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Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 2393 was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This second edition cancels and replaces the first edition (ISO 2393:1973), which has been technically revised.

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Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures

1 Scope

This International Standard specifies the equipment and procedures for the preparation, mixing and vulcanization of test mixes, as specified in the rubber evaluation procedures.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 37:1994, *Rubber, vulcanized or thermoplastic — Determination of tensile stress-strain properties*.

ISO 289-1:1994, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity*.

ISO 471:—¹⁾, *Rubber — Times, temperatures and humidities for conditioning and testing*.

ISO 3417:1991, *Rubber — Measurement of vulcanization characteristics with the oscillating disc curemeter*.

3 Definitions

For the purposes of this International Standard, the following definitions apply.

3.1 formulation batch mass: The aggregate mass, in grams, of all the constituents in a formulation, with the rubber or oil-extended rubber polymer being taken as 100 g, or as specified in the appropriate rubber evaluation procedure.

3.2 batch mass: The mass of test mix prepared in one mixing operation.

3.3 total free volume: The volume of the mixing chamber with the rotors in place.

3.4 nominal mixer capacity: The proportion of the total free volume which is used in the mixing process; a value of 0,75 times the total free volume has been found suitable for mixers with tangential rotors.

4 Compounding ingredients

The compounding ingredients required for the various standard test formulations shall be in accordance with national or international standards as specified in the appropriate rubber evaluation procedure.

1) To be published. (Revision of ISO 471:1983 and ISO 1826:1981)

5 Preparation of materials

5.1 Batch masses

5.1.1 The standard batch mass for the laboratory mill, in grams, shall be four times the formulation batch mass unless otherwise stated in the appropriate rubber evaluation procedure.

NOTE 1 Smaller batch masses are used in some countries. These may not give identical results.

5.1.2 The batch mass for the internal mixer, in grams, shall be equal to the nominal mixer capacity, in cubic centimetres, multiplied by the compound density.

5.1.3 The batch mass for the miniature internal mixer, in grams, shall be equal to the nominal mixer capacity, in cubic centimetres, multiplied by the compound density.

5.2 Weighing tolerances

5.2.1 Rubber and carbon black shall be weighed to the nearest 1 g, oil to the nearest 1 g or $\pm 1\%$ whichever is the more accurate, vulcanizing agents and accelerators to the nearest 0,02 g and zinc oxide and stearic acid to the nearest 0,1 g. All other ingredients shall be weighed to an accuracy of $\pm 1\%$.

5.2.2 For miniature internal mixer mixes, the rubber and carbon black shall be weighed to the nearest 0,1 g, oil to the nearest 0,1 g or $\pm 1\%$ whichever is the more accurate, sulfur and accelerators to the nearest 0,002 g and zinc oxide and stearic acid to the nearest 0,01 g. All other ingredients shall be weighed to an accuracy of $\pm 1\%$.

5.3 Carbon black conditioning

5.3.1 Unless otherwise specified, carbon black shall be conditioned, before weighing, by heating in an oven at a temperature of $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for 2 h. The black shall be placed in an open vessel of suitable dimensions so that the depth of the black is no more than 10 mm during conditioning. The black, conditioned as above, shall be stored in a closed moisture-proof container until it is required for mixing.

Alternatively, carbon black may be conditioned by heating in an oven at $125\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$ for 1 h. Carbon black conditioned in this manner may not give the same results as that conditioned at $105\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$.

The conditioning temperature used shall be recorded in the test report.

6 Mixing equipment

6.1 Mixing mill

The characteristics of a standard laboratory mill are as follows:

Roll diameter (OD), mm	150 to 155
Roll length (between guides), mm	250 to 280
Speed of front (slow) roll, rpm	24 ± 1
Roll-speed ratio (preferably)	1:1,4
Clearance between rolls (adjustable), mm	0,2 to 8,0
Temperature-control tolerance, $^{\circ}\text{C}$	± 5 (unless otherwise specified)

WARNING — The mill should be equipped with suitable safety devices to protect against accidents, in accordance with national safety regulations.

NOTES

2 If mills of other dimensions are used, adjustments to batch masses and mixing cycles may be required to obtain comparable results.

3 If the roll-speed ratio is other than 1:1,4, modifications may be necessary to the mixing procedure to obtain comparable results.

The roll clearance shall be determined by means of two lead strips $10\text{ mm} \pm 3\text{ mm}$ in width, at least 50 mm long and 0,25 mm to 0,50 mm thicker than the roll clearance to be measured. The lead strips shall be inserted, one at each end of the rolls, approximately 25 mm from the guides, while a piece of compounded rubber with a Mooney viscosity, determined in accordance with ISO 289-1, greater than 50 ML (1 + 4) at $100\text{ }^{\circ}\text{C}$ and measuring approximately $75\text{ mm} \times 75\text{ mm} \times 6\text{ mm}$ is passing through the centre portion of the nip. The rolls shall be at the temperature specified for mixing. After passing between the rolls, the thickness of the lead strips shall be measured at three separate positions with a micrometer to an accuracy of $\pm 0,01\text{ mm}$. The tolerance on roll clearance shall be $\pm 10\%$ or 0,05 mm, whichever is the larger.

The mill rolls shall have provision for circulation of heating or cooling media.

6.2 Internal mixer

6.2.1 Internal mixers may be divided into two basic types: those with tangential rotors and those with interlocking rotors. In the tangential type, the high shear strain and shear stress regions occur between the rotor tips and the chamber wall. The rotors also run at different speeds which aids the pumping and distributive action of the mixer.

In the interlocking type, the rotors run at the same speed, but the design of the rotor-nogs and the interlocking action produce friction between the rotors. The high shear strain and shear stress region occurs between the rotors.

6.2.2 This International Standard describes three types of laboratory internal mixer. Type A₁ and type A₂ are of the tangential type and type B is of the interlocking type. Internal mixers other than those specified may be used. As a rule, different types of internal mixer do not give the same properties in the final mix. Modification of mixing technique may lead to comparable results. For referee purposes in particular, these adjustments shall be defined and agreed on by the interested parties.

WARNING — Internal mixers should be equipped with a suitable exhaust system and suitable safety devices to prevent accidents, in accordance with national safety regulations.

6.2.3 The dimensions of the three types of laboratory internal mixer are shown in table 1.

6.2.4 All internal mixers shall be fitted with a system to measure and indicate and/or record the temperature of the mix during the mixing operation to within 1 °C.

NOTE 4 The actual mix temperature usually exceeds the indicated temperatures by an amount dependent on the mixing conditions used and the location of the measuring probe.

Table 1 — Types of laboratory internal mixer

Mixer characteristics	Type A ₁ (Tangential, i.e. non-interlocking, rotors)	Type A ₂ (Tangential, i.e. non-interlocking, rotors)	Type B (Interlocking rotors)
Nominal mixing capacity (cm ³)	1 170 ± 40	2 000	1 000
Rotor speed (fast rotor) (rpm)	77 ± 10 110 ± 10	40 ± 10	55
Rotor friction ratio	1,125:1	1,2:1	1:1
Rotor clearance (mm)			
new	2,38 ± 0,13	4,0 ± 1,0	2,45 to 2,50
worn	3,70		5,0
Power (kW/rpm)	0,13 (fast rotor)		0,227
Ram pressure on the compound (MPa)	0,5 to 0,8 (or as specified in the appropriate standard)	0,5 to 0,8 (or as specified in the appropriate standard)	0,3 (or as specified in the appropriate standard)
NOTE — Type A ₁ is commonly used.			

6.2.5 All internal mixers shall be fitted with a timer to indicate the mixing time to ± 5 s.

6.2.6 All internal mixers shall be fitted with a system to indicate or record electrical power demand or torque.

6.2.7 All internal mixers shall be fitted with an efficient heating and cooling system to control the surface temperature of the rotors and the mixing-chamber walls.

6.2.8 All internal mixers shall be closed during the mixing cycle with a ram to contain the mix in the mixing chamber.

6.2.9 When clearances exceed the values given in table 1, an overhaul is deemed necessary since mixing quality may be adversely affected. This increase in rotor clearance may be equated to an approximately 10 % increase in nominal mixer capacity.

6.2.10 A mill as described in 6.1 shall be provided for consolidating mixes.

6.3 Miniature internal mixer

The dimensions of the preferred miniature internal mixer are as follows:

Type	Non-interlocking rotors
Nominal mixer capacity (cm ³)	64 ± 1
Rotor speed (rpm)	60 $\begin{smallmatrix} +3 \\ 0 \end{smallmatrix}$ (fast rotor)
Rotor friction ratio	1,5:1

NOTE 5 The miniature internal mixer can only provide sufficient compound for curemeter testing and for a vulcanized sheet measuring 150 mm × 75 mm × 2 mm approximately.

6.3.1 The miniature internal mixer shall be fitted with a system to measure and indicate or record the temperature of the mix during the mixing operation to within 1 °C.

NOTE 6 The actual mix temperature usually exceeds the indicated temperature by an amount dependent on the mixing conditions used.

6.3.2 A timer shall be used to indicate the mixing time to ± 5 s.

6.3.3 The miniature internal mixer shall be fitted with a system to indicate or record electrical power demand or torque.

6.3.4 The miniature internal mixer shall be fitted with an efficient heating and cooling system to control the temperature of the mixing-chamber walls.

6.3.5 The miniature internal mixer shall be closed during the mixing cycle with a ram or lever to contain the mix in the mixing chamber.

7 Mixing procedures

7.1 Mill mixing procedure

7.1.1 Batches shall be mixed with the rubber banded on the front roll, unless otherwise specified in the appropriate International Standard.

7.1.2 The temperature at the middle of the surface of the rolls shall be measured during the mixing procedure, either continuously on a recorder, or with a manual device (having an accuracy of ± 1 °C or better) frequently enough to be sure that the desired temperature is being maintained. The batch may be removed momentarily from the mill to enable the surface temperature of the front roll to be measured.

7.1.3 Whenever 3/4 cuts are specified, the batch shall be cut 3/4 of the distance across the roll and the knife held in this position until the bank just disappears.

7.1.4 The compounding ingredients shall be introduced along the whole roll length. The batch shall not be cut while free powder is evident on the rolling bank or on the milling surface. Compounding ingredients falling through the nip shall be carefully collected and returned to the batch.

7.1.5 Whenever 3/4 cuts each way are specified, successive 3/4 cuts shall be made from alternate directions, allowing 20 s between successive cuts unless otherwise specified in the appropriate International Standard.

7.1.6 The mass of the mixed batch shall not differ from the specified mass of rubber and all other ingredients by more than + 0,5 % or – 1,5 %.

7.1.7 The mixed batch shall be cooled to laboratory temperature on a flat, clean, dry, metal surface. The cooled batches shall be wrapped in aluminium foil or other suitable material to prevent contamination by other compounds. Alternatively, the mixed batch may be cooled in water, but different results may be obtained.

7.1.8 A report shall be prepared for each batch mixed, indicating

- a) the friction ratio (roll-speed ratio) and roll speeds;
- b) the distance between the guides;
- c) the maximum and minimum roll temperatures;
- d) the temperature used for conditioning the carbon black;
- e) the method of cooling the mixed batch.

7.2 Internal-mixer mixing procedure

7.2.1 The mixing technique used shall be as stated in the appropriate International Standard for the rubber concerned. If no standard is available, the procedure shall be as agreed between the interested parties.

7.2.2 For each batch mixed, the internal-mixer conditions shall be the same during the preparation of a series of identical mixes. At the beginning of each series of test mixes, a machine-conditioning batch shall be mixed using the same formulation as in the mixes under test. This also acts as a machine-cleaning batch. The machine shall be allowed to cool down to a specified temperature between the end of one test batch and the start of the next. Temperature-control