tr>
<td>Magnesium oxide</td>
<td>376</td>
<td></td>
</tr>
<tr>
<td>Oil furnace carbon black—HAF</td>
<td>378</td>
<td></td>
</tr>
</tbody>
</table>

6 NIST has discontinued supply of SRM 373, 374, and 385. A substitute material
is available as IRM 2, 1, and 201 respectively from Forcoven Products, Inc., P.O.
Box 1556, Humble, TX 77338 for IRM 1 and 2. IRM 201 is available from Akron
Chemical Co., 255 Fountain St., Akron, OH 44304. Request RR: D11-1034,
available from ASTM International Headquarters.
7 Supporting data are available from ASTM International Headquarters. Request
RR: D11-1026.
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 ± 40 cm³) 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 ± 1 cm³) multiplied by the density of the rubber compound.

5.4 The rubber and carbon black shall be weighed to within a tolerance of ±1 g for mill and internal mixer compounds and with a tolerance of ±0.1 g for miniature internal mixer compounds. All other materials shall be weighed with a ±0.1-g accuracy or less for mill and internal mixer compounds and with ±0.01-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 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 blender9 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 ± 3°C (257 ± 5°F) in a B oven or equivalent, in accordance with Specification E 145.

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 ± 0.50 rad/s (24 ± 0.5 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 ±5°C (±9°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 ± 3 mm (0.4 ± 0.1 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 ±0.1% 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 ± 50 cm³ volume and two rotors with approximately 400 cm³ displacement volume, resulting in 1170 ± 40 cm³ loading capacity. The slow rotor speed shall be 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

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9 A Waring Blender has been used in this practice. Results cannot be guaranteed using another brand.
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 ±1°C (±2°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 3/4 cuts are specified, cut the batch 3/4 of the distance across the roll and hold the knife at this position until the bank just disappears. Make successive 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, remove the test specimens for Mooney viscosity in accordance with Test Method D 1646 or cure meter in accordance with Test Method D 2084, or both, and sheet out the remainder 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 (.24 in.), folding it back on itself each time.

7.2.10 After weighing and removal of test specimens, sheet out the batch to 2.2 to 2.4-mm (0.087 to 0.094-in.) thickness after shrinkage has taken place.

7.3 Miniature Internal Mixer Procedure:

7.3.1 Maintain the mixer head temperature for at least 5 min before mixing.

7.3.2 The unloaded rotor speed shall be 1.0 + 0.5, – 0 rev/s (60 + 3, – 0 rpm), unless otherwise specified. It should be frequently checked if a variable speed model is used.

7.3.3 Prepare the compound according to instructions specified for the rubber.

7.3.4 Immediately pass the discharge from the mixer twice through a standard mill maintained at specified temperature with roll separation of 0.5 mm (0.020 in.) once, then twice at a separation of 3 mm (0.12 in.), in order to dissipate the heat, and weigh.

7.3.5 After the removal of a curemeter specimen, if a compound viscosity or tension specimen, or both are required, pass the batch endwise through the mill six times with an opening of 0.8 mm (0.031 in.) to enhance the dispersion.

7.3.6 After removing the compound viscosity specimen, if a tension specimen is required, pass the batch four times through a standard mill at specified temperature. Fold it lengthwise after each pass and pass always in the same direction to obtain the effect of mill direction. The roll opening should be such that it will produce a 2.2 to 2.4-mm (0.087 to 0.094-in.) thick sheet after shrinkage.

8. Preparation of Standard Vulcanized Sheets

8.1 Preparation of Sheets:

8.1.1 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.

8.1.2 Place the sheeted compound on a flat, dry, clean metal surface and cut pieces that are 4.5 ± 1.5 mm (0.18 ± 0.06 in.) shorter in width and length than the corresponding dimensions of the mold cavity. Mark the direction of the milling on each piece.

8.1.3 The mass of a 150 by 150-mm (6 by 6-in.) sheet or a 150 by 75-mm (6 by 3-in.) sheet to be vulcanized in the molds described in 8.2.2 shall be as shown below:

<table>
<thead>
<tr>
<th>Density of Compound</th>
<th>Mass of Unvulcanized Sheet, g</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>150 by 150 mm (6 by 6 in.)</td>
</tr>
<tr>
<td>0.94</td>
<td>52 ± 3</td>
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<tr>
<td>1.30</td>
<td>70</td>
</tr>
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</table>

8.1.4 A film of suitable material, such as a nonlubricated aluminum foil 0.1 mm (0.004 in.) thick, may be placed above and below the sheet in the mold to prevent contamination with materials remaining in the mold from previous cures. The mass of the unvulcanized sheet shall be reduced to compensate for the thickness of the foil.

8.2 Vulcanization Equipment:

8.2.1 Press—The press shall be capable of exerting a pressure of not less than 3.5 MPa (500 psi) on the total cross-sectional area of the cavities of the mold during the entire period of vulcanization. It shall have heated platens of sufficient size so that no portion of the rubber will be nearer than 75 mm (3 in.) to the edge of the platen during vulcanization. The platen shall preferably be made of rolled steel, machined for steam heating or for electrical elements for heating electrically. When steam heat is used, place the steam outlet slightly below the steam chamber to allow steam to flow between the instant the pressure is applied fully and the instant the pressure is released. Hold the mold under a minimum pressure of 3.5 MPa (500 psi) on the cavity areas during vulcanization. As soon as the press is opened, remove the vulcanized sheets from the mold and cool in water (room temperature or lower) or on a metal surface (for items used for items used for electrical elements for heating electrically). Instead of a separate mold and cover, the cavities may be cut directly into the platen of the press. Unless required, do not use a mold lubricant on the mold surfaces. When a mold lubricant is required, use only a residual-type lubricant, which does not affect the vulcanized sheet, and remove the excess lubricant by vulcanizing and discarding at least one set of sheets. A silicone-type lubricant or mild soap solution has been found satisfactory.

8.3 Vulcanization Procedure:

8.3.1 Bring the mold to curing temperature within ±0.5°C (1°F) in the closed press, and hold at this temperature for at least 20 min before the unvulcanized pieces are inserted. Verify the temperature of the mold by means of a thermocouple or other suitable temperature measuring device inserted in one of the overflow grooves and in intimate contact with the mold.

8.3.2 Open the press, insert the unvulcanized pieces into the mold, and close the press in the minimum time possible. When the mold is removed from the press to insert the pieces, take precautions to prevent excessive cooling of the mold by contact with cool metal surfaces or by exposure to air drafts.

8.3.3 Consider the time of vulcanization to be the period between the instant the pressure is applied fully and the instant the pressure is released. Hold the mold under a minimum pressure of 3.5 MPa (500 psi) on the cavity areas during vulcanization. As soon as the press is opened, remove the vulcanized sheets from the mold and cool in water (room temperature or lower) or on a metal surface (for items used for electrical measurements) for 10 to 15 min. Designate in the report the type of cooling used.

8.3.4 Condition vulcanizates of compounds at a temperature of 23 ± 2°C (73 ± 3.6°F) for at least 16 h (Note 4) and for not more than 96 h before preparing and testing, unless otherwise specified.

NOTE 4—Quality control of rubber production may require testing within 1 to 6 h to provide close surveillance of the plant operation; however, slightly different results may be obtained.
FIG. 1 Design of Four-Cavity Mold

Mill four corners 3.2 mm (1/8") for prying mold apart

Mill 0.5 mm (0.020") deep below depth of cavity

Cavities to be 1.95 ± 0.005 mm (0.077 ± 0.002 in.) deep

Cover plate to be 12.5 mm (0.50") thick.

FIG. 1 Design of Four-Cavity Mold
FIG. 2 Cutoff Bar Type of Test of Slab Mold

Note 1—All other dimensions as in Fig. 1.

FIG. 3 Design for Four Cavity Small Tensile Sheet Mold

D 3182 – 89 (2001)
FIG. 4 Design of Eight Cavity Mold, for Small Tensile Sheets Made by Modifying the Mold Shown in Fig. 1