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Plastics — Compression moulding test specimens of thermosetting materials

Matières plastiques — Moulage par compression des éprouvettes en matières thermodurcissables

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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 295 was drawn up by Technical Committee ISO/TC 61, *Plastics*. It was submitted directly to the ISO Council, in accordance with clause 6.12.1 of the Directives for the technical work of ISO.

This International Standard cancels and replaces ISO Recommendation R 295-1963, as well as the Annexes A, B and C.

ISO/R 295 had been approved by the Member Bodies of the following countries :

Australia	India	Romania
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The Member Body of the following country had expressed disapproval of the Recommendation :

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Annex A to ISO/R 295 had been approved by the Member Bodies of the following countries :

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Bulgaria	Israel	Spain
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Belgium	Israel	Spain
Brazil	Japan	Sweden
Czechoslovakia	Korea, Rep. of	Switzerland
Egypt, Arab Rep. of	Netherlands	Turkey
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Plastics — Compression moulding test specimens of thermosetting materials

1 SCOPE AND FIELD OF APPLICATION

This International Standard is intended to serve as a basis for ensuring the preparation of equivalent test specimens from identical thermosetting compounds moulded under heat and pressure and the submission of equivalent and comparable statements in the reports issued by different testing organizations. This recommended practice applies only to thermosetting moulding materials such as phenoplasts, aminoplasts and polyesters and epoxy moulding compounds.

This International Standard, therefore, contains a general outline regarding the equipment and the normal methods of preparation of test specimens, as well as the particulars to be included in the reports concerning the preparation of the specimens. Complementary details concerning phenolic aminoplastic, and polyester and epoxy resin moulding materials are given in annexes.

In many cases, special procedures covering the preparation of the test specimens may be necessary depending on composition, flow properties and other variables. Such procedures should be made part of the procurement specifications, or agreed upon between seller and purchaser. Tables of the characteristic properties of test specimens should include reference to such special procedures.

2 APPARATUS

2.1 Compression mould

This moulding tool shall be designed and constructed so that the force of compression is transmitted to the plastics without appreciable losses until the conclusion of the compression cycle. A three-part mould consisting of a shell and an upper and a lower die has been found satisfactory for this purpose. Other moulds may be used, where it can be shown that equivalent results are obtained.

The dimensions of the mould cavity shall be such that the compound can be introduced in one single charge. It may be necessary however, especially for bulky materials, to agree on a method of tableting prior to introduction into the mould.

2.1.1 Ejector pin

In order to ensure the preparation of plane or flat moulded shapes without subsequent distortion, it is preferred that they be ejected from the mould with the entire mould bottom. Ejector pins may be used, if equivalent results can be obtained.

NOTES

1 In order to facilitate the removal of the moulded shapes from the mould, it is permissible to design the mould walls with a draft (taper) of not more than 3°.

2 Pronounced flashing is associated with a correspondingly strong local flow of moulding compound, resulting in test specimens exhibiting partly deformed shapes and deviating properties.

2.1.2 Temperature control device

Moulds shall be provided with efficient heat regulation so that the optimum temperatures required can be maintained constant within a range of $\pm 3^{\circ}\text{C}$ in all sections of the mould; i.e. the mould temperature shall not deviate from the nominal temperature in temporal and spatial respects¹⁾ by more than $\pm 3^{\circ}\text{C}$.

Bore-holes for the introduction of pyrometric or thermometric heat-measuring devices shall be provided in the three main parts of the moulds. The surface of the mould walls shall be polished and may be chromium plated if necessary. The compression face of the lower die shall carry a mark indicating on the moulded shape the surface which has been formed by the lower die. Care shall be taken to ensure that such marks do not interfere with subsequent testing.

NOTES

1 The two main surfaces of test specimens are not exactly equivalent with regard to all their properties, since, during the period between filling and compacting, the surface of the shape directed toward the lower die is heated longer and to a higher temperature than the other surface. It may therefore be advisable in preparing test methods, specifications, property tables, etc., to state the particular surface to which specifications or properties should be referred.

2 Standardization of moulding tools of specific design is in progress.

1) **spatial temperature differences** : Differences of temperature existing simultaneously at various points of the interior of the moulds after the temperature control has been definitely fixed and a state of permanent thermal equilibrium attained.

temporal temperature deviations : Deviations of temperature which may occur at one and the same point of the interior of the mould at different times after the temperature control has been definitely fixed and a state of permanent thermal equilibrium attained.

2.2 Compression moulding press

Any press capable of exerting and maintaining the pressure prescribed for the particular material being moulded for the required time and within the prescribed tolerances is suitable for this purpose.

2.3 Heating device

The moulds shall be heated in such manner as to ensure uniform and constant moulding temperatures within the range of tolerances stated in 2.1.2. They may be heated from press platens and/or heaters inserted in the mould or mould parts, or by heating fluids circulating in suitable passageways in the mould, or by any other convenient means.

3 CONDITIONING

3.1 Unless otherwise required by the material specifications, moulding materials shall be pressed as received.

3.2 In the case of referee tests, unless otherwise agreed, the moulding materials shall be conditioned for 72 h over a desiccant, for example anhydrous calcium chloride, immediately prior to moulding. Where moulding materials are so bulky that conventional moulds have insufficient loading capacity, the materials may be pre-compressed (tabletted). The tableting conditions shall be determined by agreement between seller and purchaser.

4 PROCEDURE

4.1 Mould release agents, i.e. substances designed to facilitate the opening of the moulds, may be employed only if such substances have been proved to exert no influence on the properties of the test specimens produced. This specification applies particularly if the specimens have to be tested for electrical properties or freedom from taste and odor.

4.2 The period of time elapsing between the filling of the mould and the application of pressure shall be as short as possible.

4.3 If, breathing of the mould is necessary, this shall be reported.

4.4 Burrs may be removed from the finished test specimens, but care shall be taken to avoid damaging the moulded skin in any other way. All work of this type shall be carried out in the longitudinal direction of the test specimens. As a rule, the specimens should be tested without further finishing treatments.

4.5 If, in special instances, finishing treatments are required, these shall be subject to mutual agreement between seller and purchaser. Unless otherwise specified in materials specifications, the time from moulding to conditioning shall not be less than 16 h.

5 REPORT

The report shall include the following particulars :

- a) date and location of test specimen preparation;
- b) moulding compound (type, designation, approximate date of manufacture, conditioning treatment, if any);
- c) mould (construction, surface, type of heating);
- d) press (manufacturer's mark, type, capacity);
- e) operating conditions (kind of preliminary treatment, temperature of mould, kinds of temperature-measuring instruments employed, pressure, time and any other procedures of probable significance such as mould ventilation (breathing), etc.).

ANNEX A

PREPARATION OF TEST SPECIMENS FROM PHENOLIC MOULDING MATERIALS

A.1 SCOPE AND FIELD OF APPLICATION

This annex describes the moulds and moulding conditions for compression moulding specimens from phenolic moulding materials, particularly for the following tests :

Impact strength (Charpy)	ISO/R 179
Impact strength (Izod)	ISO/R 180
Flexural strength	ISO 178
Tensile strength	ISO/R 527
Temperature of deflection under load	ISO 75
Incandescence resistance	ISO 181
Water absorption	ISO/R 62 ¹⁾ and ISO/R 117 ¹⁾
Electrical properties	ISO 1325

Where any contradiction arises between this annex and the main body of the document, this annex takes precedence.

A.2 APPARATUS

The moulds shall be positive or semi-positive compression moulds, single or multiple cavity as described in clause 2, except that the walls of the mould may be made with a draft (taper) of not more than 3°, and the clearance at the flash line between the vertical wall of the punch and the wall of the cavity should not normally exceed 0,10 mm at any point around the cavity, although greater clearances are permitted where it can be shown that equivalent results are obtained.

The working surfaces of the mould shall be polished, and may be chromium plated if necessary.

Moulds may be heated from press platens and/or heaters inserted in the mould or mould parts, or by heating fluids circulating in suitable passageways in the mould, or by any other convenient means.

A.3 MOULDING CONDITIONS

All parts of the inner surfaces of the mould shall preferably be maintained at a temperature of 160 ± 3 °C. Recommended moulding pressures are 10 MPa, 25 MPa, or 40 MPa depending upon the plasticity and/or composition of the materials.

If, for particular materials, it is found convenient to use temperatures, pressures, and/or curing times other than those recommended by this method, these may be agreed between seller and purchaser and the conditions used shall be reported in the test report.

The moulding material may be dried or preheated before loading into the mould, but no other conditioning is recommended.

If drying or preheating is carried out, the method, time and temperature must be stated. (See note below.)

A curing time of 1 min per millimetre of thickness is recommended as a minimum unless high-frequency electrical preheating is employed, when a curing time of 30 s per millimetre is permissible.

If the material is pre-compressed (tabletted) it should be preferably in the form of a single unbroken tablet for each cavity. (See note below.)

Breathing of the mould is permissible during the moulding operation.

NOTE — Except for electrical test specimens, high-frequency electrical preheating may be employed to reduce the curing time. When high-frequency electrical preheating is employed, a single tablet may not be feasible and is not essential because the preheated charge, being plastic, is unlikely to cause weld lines. For electrical test specimens, it is recommended that the mould charge in powder form be spread in an even layer not more than 13 mm thick on a sheet of cardboard and be preheated in a circulating-air oven for either 30 min at 90 ± 3 °C or 15 min at 105 ± 3 °C. The preheated material should be moulded immediately after being removed from the oven. Preheating in this manner is recommended to minimize test variability caused by moisture in the material and, while recommended particularly for electrical test specimens, it may be used for other test specimens as well.

1) At present under revision for publication as an International Standard.

ANNEX B

PREPARATION OF TEST SPECIMENS FROM AMINOPLASTIC MOULDING MATERIALS

B.1 SCOPE AND FIELD OF APPLICATION

This annex describes the moulds and moulding conditions for compression moulding specimens from aminoplastic moulding materials (consisting of aminoplastic resins, derived from the reaction of urea, thiourea or melamine and allied compounds with aldehydes, usually formaldehyde), particularly for the following tests :

Impact strength (Charpy)	ISO/R 179
Impact strength (Izod)	ISO/R 180
Flexural strength	ISO 178
Tensile strength	ISO/R 527
Temperature of deflection under load	ISO 75
Incandescence resistance	ISO 181
Water absorption	ISO/R 62 ¹⁾ and ISO/R 117 ¹⁾
Electrical properties	ISO 1325

usual ranges for moulding pressures and curing times :

Moulding materials	Moulding temp- erature	Moulding pressure	Curing time per millimetre of thickness
	°C	MPa	s
Urea (and thiourea) materials with cellulose and woodflour fillers	140 to 150	15 to 35	30 to 60
Melamine materials with cellulose, wood-flour or mineral fillers	150 to 160	15 to 35	30 to 60
Melamine materials with coarse fibrous filler	150 to 160	30 to 50	30 to 60

Where any contradiction arises between this annex and the main body of the document, this annex takes precedence.

If, for particular materials, it is found convenient to use temperatures, pressures and/or curing times other than those recommended in the table above, these may be agreed between seller and purchaser and the conditions used shall be reported in the test report.

B.2 APPARATUS

The moulds shall be positive or semi-positive compression moulds, single or multiple cavity, as described in clause 2, except that the walls of the mould may be made with a draft (taper) of not more than 3°, and the clearance at the flash line between the vertical wall of the punch and the wall of the cavity should not normally exceed 0,10 mm at any point around the cavity, although greater clearances are permitted where it can be shown that equivalent results are obtained.

The working surfaces of the mould shall be polished and may be chromium plated if necessary.

Moulds may be heated from press platens and/or heaters inserted in the mould or mould parts, or by heating fluids circulating in suitable passageways in the mould, or by any other convenient means.

The moulding material may be dried or preheated before loading into the mould, but no other conditioning is recommended.

If drying or preheating is carried out, the method, time and temperature must be stated. (See note below.)

In the case of preheating, a curing time of 20 to 30 s per millimetre of thickness is permissible.

If the material is pre-compressed (tabletted) it should be preferably in the form of a single unbroken tablet for each cavity. (See note below.)

Breathing of the mould is permissible during the moulding operation.

NOTE — Except for electrical test specimens, high-frequency electrical preheating may be employed to reduce the curing time. When high-frequency electrical preheating is employed, a single tablet may not be feasible and is not essential because the preheated charge, being plastic, is unlikely to cause weld lines. For electrical test specimens, it is recommended that the mould charge in powder form be spread in an even layer not more than 10 mm thick on a sheet of cardboard and be preheated in a circulating air oven for between 30 and 60 min at a temperature between 80 and 90 °C. The preheated material should be moulded immediately after being removed from the oven. Preheating in this manner is recommended to minimize test variability caused by moisture in the material and, while recommended particularly for electrical test specimens, it may be used for other test specimens as well.

B.3 MOULDING CONDITIONS

All parts of the inner surfaces of the mould shall be maintained within ± 3 °C of the moulding temperature appropriate to the material in question, within the range stated in the following table, which also comprises the

1) At present under revision for publication as an International Standard.

ANNEX C

PREPARATION OF TEST SPECIMENS FROM POLYESTER AND EPOXY RESIN MOULDING MATERIALS

C.1 SCOPE AND FIELD OF APPLICATION

This annex describes the moulds and moulding conditions for compression moulding specimens from polyester or epoxy resin moulding materials, particularly for the following tests :

Impact strength (Charpy)	ISO/R 179
Impact strength (Izod)	ISO/R 180
Flexural strength	ISO 178
Tensile strength	ISO/R 527
Temperature of deflection under load	ISO 75
Incandescence resistance	ISO 181
Water absorption	ISO/R 62 ¹⁾ and ISO/R 117 ¹⁾
Electrical properties	ISO 1325

Where any contradiction arises between this annex and the main body of the document, this annex takes precedence.

C.2 APPARATUS

The moulds shall be positive or semi-positive compression moulds, single or multiple cavity as described in clause 2, except that the walls of the mould may be made with a draft (taper) of not more than 3°, and the clearance at the flash line between the vertical wall of the punch and the wall of the cavity should not normally exceed 0,10 mm at any point around the cavity, although greater clearances are permitted where it can be shown that equivalent results are obtained.

The working surfaces of the mould shall be polished and may be chromium plated if necessary.

Moulds may be heated from press platens and/or heaters inserted in the mould or mould parts, or by heating fluids circulating in suitable passageways in the mould, or by any other convenient means.

C.3 MOULDING CONDITIONS

All parts of the inner surfaces of the mould shall be maintained within ± 3 °C of the moulding temperature appropriate to the material in question, within the range stated in the following table which also comprises the

usual ranges for moulding pressures, and curing times :

Moulding materials	Moulding temperature	Moulding pressure	Curing time per millimetre of thickness
	°C	MPa	s
Polyester resin materials	130 to 160	5 to 30	30 to 45
Epoxy resin materials	150 to 170	5 to 30	35 to 60

If, for particular materials, it is found convenient to use temperatures, pressures and/or curing times other than those recommended in the table above, these may be agreed between seller and purchaser and the conditions used shall be reported in the test report.

The materials should generally be used in the condition as received.

Because of the risk of precurcuring, drying or preheating should ordinarily be avoided, but is not prohibited.

If granulated (non-putty) material is precompressed (tabletted), it must be in the form of a single unbroken tablet for each cavity.

No other conditioning is recommended.

NOTES

1 The conditions of storing (time, temperature, humidity, etc.) of the moulding material before moulding the test specimens shall be agreed between seller and purchaser to ensure the preparation of test specimens within the shelf life of the compound.

2 To avoid premature curing before flow takes place, the mould shall be closed quickly. After the material has been loaded into the mould, no more than 20 s shall elapse before the top force is in contact with it (as indicated by deflection of the gauge for measuring pressure). However, the mould shall be closed uniformly and with care to avoid entrapment of air and to prevent excessive loss of material as flash which could cause the test specimen to be too thin. To ensure that test specimens of the required thickness are obtained regularly, it is recommended that appropriate spacers or mould stops be used and that the required mould charge be calculated by multiplying the volume of the test specimen by the density of the moulded material, plus an excess to compensate for flash loss. This excess is usually about 5 %, but should be determined experimentally to ensure consistency of dimensions when moulding a series of test specimens.

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