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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 modes opératoires

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

The committee responsible for this document is ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.

This fourth edition cancels and replaces the third edition (ISO 2393:2008), which has been technically revised:

— 7.1 has been added stating that the Laboratory Internal Mixer is the preferred equipment;

— 7.2.1.1 has become 7.3.1.1; the subclause has been revised to delete the reference to EST8, which is no longer available, and because a control mix for a specific rubber type is not relevant in this International Standard;

— dimensions of the mould cavity have been specified in 8.2.2;

— instructions for removing trapped air after inserting the blanks have been added in 8.3.2.

This corrected version of ISO 2393:2014 incorporates the following corrections:

— reinstatement of the numbering of subdivisions of subclauses 5.1, 5.2, 6.2, 7.2, 7.3.1, 7.3.2, 7.3.3, 8.1, 8.3, 9.1.

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 rubber test mixes specified in the various International Standards for the evaluation of such test mixes.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
formulation batch mass
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 evaluation procedure

3.2
batch mass
mass of test mix prepared in one mixing operation

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

3.4
nominal mixer capacity
proportion of the total free volume which is used in the mixing process

Note 1 to entry: A value of 0,75 times the total free volume has been found suitable for mixers with tangential rotors.

3.5
evaluation procedure
International Standard specifying the materials, test formulation, mixing procedure, vulcanization procedure, and test methods for the evaluation of a type of rubber or compounding ingredient

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 evaluation procedure.

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 evaluation procedure.

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

5.1.2 The batch mass for the laboratory 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 The batch mass shall be taken into consideration when determining weighing tolerances. In general, 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 When the batch mass is less than four times the formulation batch mass, the weighing tolerances shall be one tenth of those given in 5.2.1. Therefore, 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

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 batch-mixing report.

6 Mixing equipment

6.1 Mixing mill

The characteristics of a standard laboratory mill are as follows:

— roll diameter (OD) 150 mm to 155 mm;

— roll length (between guides) 250 mm to 280 mm;

— speed of front (slow) roll 24 r/min ± 1 r/min;

— roll-speed ratio preferably 1:1,4;

— clearance between rolls (adjustable) 0,2 mm to 8,0 mm;

— temperature-control tolerance ± 5 °C (unless otherwise specified).

WARNING — The mill should be equipped with suitable safety devices to protect against accidents and the operator should be provided with suitable equipment to protect against hazardous chemicals, in accordance with national safety regulations.

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

NOTE 2 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 Laboratory internal mixer

6.2.1 Laboratory internal mixers are available in a variety of sizes ranging from a nominal mixer capacity of 65 cm^3 (described previously as a miniature internal mixer) to about 3 000 cm^3. Interlaboratory test programmes (ITPs) on two different types of synthetic rubber and on natural rubber have shown that mixer capacity does not have a significant effect on the results, provided that good dispersion of all ingredients is achieved (see Annex C for a discussion of the effects of certain mixer variables).

For interlaboratory comparisons, it is preferable to use the same type of mixer and to align the mixing conditions (nominal mixing capacity, mixer head starting temperature, rotor type, and speed, mixing time) as closely as possible.

All of the mixers used in the interlaboratory test programmes (ITPs) were of the tangential-rotor type, and included Banbury, cam, and other types. No laboratory suggested using an intermeshing-rotor mixer. Therefore, the mixer described as type B in the previous edition of this International Standard has been deleted. However, an intermeshing type may be used when the interested parties agree.

6.2.2 This International Standard describes general requirements for laboratory internal mixers ranging in nominal mixer capacity from about 65 cm^3 to about 2 000 cm^3.

WARNING — Laboratory internal mixers should be equipped with a suitable exhaust system and suitable safety devices to prevent accidents, in accordance with national safety regulations. The operators should be provided with suitable equipment to protect them against hazardous chemicals, in accordance with national regulations.

NOTE The smaller laboratory internal mixers can only provide enough compound for curemeter testing and limited stress-strain testing.

6.2.3 All laboratory 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 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.

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

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

6.2.6 All laboratory 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.

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

6.2.8 When rotor clearances exceed the "as delivered" values by approximately 50 %, 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.

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

6.2.10 The smallest laboratory internal mixer can be fitted with rotors of different types, resulting in different nominal mixer capacities (see Table 1).

Table 1 — Rotor types for smallest laboratory internal mixer

Parameter	Cam	Banbury
Total free volume (cm^3)	85 ± 1	75 ± 1
Nominal mixer capacity (cm^3)	64	56
Rotor friction ratio	1,5:1	1,5:1

7 Mixing procedures

7.1 General

The Laboratory Internal Mixer is preferred over the laboratory mill

7.2 Mill mixing procedure

7.2.1 Batches shall be mixed with the rubber banded on the front roll, unless otherwise specified in the appropriate evaluation procedure.

7.2.2 The temperature at the middle of the surface of the rolls shall be measured during the mixing procedure, either continuously on a recorder or frequently enough with a manual device (having an accuracy of ±1 °C or better) 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.

7.2.3 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.

7.2.4 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.

7.2.5 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 evaluation procedure.

7.2.6 Pass the rolled batch endwise through the mill six times unless otherwise specified in the appropriate evaluation procedure.

7.2.7 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 contain small amounts of volatiles which may be lost at the temperature of mixing, with the result that the above limit may not be met. In such cases, the difference shall be reported and justified.

7.2.8 The mixed batch shall be cooled to room temperature on a flat, clean, dry, metal surface. Alternatively, the mixed batch may be cooled in water, but different results may be obtained.

The cooled batches shall be wrapped in aluminium foil or other suitable material to prevent contamination by other compounds.

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

a) the roll-speed ratio (friction ratio) and roll speeds,

b) the distance between the guides,

c) the maximum and minimum roll temperatures recorded during the mixing procedure,

d) the temperature used for conditioning the carbon black,

e) the method of cooling the mixed batch,

f) any mass loss greater than the limits given in 7.2.7, with the reason for acceptance, and

g) the number of the International Standard specifying the evaluation procedure in which the test mix is to be used.

7.3 Laboratory internal mixer mixing procedure

7.3.1 General

7.3.1.1 The mixing technique used shall be such as to achieve good dispersion of all the ingredients.

It is not possible to state in this International Standard whether a single or two stage mixing procedure is preferred. Reference shall be made to the relevant rubber evaluation procedure.

Where a technique is given in a particular evaluation procedure, it is permissible to make changes to the technique to ensure good dispersion.

NOTE The ITP data in Annex A shows that both single and two stage mixing procedures give equivalent results for the rubber types evaluated, namely ESBR, BR, and NR.

7.3.1.2 For each batch mixed, the laboratory internal mixer conditions shall be the same during the preparation of a series of identical mixes. At the beginning of each series of rubber 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 laboratory internal mixer 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 shall not be altered during the mixing of a series of test batches.

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

7.3.2 Two-stage mixing procedure

7.3.2.1 The discharged batch shall be consolidated on a standard laboratory mill in the manner specified in the appropriate evaluation procedure and allowed to cool to room temperature on a flat, clean, dry, metal surface.

7.3.2.2 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 can be lost at the temperatures of mixing, with the result that the above limit cannot be met. In such cases, the difference shall be reported and justified. This also applies to 7.3.2.5 and 7.3.3.1.

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

7.3.2.4 If the final-stage mix is to be prepared in the internal mixer, cut the batch from the first stage into strips for easier feeding and add the remaining ingredients in accordance with the instructions in the appropriate evaluation procedure.

If the final stage is to be prepared on the mill, add the ingredients in accordance with the instructions in the appropriate evaluation procedure.

Unless otherwise stated, the batch size shall be reduced to four times the formulation batch mass.

7.3.2.5 When the laboratory internal mixer is used for the final stage, the discharged batch shall be consolidated as in 7.3.2.1.

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

7.3.2.6 Remove a curemeter test piece and (if required) a compound viscosity test piece from the batch. Then pass the batch four times through the mill at a roll temperature of 50 °C ± 5 °C, unless otherwise stated in the appropriate evaluation procedure. 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.

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

a) the mixer head starting temperature,

b) the mixing time,

c) the rotor speed,

d) the ram pressure,

e) the temperature of the mix at discharge,

f) the mixing technique — order of adding ingredients, times, etc.,

g) the type of mixer used — size, rotor type, etc.,

h) any allowable mass loss outside the limits given in 7.3.2.2 and 7.3.2.5, with the reason for acceptance,

i) the temperature used for conditioning the carbon black, and

j) the number of the International Standard specifying the evaluation procedure in which the test mix is to be used.

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

For mixes where the final stage is carried out on the mill, the procedure given in 7.1 shall be used and a separate report prepared in accordance with 7.2.9 with the exception of the carbon black conditioning temperature.

7.3.3 Single-stage mixing procedure

7.3.3.1 The discharged batch shall be consolidated as in 7.3.2.1. The mass of the mixed batch shall not differ from the total mass of the ingredients by more than +0,5 % or –1,5 %.

7.3.3.2 Carry out the procedure as given in 7.3.2.6, unless otherwise stated in the appropriate evaluation procedure.

7.3.3.3 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 mixing technique — order of adding ingredients, times, etc.,

g) the type of mixer used — size, rotor type, etc.,

h) any allowable mass loss outside the limits given in 7.3.3.1, with the reason for acceptance,

i) the temperature used for conditioning the carbon black, and

j) the number of the International Standard specifying the evaluation procedure in which the test mix is to be used.

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

8.1 Conditioning of batches and blank preparation

8.1.1 Batches shall be conditioned for between 2 h and 24 h at one of the standard laboratory temperatures specified in ISO 23529, 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 less than 50 %.

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 g 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 7.3.2.6 shall be used.

Table 2 — Mass of blank

Density Mg/m^3	Mass g
0,94	47
0,96	48
0,98	49

Table 2 *(continued)*

Density Mg/m^3	Mass g
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 is 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.