INTERNATIONAL STANDARD

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

Mélanges d'essais à base de caoutchouc — Mélangeage, préparation et vulcanisation — Appareillage et mode opératoire

Reference number
ISO 2393:1994(E)

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 ± 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 ± 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 ± 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 ± 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 °C ± 5 °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 °C ± 3 °C for 1 h. Carbon black conditioned in this manner may not give the same results as that conditioned at 105 °C ± 5 °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, °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 mm ± 3 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 °C and measuring approximately 75 mm × 75 mm × 6 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 ± 0,01 mm. The tolerance on roll clearance shall be ± 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 A_1 and type A_2 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	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 $^{+3}_{0}$ (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

conditions for the machine shall not be altered during the mixing of a series of test batches.

NOTE 7 For the best results, all comparative test mixes should be mixed in the same internal mixer.

7.2.3 Material to be mixed shall be reduced in size to pieces that can be fed easily and rapidly to the internal mixer.

7.2.4 The discharged batch shall be consolidated on a standard laboratory mill in the manner specified in the appropriate International Standard and allowed to cool to one of the standard temperatures specified in ISO 471 (23 °C ± 2 °C or 27 °C ± 2 °C) on a flat, clean, dry, metal surface.

7.2.5 The mass of the mixed batch shall not differ from the total mass of the ingredients by more than + 0,5 % or − 1,5 %.

Some rubbers and compounding ingredients are known to contain small amounts of volatiles which may be lost at the temperatures of mixing, with the result that the above limit may not be met. In such cases, the difference shall be reported. This paragraph also applies to 7.2.8 and 7.3.4.

7.2.6 Rest the batch for at least 30 min, or until it reaches standard temperature, before proceeding with the final mixing stage. The maximum time between mixing stages shall be 24 h.

7.2.7 If the final-stage mix is to be prepared in the internal mixer, cut the batch from stage 1 into strips for easier feeding and add the remaining ingredients in accordance with the instructions in the relevant International Standard. If the final stage is to be prepared on the mill, add the ingredients in accordance with the instructions in the relevant International Standard. Unless otherwise stated, the batch size shall be reduced to four times the formulation batch mass.

7.2.8 When the internal mixer is used for the final stage, the discharged batch shall be consolidated as specified in the appropriate International Standard on a mill as specified in 6.1.

The final mass of the mixed batch shall not differ from the total mass of the ingredients by more than + 0,5 % or − 1,5 %.

7.2.9 Unless otherwise stated in the appropriate standard, after removal of a curemeter test piece and (if required) a compound viscosity test piece, pass the batch four times through the mill at a roll temperature of 50 °C ± 5 °C. Fold the batch lengthwise after each pass, and pass always in the same direction to obtain a grain effect. The mill opening shall be such as to give a sheet between 2,1 mm and 2,5 mm thick after shrinkage, suitable for the preparation of vulcanized sheets for dumb-bell test pieces. If vulcanized discs for ring test pieces are to be prepared, open the mill so that a sheet between 4,1 mm and 4,5 mm thick is produced.

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

a) the starting temperature;

b) the mixing time;

c) the rotor speed;

d) the ram pressure;

e) the temperature of the mix at discharge;

f) the type of mixer used;

g) any allowable mass loss outside the limits given in 7.2.5 and 7.2.8;

h) the temperature used for conditioning the carbon black.

For mixes where both initial and final stages were carried out in the internal mixer, a report shall be issued for each of the two stages.

7.3 Miniature internal mixer mixing procedure

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

7.3.2 The rotor speed shall be (1,0 $^{+0,05}_{0}$) rev/s, i.e. (60 $^{+3}_{0}$) rev/min, unless otherwise specified. It shall be frequently checked if a variable-speed model is used.

7.3.3 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.

7.3.4 Immediately pass the discharged mix from the mixer twice through a standard mill maintained at the specified temperature, preferably with the rolls running at the same speed and with a roll separation of 0,5 mm, then twice at a separation of 3 mm in order to dissipate the heat, and weigh. The mass of the

mixed batch shall not differ from the total mass of all the materials by more than 0,5 %.

Some rubbers and compounding ingredients are known to contain small amounts of volatiles which may be lost at the temperatures of mixing, with the result that the above limit may not be met. In such cases, the difference shall be reported.

8 Preparation of standard vulcanized sheets for dumb-bell test pieces

8.1 Conditioning of batches

8.1.1 Batches shall be conditioned for between 2 h and 24 h at one of the standard temperatures specified in ISO 471, preferably in a closed container to prevent absorption of moisture from the air or in a room in which the relative humidity is controlled at (35 ± 5) %.

8.1.2 The sheeted batch shall be placed on a flat, clean, dry, metal surface, and the blanks shall be cut to the corresponding dimensions of the mould cavity. The direction of the grain of the rubber shall be marked on each piece. The blanks shall be within + 3 to 0 g of the mass given in table 2 when they are vulcanized in the mould specified in 8.2.2.

Remilling shall be avoided if possible. Where remilling is necessary, the procedure given in 7.2.9 shall be used.

Table 2 — Mass of blank

Density Mg/m^3	Mass g
0,94	47
0,96	48
0,98	49
1,00	50
1,02	51
1,04	52
1,06	53
1,08	54
1,10	55
1,12	56
1,14	57
1,16	58
1,18	59
1,20	60
1,22	61
1,24	62
1,26	63
1,28	64
1,30	65

8.2 Vulcanization equipment

8.2.1 Press

The press shall be capable of exerting a pressure of not less than 3,5 MPa on the cavity areas of the mould during the entire period of vulcanization. It shall have heated platens of sufficient size that no portion of the rubber will be nearer than 30 mm to the edge of the platen during vulcanization. The platens should preferably be made of rolled steel, machined for electric, steam or thermofluid heating.

When steam heating is used, each platen shall be individually supplied. A self-bleeding trap or small vent shall be placed in the exit steam line to allow steam to flow continuously through the platens. If chamber-type platens are used, the steam outlet shall be placed slightly below the steam chamber so that good drainage is ensured.

Conduction of heat from the hot platens to the press cross-head shall be reduced as much as practicable by means of a steel grid between them or by other means. Platens shall be suitably shielded from draughts.

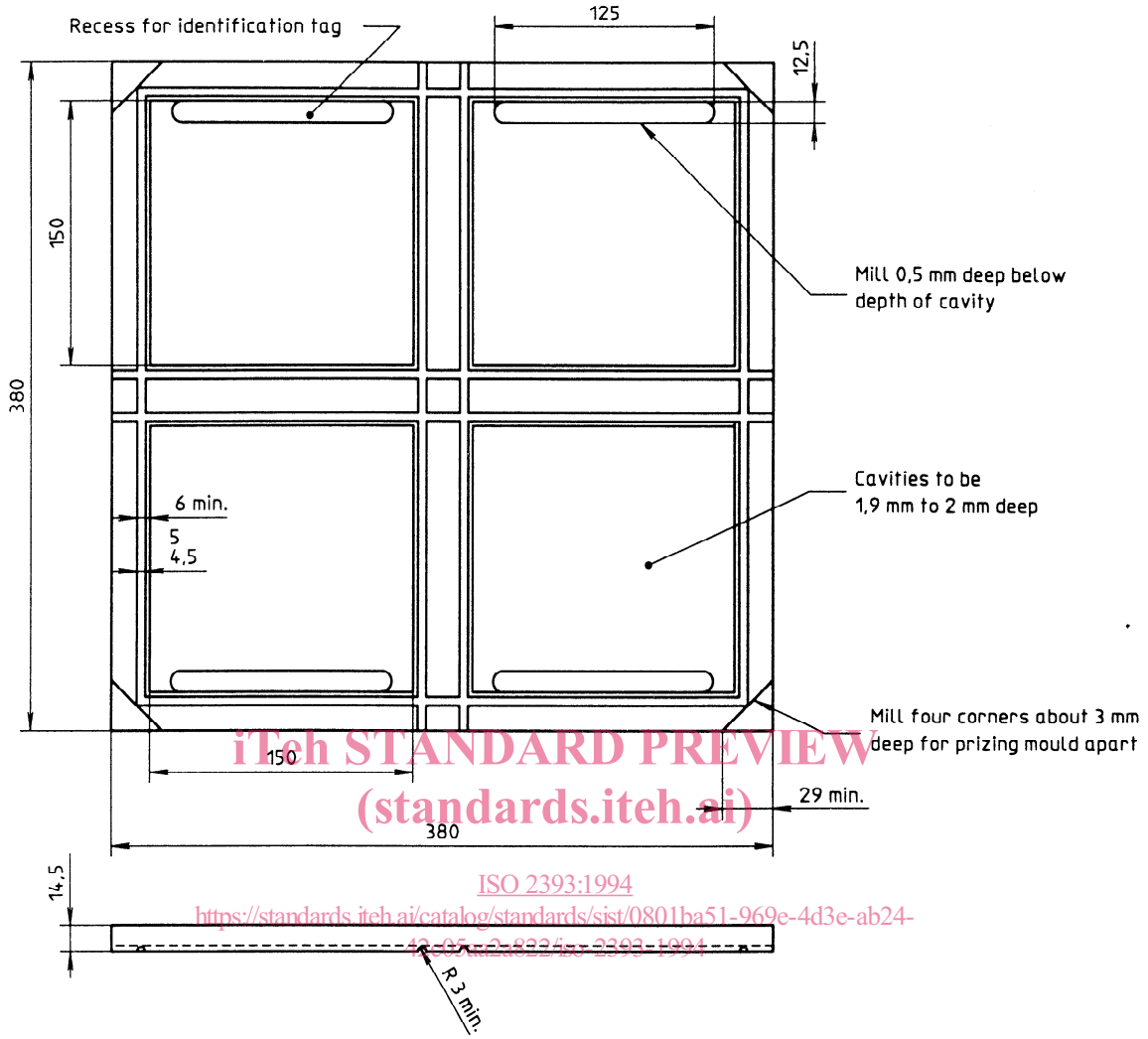
The pressing surfaces of the platens shall be plane parallel to within 0,25 mm/m when the platens are at 150 °C and closed under full pressure with a grid of soft solder or lead between them.

With either type of platen, the temperature over the mould area shall be uniform. The maximum deviation from the temperature at the centre of the platen shall not exceed ± 0,5 °C. Between adjacent platens, the temperature difference between corresponding points on the two platens shall not exceed 1 °C and the mean difference in platen temperatures shall not exceed 0,5 °C.

8.2.2 Mould

The mould shall have cavity sections of sufficient size to allow the required number of dumb-bells, as specified in ISO 37, to be cut. A suitable mould is shown in figure 1, but a preferred alternative is a mould with rectangular cavities of approximate dimensions 150 mm × 145 mm × 2 mm. This mould enables the milled sheet to be positioned unequivocally with respect to the direction of the grain.

Dimensions in millimetres



NOTE — The recesses for identification tags are optional.

Figure 1 — Design for four-cavity mould

The cavities to within 6 mm of the edges shall be between 1,9 mm and 2,0 mm deep. The corners of the cavities may be rounded with a radius not greater than 6 mm.

The moulding surfaces shall be clean and highly polished. Moulds constructed of hardened steel are preferred, but chromium-plated mild steel and stainless steel are also acceptable. The cover of the mould shall be a flat plate at least 10 mm in thickness and preferably hinged to the cavity section to minimize scratching of the mould surfaces.

Instead of a separate mould and cover, the cavities may be cut directly into the platen of the press.

Normally, a mould lubricant shall not be used on the mould surfaces. If a mould lubricant is required, only a type which does not affect the vulcanized slab shall be used. The excess lubricant shall be removed by vulcanizing and discarding at least one set of sheets. A silicone-type lubricant or mild soap solution has been found satisfactory, but silicone lubricant shall not be used when moulding silicone rubbers.

8.3 Vulcanization procedure

8.3.1 Bring the mould to within ± 0,5 °C of the vulcanization temperature 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 mould by means of a thermocouple or other suitable temperature-measuring device inserted in one of the overflow grooves and in intimate contact with the mould.