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Plastics — Compression moulding of test specimens of thermosetting materials

Plastiques — Moulage par compression des éprouvettes en matières thermodurcissables

Reference number
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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 295 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 12, *Thermosetting materials*.

This third edition cancels and replaces the second edition (ISO 295:1991), which has been technically revised.

Plastics — Compression moulding of test specimens of thermosetting materials

1 Scope

This International Standard

— establishes the general principles and lays down the procedure to prepare test specimens of heat- and pressure-moulded thermosetting material from different moulding compounds;

— specifies the details for test specimen preparation to be included with the test reports on properties;

— gives the general principles for the design of the mould intended for the preparation of the test specimens.

The conditions required for preparing, in a reproducible manner, test specimens which will give comparable results are discussed relative to the substance under consideration.

The method applies to phenolic resin, aminoplastic, melamine/phenol, epoxy and unsaturated-polyester based thermosetting powder moulding compounds (PMCs). Due to the nature of certain moulding compounds, their flow properties or other variable factors, it may be necessary to prepare the test specimens according to special methods. The latter are normally in an agreement between the interested parties and noted in the moulding report.

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 472:1999, *Plastics — Vocabulary*

ISO 1183:1987, *Plastics — Methods for determining the density and relative density of non-cellular plastics*

ISO 3167:2002, *Plastics — Multipurpose test specimens*

ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 14526-1, *Plastics — Phenolic powder moulding compounds (PF-PMCs) — Part 1: Designation system and basis for specifications*

ISO 14526-2, *Plastics — Phenolic powder moulding compounds (PF-PMCs) — Part 2: Preparation of test specimens and determination of properties*

ISO 14526-3, *Plastics — Phenolic powder moulding compounds (PF-PMCs) — Part 3: Requirements for selected moulding compounds*

ISO 14527-1, *Plastics — Urea-formaldehyde and urea/melamine-formaldehyde powder moulding compounds (UF- and UF/MF-PMCs) — Part 1: Designation and basis for specifications*

ISO 14527-2, *Plastics — Urea-formaldehyde and urea/melamine-formaldehyde powder moulding compounds (UF- and UF/MF-PMCs) — Part 2: Preparation of test specimens and determination of properties*

ISO 14527-3, *Plastics — Urea-formaldehyde and urea/melamine-formaldehyde powder moulding compounds (UF- and UF/MF-PMCs) — Part 3: Requirements for selected moulding compounds*

ISO 14528-1, *Plastics — Melamine-formaldehyde powder moulding compounds (MF-PMCs) — Part 1: Designation system and basis for specifications*

ISO 14528-2, *Plastics — Melamine-formaldehyde powder moulding compounds (MF-PMCs) — Part 2: Preparation of test specimens and determination of properties*

ISO 14528-3, *Plastics — Melamine-formaldehyde powder moulding compounds (MF-PMCs) — Part 3: Requirements for selected moulding compounds*

ISO 14529-1, *Plastics — Melamine/phenolic powder moulding compounds (MP-PMCs) — Part 1: Designation system and basis for specifications*

ISO 14529-2, *Plastics — Melamine/phenolic powder moulding compounds (MP-PMCs) — Part 2: Preparation of test specimens and determination of properties*

ISO 14529-3, *Plastics — Melamine/phenolic powder moulding compounds (MP-PMCs) — Part 3: Requirements for selected moulding compounds*

ISO 14530-1, *Plastics — Unsaturated-polyester powder moulding compounds (UP-PMCs) — Part 1: Designation system and basis for specifications*

ISO 14530-2, *Plastics — Unsaturated-polyester powder moulding compounds (UP-PMCs) — Part 2: Preparation of test specimens and determination of properties*

ISO 14530-3, *Plastics — Unsaturated-polyester powder moulding compounds (UP-PMCs) — Part 3: Requirements for selected moulding compounds*

ISO 15252-1, *Plastics — Epoxy powder moulding compounds (EP-PMCs) — Part 1: Designation system and basis for specifications*

ISO 15252-2, *Plastics — Epoxy powder moulding compounds (EP-PMCs) — Part 2: Preparation of test specimens and determination of properties*

ISO 15252-3, *Plastics — Epoxy powder moulding compounds (EP-PMCs) — Part 3: Requirements for selected moulding compounds*

3 Terms and definitions

For the purposes of this document, the terms and definitions of ISO 472 and the following apply.

3.1
spatial temperature differences
differences in temperature existing simultaneously at various points inside the mould after the temperature adjustment device has been set at a given temperature and after a permanent thermal equilibrium has been reached

3.2
temporal temperature differences
differences in temperature that may occur at a single given point on the inside of the mould at various times after the temperature adjustment device has been set at a given temperature and after a permanent thermal equilibrium has been reached

3.3
crosslinking time or cure time
time interval between the end of the mould-closing operation and the beginning of the mould-opening operation

NOTE In practice, the cure time is generally counted as from the moment when the pressure reaches the specified value.

4 Apparatus

4.1 Compression mould, made of steel, able to withstand the prescribed moulding temperatures and pressures.

The mould shall be designed so that the compression force is transmitted to the moulding material with no appreciable loss. It may be a single-cavity or a multi-cavity type mould. Figure 1 shows an example of a single-cavity positive mould. The cavity of the mould may have the shape of the multi-purpose test specimen in accordance with ISO 3167. In some cases, e.g., for aminoplastic moulding materials, a semi-positive mould is more suitable, even though the pressure on the moulding material is not as well defined. In this case, the test-specimen thickness shall be adjusted using spacers on the mould parting line.

The mould cavity may have various forms, e.g., square plate, disc or multi-purpose specimen in accordance with ISO 3167.

For moulding powders, a "type E ISO 295" single-cavity mould with dimensions of 120 mm × 120 mm is recommended. In the moulding report, the plate is designated as "type Eh ISO 295," where h is the thickness in millimetres (e.g., "type E4 ISO 295" for a plate with a thickness of 4 mm and dimensions of 120 mm × 120 mm).

The majority of the test methods require a thickness of 4 mm but, for a few, for instance the measurement of certain electrical properties, thinner plates may be required. When in doubt, the dimensions specified in the test procedure itself shall be used.

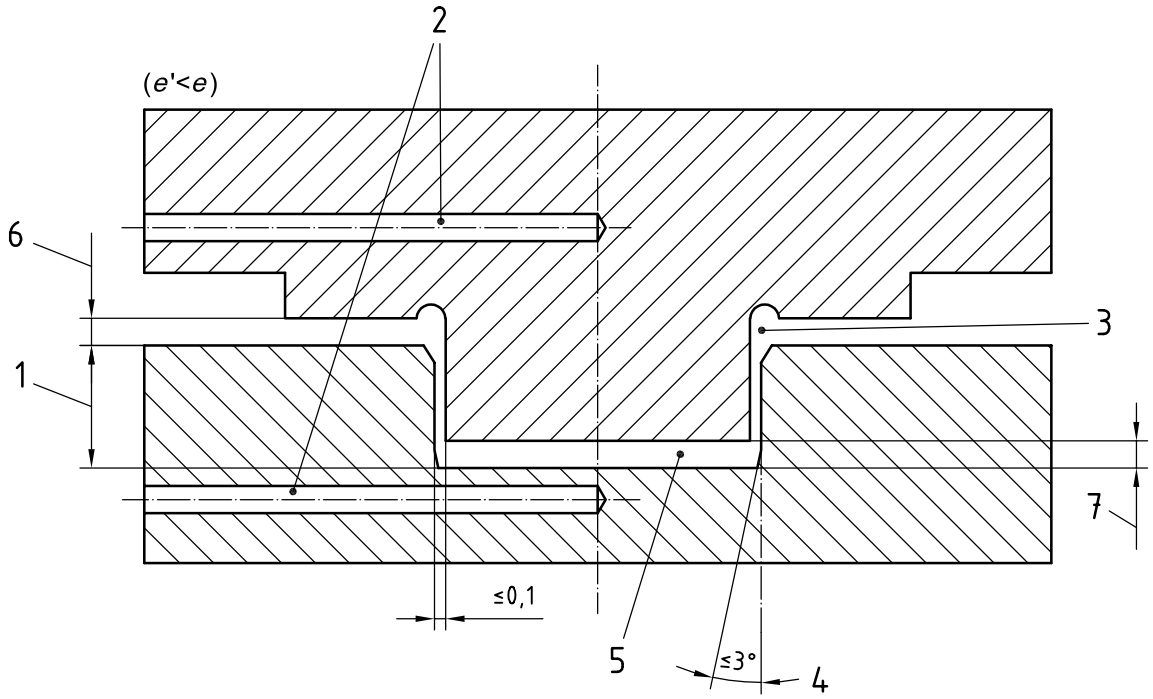
The use of a plate allows the required test specimens to be cut by machining. The test specimens shall not be taken from the edge of the plate. A margin of 10 mm is recommended.

The mould surface shall be free from superficial damage or contamination and have a roughness Ra between 0,4 µm and 0,8 µm, inclusive (see ISO 4287). Chrome plating is not always necessary, but it can prevent sticking.

The edge-taper angle, if any, shall not be greater than 3° (see Figure 1). Clearance between the vertical wall of the cavity and that of the punch shall not exceed 0,1 mm (see Figure 1). Dimension e' shall be calculated so that there is no risk of the punch damaging the die if there is no material present.

The mould shall have a cavity (see Figure 1) of sufficient volume to allow the introduction of the whole charge in one single operation. Bulk form moulding material occupies a volume two to ten times that of the object being moulded.

Dimension in millimetres



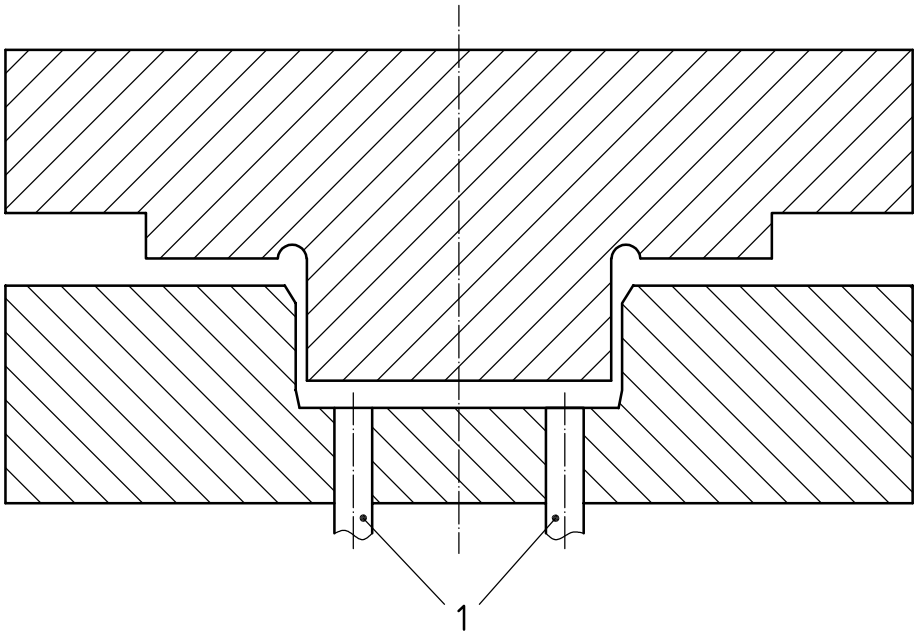
Key

1 depth of mould cavity
2 holes for probes and thermometers
3 clearance
4 edge taper
5 mould cavity
6 dimension e'
7 dimension e

Figure 1 — Example of single-cavity positive mould

The mould may be fitted with an ejector. If ejector pins are used (see Figure 2), they shall not deform the specimen in any way. If the parts are ejected by the movable bottom of the mould (see Figure 3), there shall be no significant leakage of material at the joint between the bottom and the cavity walls.

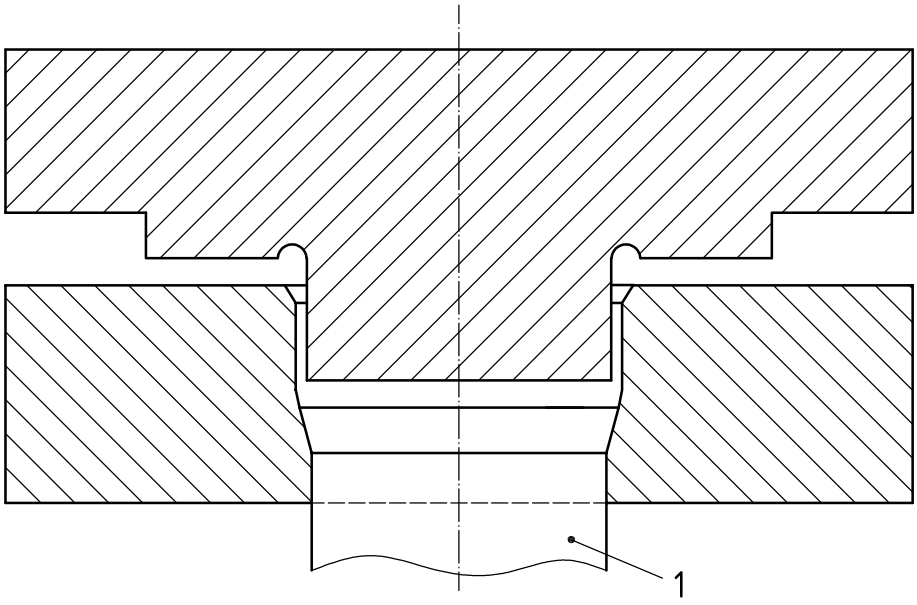
As the face of the moulded test specimen facing the lower die is heated for a longer time during the period between filling and compression, it may be worthwhile to distinguish between the two faces by means of a fixed mark in the cavity. The use of a mark in the mould cavity can also enable one to identify, if necessary, the direction in which the test specimens were taken from the plate. An example of cavity markings is given in Annex A.



Key

1 ejectors

Figure 2 — Example of a mould with ejectors



Key

1 movable bottom

Figure 3 — Example of mould with ejection by movable bottom

4.2 Heating device, capable of heating the mould so that the moulding temperature remains constant and uniform over all parts of the mould within the specified tolerances.

The mould may be heated either through the platen or by means of a built-in device (e.g., circulating fluid or electric heating elements). In the latter case, the mould shall be insulated from the press platens with a sheet of insulating material. For practical reasons, it is generally preferable to heat the mould electrically.

4.3 Mould temperature adjustment device, capable of ensuring that the optimum required temperature is maintained constant over the whole mould with an accuracy of ± 3 °C, i.e., the mould temperature shall not vary temporally or spatially by more than ± 3 °C (see 3.1 and 3.2).

4.4 Compression-moulding press, capable of ensuring that the specified pressure is applied and maintained during the whole of the curing time. The press may be hand-operated or programmed.

It is preferable to use a press having two closing speeds:

— a fast approach speed (e.g., 200 mm/s to 400 mm/s) in order to avoid precure of the material before closing;

— a slow closing speed (e.g. 5 mm/s) in order to prevent air or gases from being entrapped.

The mould shall be able to be closed within 15 s prior to pressurization in order to avoid precure before complete closure.

NOTE The oil pressure p_o, in megapascals, to be displayed on the pressure gauge in order to obtain the specified pressure p, in megapascals, is given by the equation:

$$p_o = (p \times A_1)/A$$

where

A is the area, in square metres, of the press punch head;

A_1 is the total area, in square metres, of the cavities.

4.5 Stopwatch, capable of being read to an accuracy of 1 s.

4.6 Mould-temperature measuring device, such as a pyrometer or fusible salts, with a specified accuracy.

4.7 Balance, having an accuracy of 0,1 g.

4.8 Cooling fixture: a metal plate about 20 mm thick, having at least the same area as the test specimen and a sufficient mass to prevent the warping of the test specimens during cooling down after removal from the mould (see Clause 8).

5 Conditioning of material prior to moulding

Before moulding, samples, representative of the material to be tested, shall be dried or stored in accordance with the supplier's guidelines.

Moulding materials requiring storage in sealed containers shall be stored under such conditions to prevent variations in their volatile matter content or moisture absorption.

If the material has to be restored, this shall be done in accordance with the supplier's requirements.

6 Preparation of the charge

If the volume of the material to be moulded is too great for the mould loading chamber capacity, the material may be preformed in pellet form. The pelletizing conditions shall be noted in the moulding report.

7 Moulding conditions

7.1 General

In the absence of special specifications, the moulding temperature and pressure, as well as the crosslinking time or cure time, shall be as given in the International Standard specific to the material in question:

— phenolic resin, in accordance with ISO 14526;

— aminoplastic, in accordance with ISO 14527 and ISO 14528;

— melamine/phenol, in accordance with ISO 14529;

— epoxy, in accordance with ISO 15252;

— unsaturated polyester, in accordance with ISO 14530.

Pre-treatment and moulding conditions are summarized in Table 1.

Due to the nature of certain moulding compounds, their flow properties and other factors, it may be necessary to prepare the test specimens by special methods. The latter shall form the subject of an agreement between the interested parties and shall be noted in the moulding report.

Table 1 — Pre-treatment and moulding conditions

Conditions	Type of moulding material				
	Phenolic moulding materials	Aminoplastic moulding materials		Epoxides	Unsaturated polyesters
		Urea-formaldehyde	Melamine-formaldehyde		
Pre-treatment					
Oven drying (7.2)	Permissible			Not recommended	Not recommended
Pelletizing	Permissible				
High-frequency preheating (7.3)	Permissible				Not recommended
Pre-plastification (7.4)	Permissible				Not recommended
Breathing (7.6)	Permissible			Not necessary	Not recommended
Moulding					
Temperature, in degrees C	As referenced in the International Standard specific to the material in question (see 7.1)				
Pressure, in megapascals					
Cure time, in seconds					
Mould					
Surface finish	Surface finish Ra = 0,4 µm to 0,8 µm				
Chrome plating	Preferable				Required

7.2 Drying

Phenolic and aminoplastic moulding materials may be dried. The material shall be spread out in a thin layer and oven dried under the following conditions:

— phenolic moulding materials: 30 min at (90 ± 3) °C, or 15 min at (105 ± 3) °C;

— aminoplastic moulding materials: 60 min at (90 ± 3) °C.

The material shall be moulded immediately upon removal from the oven. Drying conditions shall form the subject of an agreement between the interested parties and the conditions shall be noted in the moulding report.

7.3 High-frequency preheating

High-frequency preheating is permissible in the case of epoxy moulding materials, phenolic moulding materials, aminoplastic moulding materials and dry granulated polyesters. It permits a reduction in curing time. The preheated material shall be moulded immediately after preheating. High-frequency preheating conditions shall form the subject of an agreement between the interested parties and the conditions shall be noted in the moulding report.

7.4 Pre-plastification

Pre-plastification is permissible in the case of epoxy, phenolic and aminoplastic moulding materials. It ensures thermal and mechanical homogenization of the material. The pre-plasticized material shall be moulded immediately after pre-plastification. Pre-plastification conditions shall form the subject of an agreement between the interested parties and the conditions shall be noted in the moulding report.

7.5 Release agents

In general, moulding materials incorporate internal lubricants to facilitate mould release.

Release agents, i.e., substances used in order to facilitate removal from the mould, may be used only if it has been shown that they have no influence on the properties of the moulded test specimen. This requirement applies particularly when the specimens have to undergo testing such as for electrical properties, taste, colour or spectroscopic analysis, all which could be affected by release agents. If a release agent is used, it shall be mentioned in the moulding report.

7.6 Breathing

If it is necessary to open the mould for the purpose of breathing, this shall be noted in the moulding report.

8 Procedure

a) Determine the moulding conditions to be used (see Clause 7).

b) Wait until the temperature remains constant to within ± 3 °C.

c) Check the temperature in the cavity using, e.g., a pyrometer or fusible salts (see 4.6).

d) Prepare the charge in accordance with Clauses 6 and 7.

e) Weigh out the quantity of material required to obtain the desired specimen thickness. This quantity corresponds to the product of the test specimen density and the test specimen volume, to which are added flash losses as determined by previous testing.

f) Place the material in the cavity and close the press (4.4). Allow to breathe, if necessary.

NOTE In the case of a programme-controlled press, the breathing and opening operations are automatic.

g) Start the stopwatch (4.5) as soon as the pressure has reached the specified value.

h) When the curing time is completed, open the press (see note). Immediately remove the specimen from the mould and, unless otherwise specified in the test method, place it on a slightly heat-conducting support and allow it to cool down underneath a metal plate used as a cooling fixture (4.8).

i) Check that the moulding is satisfactory as regards filling of the mould, appearance, absence of porosity, discoloration, flash and warping. If necessary, check the density as determined in accordance with ISO 1183.

9 Precision

In the absence of inter-laboratory data, it is impossible to determine the precision of the method. As soon as data are available, a statement will be added at the following revision.

10 Moulding report

The moulding report shall make reference to this International Standard, i.e., ISO 295:2004, and shall contain all the information given in Table 2.

Table 2 — Information to be included in the moulding report

Physical form of material to be moulded			
Pre-treatment	Drying	without	
		time	
		temperature	
	Pelletizing	pressure	
		temperature	
		pellet mass	
		pellet size	
	High-frequency preheating	preheater power	
		time	
		amperage	
		number of pellets	
		temperature of pellets	
	Pre-plastification	cylinder temperature	
		dynamic pressure	
		screw speed	
		temperature of material	
Compression moulding		temperature	
		temperature measurement device	
		pressure	
		cure time	
		breathing	
Mould		type	
		number of cavities	
		chrome plated	
		heating device	

Annex A
(informative)

Marking of test specimens

The purpose of marking is to make it possible

— to differentiate between the test specimen faces;

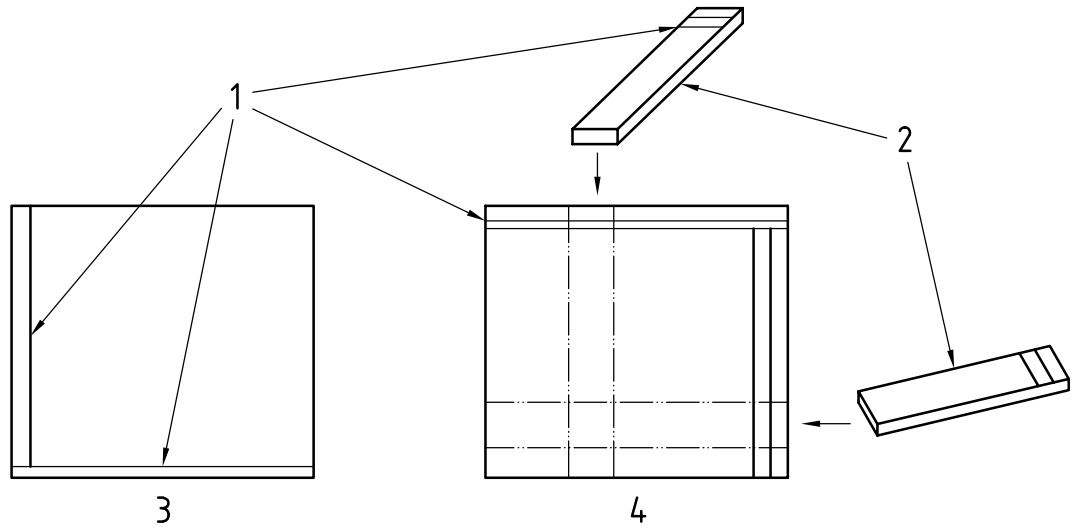
— to identify the direction in which the test specimens were taken from a plate.

Lines engraved parallel and close to the edges of the cavities (and hence the moulded plates) should be used. In the case of multiple cavities, one line indicates cavity No 1, two lines cavity No 2, etc. (see Figure A.1).

Lines should be located close to two edges which are perpendicular to each other, i.e. outside the test specimen test area.

The widths of the perpendicular lines should differ depending on the direction (thus indicating that test specimens marked with thin lines have been taken in one direction and that test specimens marked with broader lines have been taken in the perpendicular direction, thereby avoiding any confusion (see Figure A.1).

The lines should be just visible (i.e. not very deeply engraved) in order to avoid any damage to the cavity surface and sticking at the time of removal of the plate from the mould.



Key

1 marking lines
2 test specimens
3 cavity 1
4 cavity 2

Figure A.1 — Positions of the marking lines

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