Designation: D3182 – 21a

Standard Practice for Rubber—Materials, Equipment, and Procedures for Mixing Standard Compounds and Preparing Standard Vulcanized Sheets[1]

This standard is issued under the fixed designation D3182; 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.

This standard has been approved for use by agencies of the U.S. 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For a specific warning statement, see 5.4.

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]

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

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

D5289 Test Method for Rubber Property—Vulcanization Using Rotorless Cure Meters

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

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 Industry Reference Materials (IRM) as specified in Specification D5900. The current list of Industry Reference Materials for rubber can be found on the webpage of the Committee D11 under the section Additional Information (www.astm.org/COMMITTEE/D11).

4.1.2 In case of dispute in the United States of America, reference materials from the National Institute of Standards and Technology (NIST) shall be used.

4.2 Other standard or industry reference materials are as follows: Industry Reference Black, Current Lot in use at time of testing.

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 multiplied by the density of the rubber compound and multiplied by the batch factor.

[1] This practice is under the jurisdiction of ASTM Committee D11 on Rubber and Rubber-like Materials 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.

5.2.1 The batch mass (in grams) for a standard internal mixer (SIM) shall be the nominal capacity (1570 ± 40 cm^3; standard two wing rotors) multiplied by the density of the rubber compound and multiplied by the batch factor of 0.65 – 0.75. The batch factor depends on rubber viscosity. It is on the high end for low viscous polymers and on the lower end for high viscous polymers.

5.2.2 The batch mass (in grams) for a miniature internal mixer (MIM) shall be the nominal capacity multiplied by the density of the rubber compound and multiplied by the batch factor of 0.65 – 0.75.

5.2.2.1 Nominal capacity for cam blades 84 ± 4 cm^3.

5.2.2.2 Nominal capacity for Banbury blades 74 ± 4 cm^3.

5.2.3 The batch mass (in grams) for an internal intermeshing or tangential mixer shall be the nominal capacity multiplied by the density of the rubber compound and multiplied by the batch factor of 0.65 – 0.75.

5.2.3.1 Nominal capacity for intermeshing blades is 318 ± 10 cm^3.

5.2.3.2 Nominal capacity for tangential cam blades 440 ±10 cm^3.

5.2.3.3 Nominal capacity for tangential Banbury blades 380 ± 10 cm^3.

5.3 The rubber and carbon black shall be weighed 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.

5.4 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 blender 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.)

5.5 Unless otherwise specified, carbon black shall be conditioned by heating for 1 h at 125 ± 3°C (257 ± 5°F) in a 1 B oven or equivalent, in accordance with Specification E145.

5.5.1 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 100 mm and 155 mm (4 in. and 6.1 in.) in diameter. The mill shall be equipped with retaining guides, with a distance between the guides at the nip of 180 mm to 380 mm (7.25 in. to 15 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 1.68 ± 0.05 rad/s (16 ± 0.5 rpm) and the ratio between slow and fast roll shall be 1:1.4 (fast roll speed 2.36 rad/s (22.5 ± 0.5 rpm)).

NOTE 2—If other ratios are used, 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 ±2°C (±3.6°F).

6.1.4 The clearance between rolls shall be adjustable max. up to 8.0 mm (0.31 in.). The roller clearance shall be adjusted with a tolerance of ±0.5 mm (±0.02 in.). The clearance shall be measured electronically. The rolls shall be at the temperature specified for mixing.

NOTE 3—If other than electronical gap measurement is used, the tolerance of ±0.5 mm (±0.02 in.) must be maintained.

6.2 *Standard Internal Mixer (SIM)*—The standard internal mixer shall have a chamber of 1570 ± 50 cm^3 volume and two rotors with approximately 400 cm^3 displacement volume, resulting in 1170 ± 40 cm^3 loading capacity. The slow rotor speed shall be 8.06 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 + 0.012, – 0.004 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 × 140 ± 8 mm (2.2 ± 0.1 × 5.5 ± 0.3 in.) shall exert a force of 1.27 ± 0.06 kN (285 ± 13 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 4—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. The use of other sizes, batch mass and rotor speed shall be recorded with the reported data.

6.3 *Miniature Internal Mixer (MIM)*—The standard miniature internal mixer shall be equipped with a stainless steel mixer head having a bowl of 120 ± 3 cm^3 volume and stainless steel rotors. The recommended loading is 70 to 75 %. 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 backplate, mixing chamber part and the front plate shall be maintained at the required temperature either electrically or by means of a thermal liquid medium.

6.3.1 The standard miniature internal mixer shall be equipped with the gear ratio (drive to driven – left to right blade) 1.5:1 [alternatively 1:1.5]. The driving rotor (left) shall turn at 6.28 + 0.314 rad/s (60 +3, –0 rpm) [alternatively for 1:1.5 – 4.19 +0.314 rad/s (40 +3, –0 rpm)].

6.3.1.1 The miniature internal mixer may be equipped with stainless steel cam-style mixer rotors of 33 to 35 cm^3 displacement, thus resulting in a 84 ± 4 cm^3 volume.

6.3.1.2 The miniature internal mixer may be equipped with stainless steel Banbury-style mixer rotors of 43 to 45 cm^3 displacement, thus resulting in a 74 ± 4 cm^3 volume.

6.3.1.3 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.

NOTE 5—If miniature internal mixers equipped with rotors or heads of other sizes or geometry are used, adjustments of batch masses, rotor speeds, or mixing cycles will be required to obtain equivalent results.

6.4 *Internal Intermeshing Medium Mixer (IIMM)*—The internal intermeshing medium mixer shall be equipped with a stainless-steel mixer head having a bowl in a range of 862 to 898 cm^3 volume and stainless-steel mixer rotors (removable or fixed). The recommended loading is 60 to 70 %. The mixer shall be equipped with a thermocouple, for example, installed in the sidewall or through the ridge in the bowl for measuring and recording the batch temperature. The mixing chamber shall be closed during the mixing cycle by means of a pneumatic loading system, lever or ram. The backplate, mixing chamber part and the front plate shall be maintained at the required temperature either electrically or by means of a thermal liquid medium. The mixing chamber shall be divided horizontally in the middle so that the parts can be turned for easy discharge and chamber cleaning.

6.4.1 The internal intermeshing medium mixer has two rotors with approximately 500 to 580 cm^3 displacement volume, resulting in 318 to 355 cm^3 loading capacity. The speed of the mixer shall be 1.047 ± 0.052 up to 14.13 ± 0.314 rad/s (10 ± 0.5 to 135 ± 3 rpm) and the gear ratio (drive to driven) shall be 1:1.

6.4.2 The internal intermeshing medium mixer may be equipped with a drive current or a torque measuring instrument and recorder, which are not essential for the mixing operation. It must be calibrated during installation and after each overhaul of the internal intermeshing medium mixer using the manufacturer's instructions.

NOTE 6—If internal intermeshing medium mixers with other rotors or heads of other sizes are used, adjustments of batch masses, rotor speeds, or mixing cycles will be required to obtain equivalent results.

6.5 *Internal Tangential Prep Mixer (ITPM)*—The internal tangential prep mixer shall be equipped with a stainless-steel mixer head having a bowl of 683 ± 5 cm^3 volume and stainless-steel mixer rotors (removable or fixed). The recommended loading is 70 to 75 %. 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 pneumatic loading system, lever or ram. The backplate, mixing chamber part and the front plate shall be maintained at the required temperature either electrically or by means of a thermal liquid medium. The mixing chamber shall be horizontally divided into the upper and lower part to be turned upwards or downwards for easy discharge and chamber cleaning.

6.5.1 The gear ratio (drive to driven – left to right blade) shall be 1:1.5 [1:1.11 for Banbury blades]. The driving rotor (left) shall turn at 6.28 + 0.314 rad/s (60 +3, –0 rpm)

6.5.2 The internal tangential prep mixer may be equipped with stainless steel cam-style mixer rotors of 243 ± 5 cm^3 displacement, thus resulting in a 440 ± 10 cm^3 volume.

6.5.3 The internal tangential prep mixer may be equipped with stainless steel Banbury-style mixer rotors of 299 ± 8 cm^3 displacement, thus resulting in a 380 ± 10 cm^3 volume.

6.5.4 The internal tangential prep 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 internal tangential prep mixer using the manufacturer's instructions.

NOTE 7—If special internal tangential prep mixers equipped with other rotors or heads of other sizes are used, adjustments of batch masses, rotor speeds, or mixing cycles will be required to obtain equivalent results.

6.6 *Internal Tangential Medium Mixer (ITMM)*—The internal tangential medium mixer shall be equipped with a stainless-steel mixer head having a bowl of 683 ± 5 cm^3 volume and stainless-steel mixer rotors (removable or fixed) The recommended loading is 70 to 75 % 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 pneumatic loading system, lever or ram. The backplate, mixing chamber part and the front plate shall be maintained at the required temperature either electrically or by means of a thermal liquid medium.

6.6.1 The gear ratio (drive to driven – left to right blade) shall be 1.5:1 [1:1.11 for Banbury blades]. The driving rotor (left) shall turn at 6.28 + 0.314 rad/s (60 +3, –0 rpm) [alternatively for 1:1.11 – 5.56 + 0.314 rad/s (54 rpm +3, –0 rpm)].

6.6.2 The internal tangential medium mixer may be equipped with stainless steel cam-style mixer rotors of 243 ± 5 cm^3 displacement, thus resulting in a 440 ± 10 cm^3 volume.

6.6.3 The internal tangential medium mixer may be equipped with stainless steel Banbury-style mixer rotors of 299 ± 8 cm^3 displacement, thus resulting in a 380 ± 10 cm^3 volume.

6.6.4 The internal tangential medium 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 internal tangential medium mixer using the manufacturer's instructions.

NOTE 8—If internal tangential medium mixers equipped with other rotors or heads of other sizes or geometry are used, adjustments of batch masses, rotor speeds, or mixing cycles will be required to obtain equivalent results.

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