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Plastics — Injection moulding of test specimens of thermoplastic materials —

Part 4:

Determination of moulding shrinkage

Plastiques — Moulage par injection des éprouvettes de matériaux thermoplastiques —

Partie 4: Détermination du retrait au moulage



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ISO 294-4:2001(E)

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 3.

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 part of ISO 294 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 294-4 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This second edition cancels and replaces the first edition (ISO 294-4:1997), which has been technically revised.

ISO 294 consists of the following parts, under the general title *Plastics* — *Injection moulding of test specimens of thermoplastic materials*:

- Part 1: General principles, and moulding of multipurpose and bar test specimens
- Part 2: Small tensile bars
- Part 3: Small plates
- Part 4: Determination of moulding shrinkage
- Part 5: Preparation of standard specimens for investigating anisotropy

Annex A of this part of ISO 294 is for information only.

Introduction

See ISO 294-1.

In the injection moulding of thermoplastics, the difference between the dimensions of the mould cavity and those of the moulded articles produced from it may vary with the design and operation of the mould. Such differences may depend on the size of the injection-moulding machine, the shape and dimensions of mouldings including any restrictive action this may have on the shrinkage, the degree and direction of flow or movement of the material in the mould, the sizes of the nozzle, sprue, runner and gate, the cycle on which the machine is operated, the temperature of the melt and the mould, and the magnitude and duration of the hold pressure. Moulding and post-moulding shrinkage are caused by crystallization, volume relaxation and orientation relaxation of the material and by thermal contraction of both the thermoplastic material and the mould. Post-moulding shrinkage may also be influenced by humidity uptake.

The measurement of moulding and post-moulding shrinkage is useful in making comparisons between thermoplastics and in checking uniformity of manufacture.

The method is not intended as a source of data for design calculations of components. Information on the typical behaviour of a material can be obtained, however, by carrying out measurements at different melt and mould temperatures, injection velocities and hold pressures, as well as at different values of other injection-moulding parameters. The information thus obtained is important in establishing the suitability of the moulding material for the production of moulded articles with accurate dimensions.

Plastics — Injection moulding of test specimens of thermoplastic materials —

Part 4:

Determination of moulding shrinkage

1 Scope

This part of ISO 294 specifies a method of determining the moulding shrinkage and post-moulding shrinkage of injection-moulded test specimens of thermoplastic material in the directions parallel to and normal to the direction of melt flow.

For the determination of shrinkage of thermosets see ISO 2577^[2].

Moulding shrinkage as defined in this part of ISO 294 excludes the effects of humidity uptake. This is included in post-moulding shrinkage and thus in total shrinkage. For cases when post-moulding shrinkage is caused by the uptake of humidity only, see ISO 175^[1].

Moulding shrinkage as defined in this part of ISO 294 represents the so-called free shrinkage with unrestricted deformation of the cooling plates in the mould during the hold period. It may be considered therefore as the maximum value of any restricted shrinkage.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 294. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 294 are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 294-1:1996, Plastics — Injection moulding of test specimens of thermoplastic materials — Part 1: General principles, and moulding of multipurpose and bar test specimens

ISO 294-3:—1), Plastics — Injection moulding of test specimens of thermoplastic materials — Part 3: Small plates

¹⁾ To be revised. (Revision of ISO 294-3:1996)

3 Terms and definitions

For the purposes of this part of ISO 294, the terms and definitions given in ISO 294-1 together with the following apply.

3.1

moulding shrinkage

 S_{M}

difference in dimensions between a dry test specimen and the mould cavity in which it was moulded, both the mould and the test specimen being at room temperature when measured

NOTE 1 It is expressed as a percentage (%) of the mould cavity dimension concerned.

NOTE 2 The moulding shrinkage S_{Mp} parallel to the melt flow direction is determined at the mid-point of the width of the test specimen, and the moulding shrinkage S_{Mn} normal to the flow direction at the mid-point of the length.

3.2

post-moulding shrinkage

 S_{P}

difference in the dimensions of a moulded test specimen before and after a post-moulding treatment, measured at room temperature

NOTE 1 It is expressed in percent (%).

NOTE 2 The post-moulding shrinkage S_{Pp} parallel to the melt flow direction and the post-moulding shrinkage S_{Pn} normal to the flow direction are defined in analogous fashion to S_{Mp} and S_{Mn} in 3.1.

3.3

total shrinkage

 S_{T}

difference in dimensions between a test specimen after a post-moulding treatment and the mould cavity in which it was moulded, measured at room temperature

NOTE 1 It is expressed in percent (%).

NOTE 2 The total shrinkage $S_{\rm Tp}$ parallel to the melt flow direction and the total shrinkage $S_{\rm Tn}$ normal to the flow direction are defined in analogous fashion to $S_{\rm Mp}$ and $S_{\rm Mn}$ in 3.1.

3.4

cavity pressure

 p_{C}

pressure of the thermoplastic material in the cavity at any time during the moulding process, measured centrally near the gate

NOTE It is expressed in megapascals (MPa).

3.5

cavity pressure at hold

 p_{CH}

cavity pressure (3.4) 1 s after the end of the injection time t_1 (see Figure 1)

NOTE It is expressed in megapascals (MPa).

4 Apparatus

4.1 Type D2 ISO mould, giving $60 \text{ mm} \times 60 \text{ mm} \times 2 \text{ mm}$ plate specimens, as specified in ISO 294-3:—, subclause 4.1.

Reference marks may be engraved in the mould cavity to facilitate the measurement of the dimensions of the test specimens produced from the mould using optical techniques. Such reference marks, if used, shall be located at a distance of (4 ± 1) mm from the edges of the mould cavity.

It is recommended that such marks be at most $5 \mu m$ in depth in order to ensure that they do not restrict the shrinkage process in any way (see Introduction). Pins inserted in the correct plane have also been used successfully.

Installation of a pressure sensor P, recommended for parts 1 to 3 of this International Standard [see ISO 294-1:1996, 4.1.1.4, item k) and ISO 294-3:—, Figure 2], is mandatory for shrinkage measurements.

The mould plates used shall be rigid enough to avoid the moulded plates being thicker than the depth of the cavity, for the whole range of hold pressures that result in positive shrinkage in length or width.

4.2 Injection-moulding machine, in accordance with 4.2 of ISO 294-3:—, but adding the following tolerance limits to the list of operating conditions given in 4.2.2 of ISO 294-1:1996:

Cavity pressure, $p_{\rm C}$ \pm 5 %

4.3 Measuring equipment, capable of measuring the length and width of each test specimen and of the mould cavity to within 0,02 mm, the measurements being made between the centres of opposite sides or between the opposite edges or between pairs of reference marks (see annex A). When measuring the length of a test specimen, take care to include the 0,5-mm-high step at the gate end of the specimen. If a mechanical instrument is used, ensure that the jaws of the instrument do not produce a significant indentation.

It is recommended that a calibration plate be used to periodically check the measuring equipment.

4.4 Oven, necessary only if post-moulding shrinkage is to be measured, by agreement between the interested parties.

5 Procedure

5.1 Conditioning of material

As specified in ISO 294-1:1996, 5.1.

5.2 Injection moulding

- **5.2.1** For the basic injection-moulding conditions, see ISO 294-3:—, 5.2.
- **5.2.2** The moulding shrinkage is preferably determined for one or more values of the cavity pressure at hold $p_{\rm CH}$ (see 3.5) selected from 20 MPa, 40 MPa, 60 MPa, 80 MPa and 100 MPa. Intermediate values may also be used, however.

NOTE For values higher than 80 MPa, a correspondingly high locking force will be necessary, and this may not be possible with normal commercial equipment.

- **5.2.3** Determine the hold pressure $p_{\rm H}$ which corresponds to each selected value of $p_{\rm CH}$ and mould test specimens at each of these pressures, taking into account of the following additional instructions.
- a) Select the change-over point, between the injection and hold periods carefully to avoid a depression in the time against pressure curve (see Figure 1, Curve c) and to avoid a peak that, during the 1 s following the change-over point, exceeds the cavity pressure at hold by more than 10 % (see Figure 1, Curve b).

Due to the inertia of the injection-moulding machine, the effective change-over time is longer than its nominal value. The correct change-over point shall therefore be adjusted individually for each value of the injection speed and for each material under test.

NOTE Peaks in the cavity pressure lead to transient overloading of the cavity, followed by partial backflow of the melt. Thus the mass of material injected into the cavity is not clearly defined and the orientation of the material near the gate will be perturbed.

- b) Keep the hold pressure constant during the hold period.
- c) For the hold time, see 5.2.4 of ISO 294-1:1996. The decrease in the cavity pressure at hold to zero indicates that the material in the gate has solidified sufficiently to stop flow into the cavity.
- d) Select the cooling time to be the minimum value at which the mouldings can be removed from the mould without distortion. As the cooling rate of the material is proportional to the square of the reciprocal of the thickness, the minimum cooling time (for the cavity) can be expected to be close to 1,8 times the hold time (cooling time for the gate) for the gate height to plate thickness ratio of 3:4 in ISO 294-3:—.
- e) For the maintenance of steady-state conditions, see 5.2.5 of ISO 294-1:1996.

The change in curvature near A in Figure 1 indicates the transition from the melt-flow period to the bulk-compression period. At point R, the value of the cavity pressure at hold is recorded. The minimum hold time can be read from the decrease of the cavity pressure to zero.

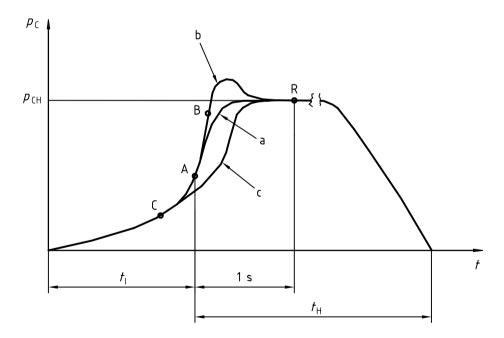


Figure 1 — Schematic plot of cavity pressure versus time showing the influence of the injection time when selected correctly (near point A, resulting in Curve a), when too late (e.g. at point B, resulting in Curve b) and when too early (e.g. at point C, resulting in Curve c)

5.3 Measurement of mould temperature

As specified in ISO 294-1:1996, 5.3.

5.4 Measurement of melt temperature

As specified in ISO 294-1:1996, 5.4.

5.5 Treatment of test specimens after demoulding

- **5.5.1** In order to minimize warpage, cut each test specimen from the runner immediately after demoulding. Take care that the sides which will be used for the measurement of the dimensions are not damaged during the cutting operation.
- **5.5.2** Allow the test specimens to cool to room temperature by placing them flat on a thermally non-conducting surface. After cooling, store them at a temperature of (23 ± 2) °C for between 16 h and 24 h. If a material shows a marked difference in moulding shrinkage when stored in humid and dry atmospheres, store specimens made of such material in a dry atmosphere (e.g. in an airtight box together with desiccant).

5.6 Measurement of moulding shrinkage

- **5.6.1** Check by comparing the plate thickness, especially near the gate at mid-width, with the cavity depth that the mould plates used have sufficient rigidity (see 4.1).
- **5.6.2** If not already known, measure, at a temperature of (23 ± 2) °C, to the nearest 0,02 mm, the length $l_{\rm C}$ and width $b_{\rm C}$ of the cavity between appropriate reference points on opposite sides. These may be the centres of the sides and of the step at the gate end, the centres of the edges, or reference marks engraved in the mould cavity (see annex A).

Record these measurements for use in the calculation of the shrinkage.

NOTE From time to time, it is advisable to check the reference marks engraved in the mould cavity for wear.

- **5.6.3** Before measuring the dimensions of a specimen, place it on a flat surface or against a straight edge in order to determine any warpage. Discard any specimen for which the warpage exceeds 2 mm in height (i.e. out-of-plane deformation).
- **5.6.4** Measure, at a temperature of (23 ± 2) °C, to the nearest 0,02 mm, the length l_1 and width b_1 of the specimen between reference points corresponding to those used for the mould cavity (see 5.6.2).

Minor warpage (less than 2 mm) may be reduced by compressing to give a flat surface. During measurement of the dimensions, any warpage shall be less than 1 mm.

NOTE Warpage decreases a dimension in accordance with the following approximate equation:

$$\Delta x \approx h^2/3x$$
 (1)

where

 \hat{x} is the magnitude, in millimetres, of the dimension (length l or width b);

 $-\Delta x$ is the decrease in magnitude, in millimetres, of the dimension (length l or width b);

h is the warpage height (out-of-plane deformation), in millimetres.

For a magnitude x of 60 mm and a warpage height h of 1 mm for instance, the decrease in x is 0,02 mm, which corresponds to the tolerance limit given in 5.6.2 and 5.6.4.

5.6.5 Carry out the measurements using at least five test specimens for each set of moulding conditions.

5.7 Treatment following measurement of moulding shrinkage

The conditions of treatment (temperature, humidity or other environments) for the time period between the measurement of moulding shrinkage and the measurement of post-moulding shrinkage shall be as specified in the relevant material standard or as agreed between the interested parties.

NOTE The conditions of post-moulding treatment may reflect those of storage or those of use.

5.8 Measurement of post-moulding shrinkage

After the post-moulding treatment, measure the test specimens again, at a temperature of (23 ± 2) °C, to the nearest 0,02 mm (see 5.6.3 to 5.6.5) and record the new length l_2 and width b_2 .

6 Expression of results

6.1 Moulding shrinkage

The moulding shrinkage S_{Mp} parallel to the melt flow direction and the moulding shrinkage S_{Mn} normal to the flow direction are given as a percentage by the following equations:

$$S_{\rm Mp} = 100 \, \frac{l_{\rm C} - l_{\rm 1}}{l_{\rm C}} \tag{2}$$

$$S_{\rm Mn} = 100 \, \frac{b_{\rm C} - b_{\rm 1}}{b_{\rm C}} \tag{3}$$

where

 $l_{\rm C}$ and $b_{\rm C}$ are the length and width, in millimetres, across the centre of the cavity (see 5.6.2);

 l_1 and l_2 are the corresponding length and width, in millimetres, of the test specimen (see 5.6.4).

6.2 Post-moulding shrinkage

The post-moulding shrinkage S_{Pp} parallel to the melt flow direction and the post-moulding shrinkage S_{Pn} normal to the flow direction are given as a percentage by the following equations:

$$S_{\mathsf{Pp}} = 100 \, \frac{l_1 - l_2}{l_1} \tag{4}$$

$$S_{\mathsf{Pn}} = 100 \, \frac{b_1 - b_2}{b_1} \tag{5}$$

where l_2 and b_2 are the length and width, in millimetres, of the test specimen after the post-moulding treatment (see 5.8).

6.3 Total shrinkage

The total shrinkage S_{Tp} parallel to the melt flow direction and the total shrinkage S_{Tn} normal to the flow direction are given as a percentage by the following equations:

$$S_{\rm Tp} = 100 \, \frac{l_{\rm C} - l_2}{l_{\rm C}} \tag{6}$$

$$S_{\rm Tn} = 100 \, \frac{b_{\rm C} - b_2}{b_{\rm C}} \tag{7}$$

where the symbols are as defined above.

Moulding, post-moulding and total shrinkage, expressed as a percentage, are related to each other by the following equation:

$$S_{\rm T} = S_{\rm M} + S_{\rm P} - \frac{S_{\rm P} S_{\rm M}}{100} \tag{8}$$

Moulding and post-moulding shrinkage are not expressed as a percentage of the same starting dimension [see equations (2) and (3), and equations (4) and (5), respectively]. The total shrinkage, therefore, is not exactly the sum of the moulding and post-moulding shrinkage. The last term in equation (8) can be ignored, however, because it is two orders of magnitude smaller than the other terms.

7 Precision

The precision of this test method is not known because interlaboratory data are not available. When interlaboratory data are obtained, a precision statement will be added at the following revision.

8 Test report

The test report shall include the following information:

- a) a reference to this part of ISO 294, i.e. ISO 294-4;
- b) to h) see ISO 294-1:1996, clause 6, items b) to h), but replacing, in item g), the hold pressure $p_{\rm H}$ by the cavity pressure at hold $p_{\rm CH}$;
- i) the moulding, post-moulding and total shrinkage, parallel and normal to the melt flow direction, expressed as a percentage to the nearest 0,1 %.

Annex A

(informative)

Reference points for length and width measurement

Figure A.1 shows a perspective view of a test specimen with, in the foreground, the step at the gate end and the section (hatched), through which it has been cut from the runner.

For measurements made using a mechanical instrument of the lengths l_1 and l_2 and the widths b_1 and b_2 , the centres S of the three moulded sides of the specimen and the centre G of the gate step are suitable reference points.

For optical measurements, the centres E of the moulded edges may be used, or marks M produced by the mould cavity plate at a distance of (4 ± 1) mm from the edges (see 4.1) (Figure A.1 shows one such mark only).

The difference in height between a pair of reference points has no significant influence on the determination of the shrinkage provided the same reference points are used to measure the cavity as are used to measure the specimen. This correspondence avoids any effect due to the draft angle and allows different types of reference point to be used to measure a dimension, e.g. a "mechanical" reference point S on one side combined with an "optical" reference point E or M on the opposite side.

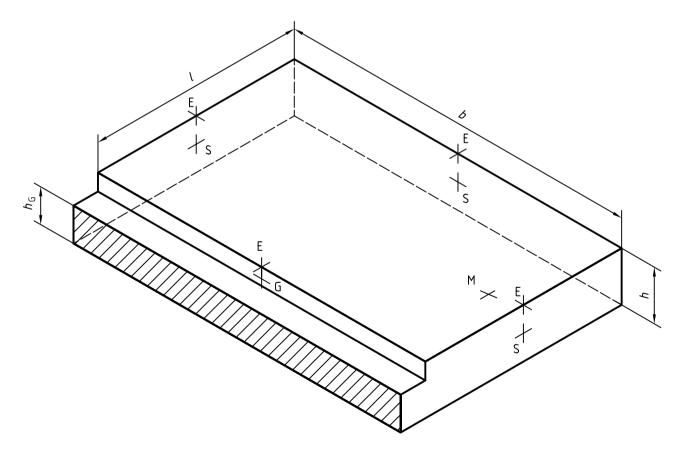


Figure A.1 — Perspective view of an injection-moulded plate specimen

Bibliography

- [1] ISO 175:1999, Plastics Methods of test for the determination of the effects of immersion in liquid chemicals
- [2] ISO 2577:1984, Plastics Thermosetting moulding materials Determination of shrinkage

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