INTERNATIONAL STANDARD ISO 2393

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

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FOREWORD

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Draft International Standards adopted by the Technical Committees are circulated to the Member Bodies for approval before their acceptance as International Standards by the ISO Council.

International Standard ISO 2393 was drawn up by Technical Committee ISO/TC 45, *Rubber and rubber products*, and circulated to the Member Bodies in April 1971.

It has been approved by the Member Bodies of the following countries :

Canada	India	South Africa, Rep. of
Ceylon	Italy	Spain
Czechoslovakia	Korea, Dem.P.Rep. of	Switzerland
Egypt, Arab Rep. of	Netherlands	Thailand
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The Member Bodies of the following countries expressed disapproval of the document on technical grounds :

Sweden
U.S.A.

Printed in Switzerland

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

1 SCOPE AND FIELD OF APPLICATION

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

2 REFERENCE

ISO/R 471, *Standard atmospheres for the conditioning and testing of rubber test pieces.*

3 STANDARD MATERIALS

The standard materials required for the various standard test recipes shall be NBS[1] Standard reference materials as indicated in the appropriate International Standard, or shall be in accordance with equivalent national standards.

4 COMPOUNDING PROCEDURE

4.1 The standard laboratory mill batch mass, in grams, shall be based on four times the recipe mass unless otherwise stated in the appropriate International Standard.

4.2 The rubber and carbon black shall be weighed to the nearest 1 g; the sulphur and the accelerator to the nearest 0,02 g and the zinc oxide and stearic acid to the nearest 0,1 g. All other ingredients shall be weighed with an accuracy of ± 1 %.

4.3 Unless otherwise specified, carbon black shall be conditioned, before weighing, by heating in an oven at a temperature of $100 \begin{smallmatrix}+10\\0\end{smallmatrix}$ °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.

NOTE — The standard batch, in grams, for internal mixers shall be equal to the normal mixer capacity in cubic centimetres multiplied by the density of the compound.

5 MIXING MILL

5.1 The mixing mill shall be a mill having rolls of 150 ± 5 mm outside diameter. The mill shall be equipped with guides spaced 250 to 280 mm apart to retain the rubber at the nip.

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

Preferably the mill should be capable of operating at friction or at even speed.

5.2 The speed of the slow roll (front roll) shall be 24 ± 1 rev/min, and the ratio between the fast and slow roll shall be preferably 1,4 : 1. Other ratios may be used, but modifications in mixing procedure may be required to obtain comparable results.

5.3 Means shall be provided for controlling the mill roll temperatures to the specified temperature within a tolerance of ± 5 °C unless otherwise specified in the appropriate International Standard.

5.4 The clearance between the rolls shall be adjustable at least from 0,2 to 3,0 mm. Roll clearance shall be determined by means of two lead strips 10 ± 3 mm in width, at least 50 mm long and 0,25 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[2] greater than 50 (ML 1 + 4 100°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 strip shall be measured with a micrometer to an accuracy of ± 0,01 mm. Tolerance on roll clearance shall be ± 10 % or ± 0,05 mm, whichever is the larger.

1) National Bureau of Standards of the U.S.A.

2) Determined in accordance with ISO/R 289, *Determination of viscosity of natural and synthetic rubbers by the shearing disk viscometer.*

6 MILL MIXING PROCEDURE

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

6.2 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 maintain the desired temperature. The batch may be removed momentarily from the mill to enable the surface temperature of the front roll to be measured.

6.3 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.

6.4 The batch must not be cut while free powder is evident on the bank or on the milling surface. Compounding materials falling through the roll shall be carefully collected and returned to the batch.

6.5 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.

6.6 The mass of the mixed batch shall not differ from the total mass of all the materials by more than ± 1,0 %.

6.7 The mixed batch shall be cooled to room temperature on a flat, clean, dry metal surface.

7 PREPARATION OF STANDARD VULCANIZED SHEETS FOR DUMB-BELL TEST PIECES

7.1 Preparation of batches

7.1.1 Batches shall be conditioned for between 2 and 24 h at one of the standard temperatures specified in ISO/R 471, (20 °C, 23 °C, 27 °C) ± 2 °C, preferably in a closed container to prevent absorption of moisture from the air.

7.1.2 Where re-milling is required, this shall be done by passing the batch, without banding, endways ten times through the rolls. After each pass through the rolls, the batch shall be rolled on itself. Clearance between the rolls shall be 0,2 mm and the temperature shall be as prescribed for mixing. After the last pass, adjust the distance between the rolls to 1,4 mm and band the batch on the front roll. Three 3/4 cuts shall be made each way and the batch sheeted from the mill to give a sheet, after cooling, of approximately 2,2 mm thickness.

7.1.3 After mixing or re-milling, the sheeted batch shall be placed on a flat, clean, dry metal surface and the pieces 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 uncured pieces shall be within $^{+3}_{0}$ g of the mass given in the table when they are vulcanized in the mould specified in 7.2.2.

TABLE – **Mass of uncured pieces**

Density	Mass
Mg/m^3	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

7.2 Vulcanization equipment

7.2.1 *Press*

The press shall be capable of exerting a pressure of not less than 3,5 MN/m^2 on the cavity areas of the mould during the entire period of vulcanization. It shall have heated platens of sufficient size so that no portion of the rubber will be nearer than 75 mm from the edge of the platen during vulcanization. The platens shall preferably be made of rolled steel machined for electric or steam heating.

When the steam heating is used, either 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 should be suitably shielded from draughts.

The pressing surfaces of the platen 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.

7.2.2 *Mould*

The mould shall have cavity sections similar in dimensions to that shown in Figure 1, which gives sheets approximately 150 mm × 150 mm × 2 mm. The cavities to within 6 mm of the edges shall be between 1,9 and 2,0 mm deep. The corners of the cavities may be rounded with a radius not greater than 6 mm.

An alternative type of test slab mould, made by the cut-off bar method with a lower plate thickness of about 20 mm, is shown in Figure 2. The moulding surfaces shall be clean, highly polished, and hard chromium plated. 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 residual type lubricant, which does not affect the cured slab, should be used, and the excess lubricant should be removed by curing and discarding at least one set of sheets. A silicone-type lubricant or mild soap solution has been found satisfactory.

A film of suitable material such as a non-lubricated aluminium foil, 0,1 mm thick, may be placed above and below the sheet in the mould to prevent contamination with materials remaining in the mould from previous cures. Compensation should be made in the mass of unvulcanized pieces.

7.3 Vulcanization procedure

7.3.1 Bring the mould to vulcanization temperature within ± 0,5 °C 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.

7.3.2 Open the press, insert the unvulcanized pieces in the mould and close the press in the minimum time possible. When the mould is removed from the press to insert the pieces, precautions should be taken to prevent excessive cooling of the mould by contact with cool metal surfaces or by exposure to air draughts.

7.3.3 The time of vulcanization shall be considered to be the period between the instant the pressure is applied fully and the instant the pressure is released. Hold the mould under a minimum pressure of 3,5 MN/m^2 on the cavity areas during vulcanization.

As soon as the press is opened, remove the vulcanized sheets from the mould and cool in water (room temperature or lower) or on a metal surface (for items used for electrical measurements) for 10 to 15 min. Then wipe dry the sheets cooled in water and reserve for test. In both of the preceding operations, take care to prevent undue stretching or deformation.

7.3.4 Store vulcanizates at one of the standard temperatures specified in ISO/R 471.

7.3.5 For all test purposes the minimum time between vulcanization and testing shall be 16 h.

7.3.6 Maximum time between vulcanization and testing shall be 4 weeks and for evaluations intended to be comparable, the tests, as far as possible, shall be carried out after the same time interval.

8 PREPARATION OF STANDARD VULCANIZED DISCS FOR RING TEST PIECES

8.1 Preparation of batches

8.1.1 Batches shall be conditioned for between 2 and 24 h at one of the standard temperatures specified in ISO/R 471 (20 °C, 23 °C, 27 °C) ± 2 °C, preferably in a closed container to prevent absorption of moisture from the air unless the relative humidity is controlled at 35 ± 5 %.

8.1.2 Where re-milling is required, this shall be done by passing the batch, without banding, endways, ten times through the rolls. After each pass through the rolls, the batch shall be rolled on itself. Clearance between the rolls shall be 0,2 mm and the temperature shall be as prescribed for mixing. After the last pass, adjust the distance between the rolls to 1,4 mm and band the batch on the front roll. Three 3/4 cuts shall be made each way and the batch sheeted from the mill to give a sheet, after cooling, of approximately 4,2 mm thickness.

8.1.3 After mixing or re-milling, the sheeted batch shall be placed on a flat, clean, dry metal surface. Circular pieces 63 to 64 mm in diameter are stamped from the sheet so that they fit easily into the cylindrical mould cavities of the mould shown in Figure 3.

8.2 Vulcanization equipment

8.2.1 *Press*

As specified in 7.2.1.

8.2.2 *Mould*

The mould shall have cavity sections similar in dimensions to those shown in Figure 3, which gives discs 65 mm in diameter and 4 mm thick. The mould consists of a lid and a cavity section hinged to each other. The hinges are provided with oblong holes so as to provide a plane parallel position of the pressing surfaces to prevent distortion of the lid, if the press is closed when loaded with thick discs.

The cavity section contains several groups of cylindrical cavities for the moulding of three interconnected discs. Close to each group of cavities is a 10 mm wide recess which can be used for the identification of individual compounds. For technical reasons the depth of the recess is less than the disc cavities. For identification purpose, embossed aluminium strips are placed in the recess, which on moulding leave an identity tag attached to the group of three discs.

The number of cavities depends on the platen size of the curing press available. Hard aluminium alloys have proved to be suitable for the manufacture of the moulds shown in Figure 3. Thinner moulds (for example lid 4 mm; cavity section 8 mm) can be made from steel, but hinges required for thinner moulds are more difficult to make.

The cavities shall be uniform in depth to within 0,05 mm. The corners of the cavities may be rounded with a radius not greater than 0,5 mm.

The moulding surface shall be clean and highly polished.

8.3 Vulcanization procedure

8.3.1 Bring the mould to vulcanization temperature within ± 0,5 °C 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.

8.3.2 Open the press, insert the unvulcanized pieces in the mould and close the press in the minimum time possible. When the mould is removed from the press to insert the pieces, precautions should be taken to prevent excessive cooling of the mould by contact with cool metal surfaces or by exposure to air draughts.

8.3.3 The time of vulcanization shall be considered to be the period between the instant the pressure is applied fully and the instant the pressure is released. Hold the mould under a minimum pressure of 3,5 MN/m^2 on the cavity areas during vulcanization.

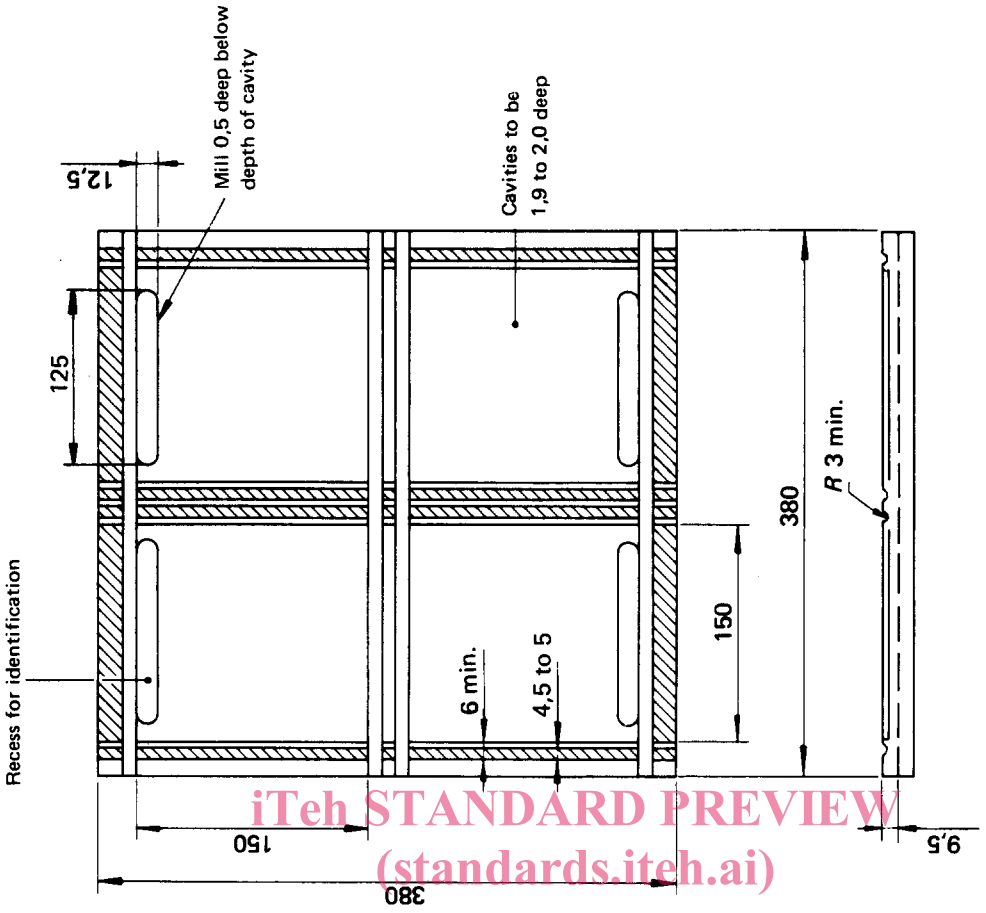
As soon as the press is opened, remove the vulcanized sheets from the mould and cool in water (room temperature or lower) or on a metal surface (for items used for electrical measurements) for 10 to 15 min. Then wipe dry the sheets cooled in water and reserve for test. In both of the preceding operations, take care to prevent undue stretching or deformation.

8.3.4 Store vulcanizates at one of the standard temperatures specified in ISO/R 471.

8.3.5 For all test purposes the minimum time between vulcanization and testing shall be 16 h.

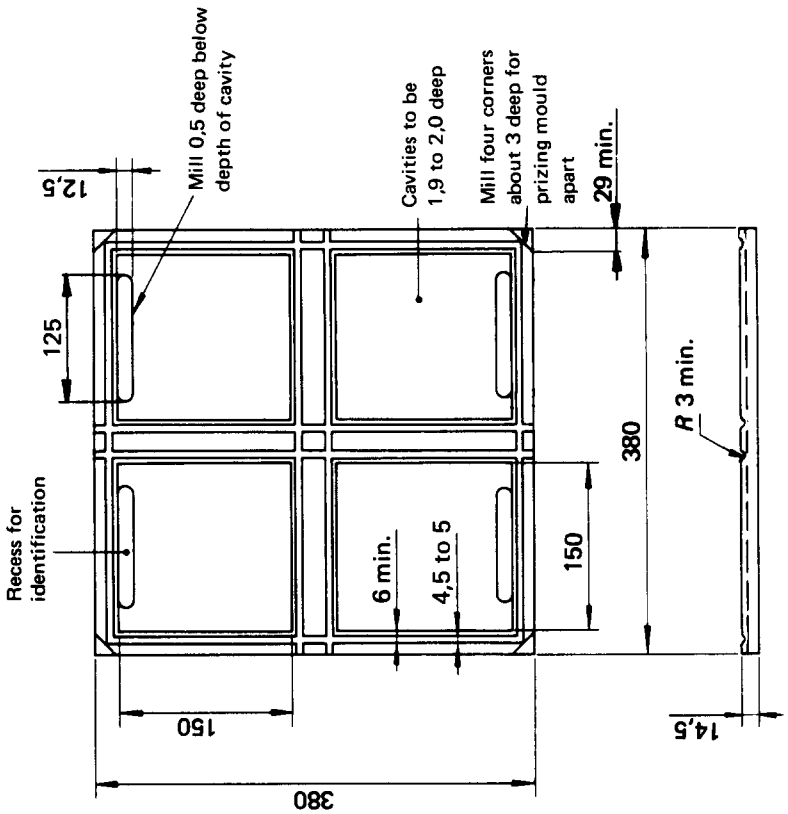
8.3.6 Maximum time between vulcanization and testing shall be 4 weeks and for evaluations intended to be comparable, the tests, as far as possible, shall be carried out after the same time interval.

Dimensions in millimetres



NOTE – The recesses for identification are optional.

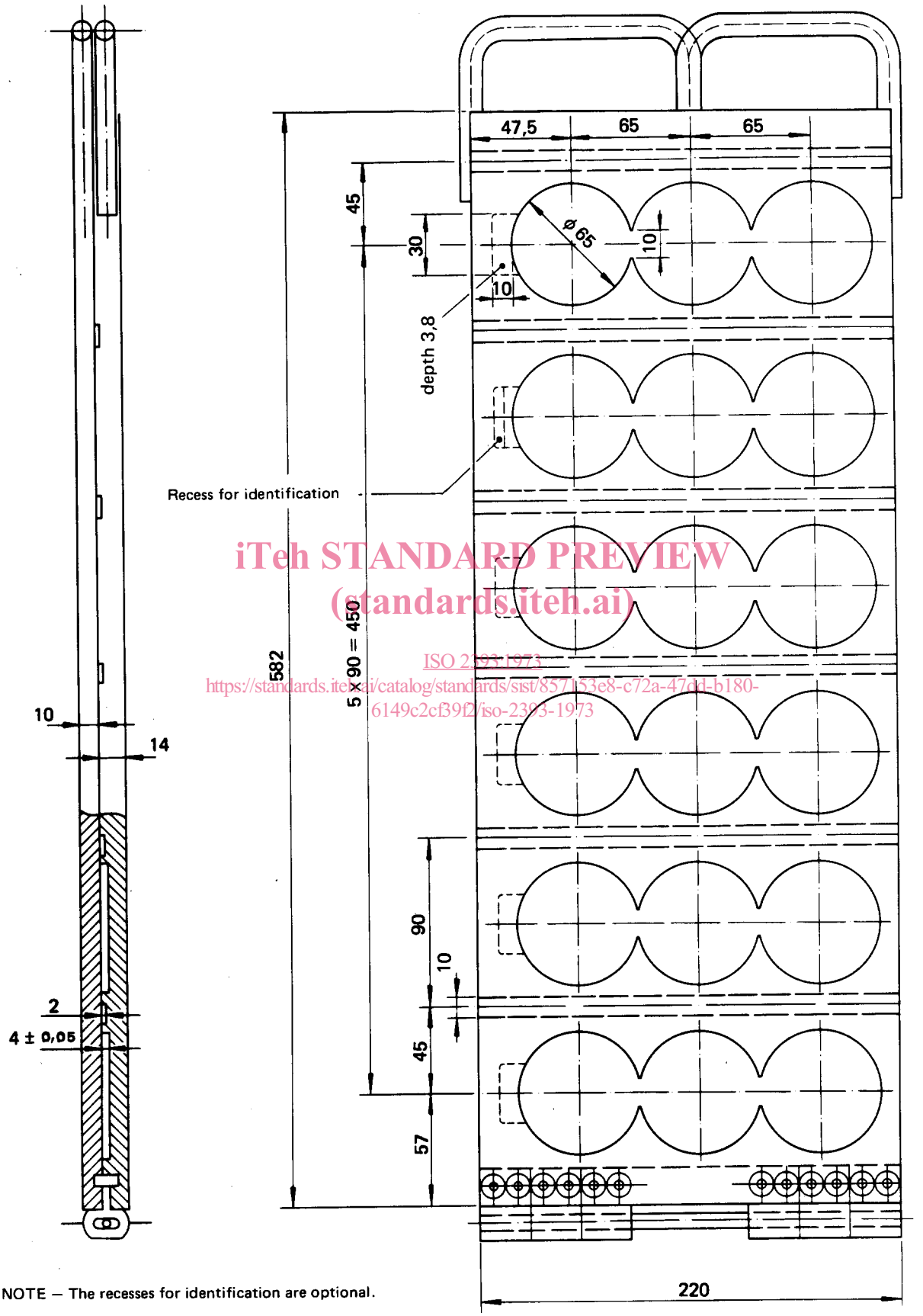
FIGURE 2 – **Cut-off bar type of test slab mould**



NOTE – The recesses for identification are optional.

FIGURE 1 – **Design for four-cavity mould**

Dimensions in millimetres



NOTE — The recesses for identification are optional.

FIGURE 3 — **Mould for vulcanizing discs for ring test pieces**