INTERNATIONAL STANDARD ISO 2557

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION • МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ • ORGANISATION INTERNATIONALE DE NORMALISATION

Plastics — Amorphous thermoplastic moulding materials — Preparation of test specimens with a defined level of shrinkage

Matières plastiques — Matières à mouler thermoplastiques amorphes — Préparation d'éprouvettes à niveau défini de retrait

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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 2557 was drawn up by Technical Committee ISO/TC 61, *Plastics*, and circulated to the Member Bodies in July 1973.

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

Australia	Hungary	South Africa, Rep. of
Austria	Israel	Sweden
Belgium	Italy	Thailand
Bulgaria	Japan	United Kingdom
Czechoslovakia	Netherlands	U.S.A.
Egypt, Arab Rep. of	New Zealand	Yugoslavia
France	Poland	
Germany	Romania	

No Member Body expressed disapproval of the document.

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[New title :]

Plastics — Amorphous thermoplastic moulding materials — Preparation of test specimens with a defined level of shrinkage — Part 1 : Test specimens in the form of parallelepipedic bars (Injection moulding and compression moulding)

Plastiques — Matières à mouler thermoplastiques amorphes — Préparation d'éprouvettes à niveau défini de retrait — Partie 1 : Éprouvettes sous forme de barreaux parallélépipédiques (Moulage par injection et moulage par compression)

ERRATUM

Cover page and page 1

Amend the title as shown above.

All pages

Replace the reference number "2557" by "2557/1".

Plastics — Amorphous thermoplastic moulding materials — Preparation of test specimens with a defined level of shrinkage

0 INTRODUCTION

The properties of test specimens of thermoplastic materials are influenced by their molecular orientation. The degree and direction of such orientation are dependent upon the methods used to prepare the specimens. The amount of orientation induced during processing can be assessed by measuring the shrinkage of the specimens at an elevated temperature. Reproducible test results can best be obtained by using test specimens which are in the same state of orientation. For industrial purposes, the conditions of similar state are reasonably fulfilled when the measured shrinkages of test specimens produced by a specified heat treatment are equal.

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies procedures for the preparation of test specimens with a defined level of shrinkage which is measured by determining the longitudinal shrinkage of a sample of the specimens by a standardized test method. The method is suitable only for specimens exhibiting uniaxial orientation, i.e. specimens in the form of bars and made from rigid amorphous thermoplastics other than those containing fibre reinforcements.

By modifying the conditions of the moulding operation for the mould and machine used, test specimens with a defined level of shrinkage and, consequently, in the same state of molecular orientation can be prepared. Those conditions need to be determined for each type of moulding material. Unless otherwise agreed, the choice between determining partial or maximum shrinkage shall be decided by the material specification which shall also define the required level of shrinkage and the conditions of its determination.

2 DEFINITIONS

For the purposes of this International Standard, the following definitions apply :

2.1 state (of a specimen) : The condition of a specimen as characterized by its shrinkage.

2.2 defined state (of specimens) : The state in which the specimens have a defined level of shrinkage which is either specified for the material under consideration or established by agreement.

2.3 basic state (of specimens) : The state in which the specimens are considered to be if they do not shrink more than 2 % in length when tested as specified in 4.3 and if, after that treatment, they are not changed significantly either in appearance or in mechanical properties.

2.4 shrinkage : The percentage change in length measured as defined in 4.2 when the specimen is subject to a specified heat treatment.

$$S\,\% = \frac{l_0 - l_1}{l_0} \times 100$$

where

S is the shrinkage;

l_0 is the original length;

l_1 is the length after shrinkage;

2.4.1 maximum shrinkage (S_m) : The shrinkage determined at a relatively high temperature. Its value is not influenced by small changes of time or temperature.

2.4.2 partial shrinkage (S_p) : The shrinkage measured under less extreme, but specified, conditions; for example VST/B + 20 °C, 45 min.[1)]

S_p is influenced by the temperature and the time of treatment ($S_p < S_m$).

NOTE — The temperature used for the heat treatment should be above the glass transition temperature of the material.

1) For convenience, the temperature chosen may be related to the Vicat softening temperature, method B (VST/B, see ISO 306, *Plastics — Determination of the Vicat softening temperature of thermoplastics)*.

3 PREPARATION OF TEST SPECIMENS

3.1 Apparatus

3.1.1 *Injection moulds*

Single-cavity moulds are recommended. If multi-cavity moulds are used, the cavities must be similar and should be fed in parallel and not in series. The sprues and runners should be as short as possible and the gate located at one end of the specimen. Gate diameters should be as large as possible and so selected that a balance of shrinkages between specimens from all of the cavities may be attained.

NOTE – Bending of specimens during shrinkage is due to an asymmetric distribution of stresses and orientation. The difficulty can be obviated by careful mould construction, in particular balanced cooling.

3.1.2 *Compression moulds*

The mould assembly is illustrated in figures 1 and 2. It consists of a multi-cavity mould, two metal plates, and 0,1 mm foils made of soft aluminium. The mould shall be made of tool steel or stainless steel.

For the moulding of sheets, the assembly of the mould is the same except that a "picture-frame" type of mould is used instead of a multi-cavity mould.

NOTE – Foils are helpful for equalizing cooling and in dissipating volatile material which could cause bubbles.

3.2 Procedure

3.2.1 *Injection-moulded specimens*

The orientation, and consequent shrinkage on heating, of injection-moulded specimens is a complicated function of many variables. To obtain minimum orientation and shrinkage requires high melt temperatures and/or low rates of flow, coupled with slow cooling.

To obtain specimens with a defined level of shrinkage, a trial may first be made at conditions stated in the specification for the material or recommended by the manufacturer. Sufficient mouldings should be made to attain steady conditions (determined for instance by mass or dimensions of the mouldings) before making the required batch of test specimens, which should then be sampled and tested for shrinkage in accordance with clause 4.

If the required level of shrinkage is not obtained, then moulding conditions may be adjusted in accordance with the principles given above, taking care to attain steady conditions before repeating the determination of shrinkage.

The need to experimentally determine the moulding conditions required to produce mouldings with a defined level of shrinkage may be minimized by establishing a correlation between cylinder temperature and shrinkage. For a particular injection machine and mould, a given material may be moulded at several cylinder temperatures within the range recommended for it while maintaining all other machine control settings constant. Shrinkages of the resultant specimens determined in accordance with 4.2 are then plotted versus cylinder temperatures as shown in figure 3 and a curve is drawn. Subsequently, when the same kind of material is to be moulded with that particular machine and mould, the cylinder temperature expected to produce the desired level of shrinkage can be read off the curve usually without need for additional experimentation.

Alternatively, another processing variable such as injection speed (injection pressure) may be correlated with shrinkage, while maintaining other machine control settings constant. The injection speed required to produce a desired shrinkage value may then be estimated.

3.2.2 *Compression-moulded specimens*

The specimens shall be compression moulded direct to the required shape or be machined from moulded sheet (see 3.1.2). The time, temperature and pressure and the quantity of the granules or plastic powder used shall conform to the directions given in the specification of the moulding material. If there are no recommendations, the amount of material shall be such that the mould cavity is completely filled after moulding and a minimum of flash is formed.

NOTE – Pre-compaction by milling or extrusion may also be used.

3.2.3 *Specimens in a basic state*

If specimens in the basic state are required, they may be prepared by compression moulding (see 3.2.2). Alternatively, injection-moulded specimens may be placed in a mould cavity (the dimensions of which are identical to those of the injection-mould) and placed between plates (and foils) as shown in figure 1. The assembly is then heated and cooled in a press under conditions for compression moulding given in the specification for the material in question. In that way, orientation present in injection-moulded specimens may be released.

The specimens shall conform to the requirements specified in 2.3.

4 DETERMINATION OF SHRINKAGE

4.1 Apparatus

4.1.1 *Specimen holder* (conditioning chamber)

The specimen holder must have a high thermal capacity and a small working volume so that the temperature is kept constant to within ± 2 °C during the insertion and removal of test specimens.

The specimen holder consists of two metal blocks separated by two spacers – for example 13 mm for specimens with a maximum thickness of 6,35 mm; 25 mm for specimens with a maximum thickness of 12,7 mm – with a metal back plate 5 mm thick.

The test specimens are placed on an aluminium sheet which just fits in notches in the spacers and projects from the specimen holder when in position (see figure 4). The whole assembly is mounted in a suitable oven; an asbestos sheet is placed in front of the assembly to avoid a fall in temperature when the oven door is opened. The asbestos sheet has a slot to allow the aluminium sheet, with specimens, to be inserted or withdrawn (see figure 5).

4.1.2 *Liquid bath*

The bath must be large enough so as not to interfere with the shrinkage of the specimens and shall be filled with a liquid which is inert with respect to the test specimen material. The liquid must also be less dense than the specimen material. The temperature of the bath shall be kept constant to within ± 2 °C with adequate agitation. It is recommended that the test specimens be placed in the bath on a smooth support of a material which has no deleterious effect on either the specimen or the liquid.

4.2 Test specimen

Unless otherwise stated in the specification for the material, whole specimens shall not be used for the determination of shrinkage. They shall be reduced to a length of 30 mm by trimming a similar portion from both ends. The length of the centre lines of the two opposite sides shall be measured, at room temperature, to within ± 0,1 mm and the mean recorded as the initial length, l_0.

Five test specimens shall be used for each determination.

NOTE — In evaluating the results for specimens longer than 30 mm, it must be borne in mind that the orientation, and therefore the shrinkage, may vary along the specimens. For this reason, the overall shrinkage may be less than that measured on the 30 mm test portion.

4.3 Determination of maximum shrinkage

For determining maximum shrinkage, the test specimens shall be placed on the aluminium sheet, which has been coated with a mixture consisting of 20 parts talc and 80 parts silicone oil[1]. The assembly is then placed in the specimen holder (4.1.1) fot the specified period of time. The specimen holder shall be at the temperature given in the specification for the relevant type of material or as otherwise agreed.

After the specified time, the test specimens, while still on their support, shall be removed from the specimen holder and allowed to cool in still air, the support resting on a heat-insulating surface. The final length of the test specimens shall be measured after they have reached room temperature. The length of the centre lines of the two opposite sides shall be measured to within ± 0,1 mm and the mean recorded as the final length l_1 (see figure 6).

NOTE — Owing to the decrease of orientation in the specimen from the surface to the centre, the latter shrinks less; thus a protuding nose occurs as outlined in figure 6. This nose has to be neglected for the determination of l_1.

1) Silicone oil 200 mPa·s, stabilized.

4.4 Determination of partial shrinkage

For determining partial shrinkage, the test specimens shall be placed on supports in the liquid bath and kept at the required temperature for the specified period of time in the liquid bath preheated to the temperature given in the specification for the relevant type of material or as otherwise agreed.

NOTE — The liquid bath is used to provide good heat transfer where the shrinkage is time and temperature dependent.

After the specified time, the test specimens, while still on their support, shall be removed from the bath and allowed to cool in still air, the support resting on a heat-insulating surface. The final length of the test specimens shall be measured after they have reached room temperature, as specified in 4.3.

4.5 Precision of measurement

The percentage shrinkage for the individual specimens should not differ by more than 1 to 2 %, the higher difference relating to a group of specimens with relatively high shrinkages. If greater differences are found, it is necessary to examine both the moulding procedure and the test conditions to find and hence eliminate the cause.

5 REPORT

The report shall include the following particulars :

a) the moulding material (type, designation);

b) the dimensions of the test specimens;

c) details of the preparation of the specimens :

1) for injection-moulded specimens :

type of mould and moulding conditions (relevant to temperature and pressure);

2) for compression-moulded specimens :

moulding temperature, pressure, time and cooling conditions;

3) *for relaxation in a mould :*

moulding temperature, pressure, time and cooling conditions;

d) all details of the shrinkage testing of the specimens, including the length of the test bars before and after shrinkage, the shrinkage expressed as a percentage (individual and average rounded to the nearest whole number), the test temperature, test period and, if a liquid bath is used, the immersion medium;

e) other relevant details.

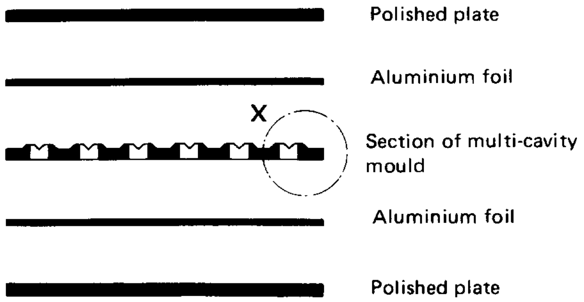
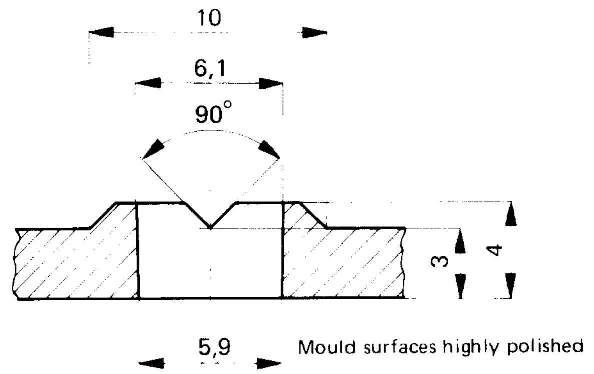
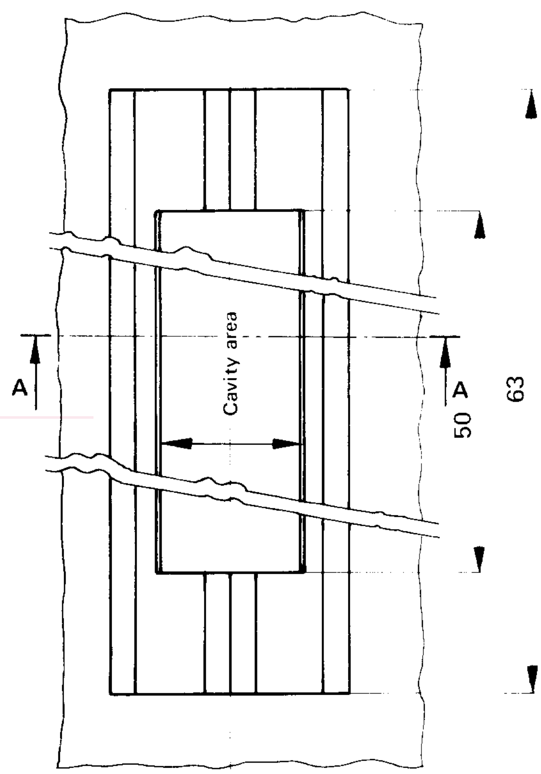


FIGURE 1 – **Schematic diagram of a mould for compression moulding (3.2.2) and relaxation (3.2.3)**

Dimensions in millimetres



Enlarged view of area X on figure 1 and section A-A on figure 2



Top view

FIGURE 2 – **Top and side views of part of a multi-cavity compression mould, for example for standard bars 50 mm × 6 mm × 4 mm**

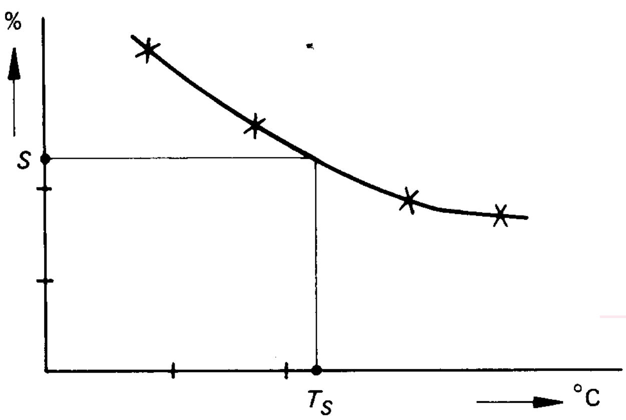
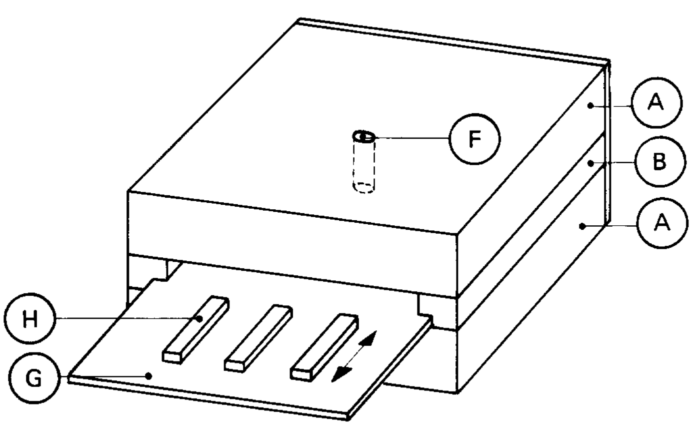
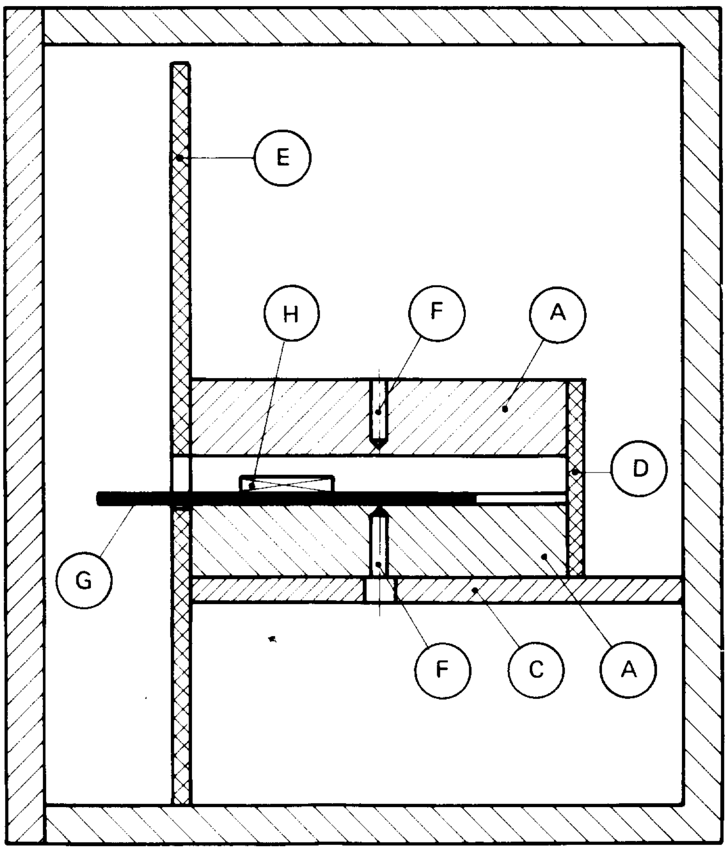


FIGURE 3 – **Determination of cylinder temperature for a given shrinkage rate**



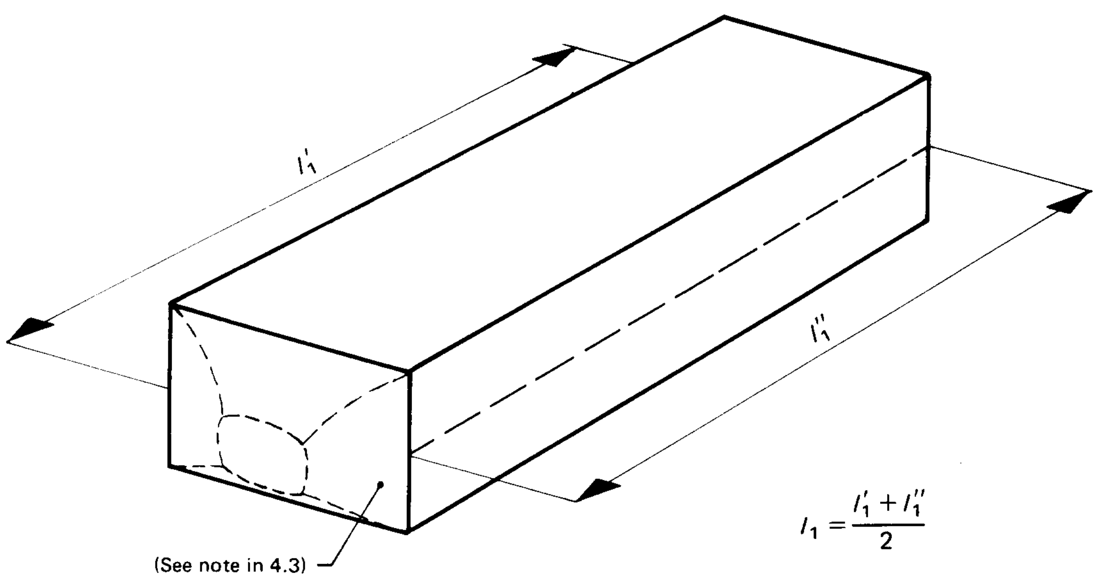
See figure 5 for key.

FIGURE 4 – **Specimen holder (conditioning chamber)**



Ⓐ Metal block (250 mm X 250 mm X 50 mm)
Ⓑ Spacer (20 mm X 250 mm X 13 mm or 25 mm)
Ⓒ Support for the specimen holder
Ⓓ Metal back plate (100 mm X 250 mm X 5 mm)
Ⓔ Asbestos sheet, 10 mm approx.
Ⓕ Drilled hole for thermocouple
Ⓖ Aluminium sheet (215 mm X 300 mm X 0,3 mm)
Ⓗ Test specimen

FIGURE 5 – **Location of specimen holder in the shrinkage oven**



$$l_1 = \frac{l'_1 + l''_1}{2}$$

FIGURE 6 – **Measurement of the mean length after shrinkage**