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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

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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 third edition cancels and replaces the second edition (ISO 2393:1994), which has been technically revised.

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 referenced documents are indispensable for the application of this document. 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 2322, *Styrene-butadiene rubber (SBR) — Emulsion- and solution-polymerized types — Evaluation procedures*

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 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 rpm ± 1 rpm;

— 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 (previously described as a miniature internal mixer) to about 3 000 cm^3. Interlaboratory test programmes (ITPs) on two different types of synthetic 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 "new" 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}_{-1}	75^{+1}_{-1}
Nominal mixer capacity (cm^3)	64	56
Rotor friction ratio	1,5:1	1,5:1

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 evaluation procedure.

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 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.

7.1.3 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 evaluation procedure.

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

7.1.7 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.

7.1.8 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.

7.1.9 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 7.1.7, with the reason for acceptance;

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

7.2 Laboratory internal mixer mixing procedure

7.2.1 General

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

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

In order to check whether the mixing technique is satisfactory, a control mix should preferably be made using SBR 1500 EST8, in accordance with series A in ISO 2322. Test results close to those quoted in Tables A.3 and A.4 should be obtained. If EST8 is not available, then SBR 1500 may be used, but the results should be interpreted with caution as material from different suppliers can differ markedly in cure rate.

7.2.1.2 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.

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

7.2.2 Two-stage mixing procedure

7.2.2.1 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.

7.2.2.2 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 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 and justified. This also applies to 7.2.2.5 and 7.2.3.1.

7.2.2.3 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.

7.2.2.4 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.

7.2.2.5 When the laboratory internal mixer is used for the final stage, the discharged batch shall be consolidated as in 7.2.2.1.

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

7.2.2.6 Unless otherwise stated in the appropriate evaluation procedure, 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.

7.2.2.7 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 7.2.2.2 and 7.2.2.5, with the reason for acceptance;

i) the temperature used for conditioning the carbon black;

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 7.1.9 with the exception of the carbon black conditioning temperature.

7.2.3 Single-stage mixing procedure

7.2.3.1 The discharged batch shall be consolidated as in 7.2.2.1. The mass of the mixed batch shall not differ from the total mass of the ingredients by more than + 0,5 % or – 1,5 %.

7.2.3.2 Unless otherwise stated in the appropriate evaluation procedure, 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.

7.2.3.3 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 7.2.3.1, with the reason for acceptance;

i) the temperature used for conditioning the carbon black;

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 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 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 7.2.2.6 shall be used.

Table 2 — Mass of blank

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