INTERNATIONAL STANDARD

ISO 11359-3

First edition
2002-10-01

Plastics — Thermomechanical analysis (TMA) —

Part 3: Determination of penetration temperature

Plastiques — Analyse thermomécanique (TMA) —

Partie 3: Détermination de la température de pénétration

Reference number
ISO 11359-3:2002(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

Printed in Switzerland

Contents

Page

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 11359 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 11359-3 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

ISO 11359 consists of the following parts, under the general title *Plastics — Thermomechanical analysis (TMA)*:

— *Part 1: General principles*

— *Part 2: Determination of coefficient of linear thermal expansion and glass transition temperature*

— *Part 3: Determination of penetration temperature*

Plastics — Thermomechanical analysis (TMA) —

Part 3:
Determination of penetration temperature

1 Scope

This part of ISO 11359 specifies a method for the determination of the penetration temperature of thermoplastics using thermomechanical analysis.

NOTE This method can also be used to measure the softening temperature.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of ISO 11359. 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 11359 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 472, *Plastics — Vocabulary*

ISO 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 11359-1, *Plastics — Thermomechanical analysis (TMA) — Part 1: General principles*

ISO 11359-2, *Plastics — Thermomechanical analysis (TMA) — Part 2: Determination of coefficient of linear thermal expansion and glass transition temperature*

3 Terms and definitions

For the purposes of this part of ISO 11359, the terms and definitions given in ISO 11359-1 and the following apply.

3.1
penetration mode
TMA mode used to measure the displacement of a penetration probe brought about by the softening of the test specimen

3.2
penetration temperature
temperature at which an abrupt probe displacement takes place during penetration mode TMA

4 Principle

The temperature at which the probe of a TMA apparatus begins to penetrate or change its rate of penetration is determined under a defined load when the temperature is raised at a constant rate.

5 Apparatus

5.1 TMA apparatus

See ISO 11359-1:1999, clause 6.

The apparatus shall, in addition, meet the following specifications:

a) it shall be capable of operating in the penetration mode;

b) it shall be capable of maintaining the specimen under a controlled atmosphere;

c) it shall be capable of raising the temperature at a constant rate;

d) it shall be capable of applying the specified constant stress to the specimen.

5.2 Penetration probe

The penetration probe shall be installed in the TMA apparatus so that the axis of the detector and the probe are parallel. For normal materials, the probe shall be cylindrical in shape with a flat tip. The diameter of the tip shall be 0,5 mm ± 0,05 mm or 1,0 mm ± 0,05 mm and its length not less than 1 mm. When testing highly aerated plastic foams, a larger, spherical-tip probe shall be used.

6 Test specimen

6.1 Preparation

In general, the test specimen shall be prepared from a sample of thickness between 0,5 mm and 5 mm. Thinner specimens down to 0,01 mm can be measured, however. Prepare the specimen by cutting it to a size appropriate to the apparatus. The surface of the test specimen shall be smooth and flat so that the whole of its surface is in contact with the sample holder.

NOTE A test specimen approximately 5 mm square or about 5 mm in diameter is recommended.

6.2 Conditioning

Refer to the relevant material standard for the conditioning of the test specimen before measurement.

7 Procedure

7.1 Calibration of apparatus

See ISO 11359-1:1999, subclause 8.1.

7.2 Determination

Place the test specimen in the centre of the sample holder. Position the penetration probe at the centre of the upper surface of the test specimen.

Apply a force of 0,50 N ± 0,01 N (50 gf ± 1 gf) or, by agreement between the interested parties, another force to the penetration probe. Maintain for 5 min to 10 min.

Maintain a constant gas flow, preferably of dry air, high-purity nitrogen or another inert gas, around the specimen within a flowrate range of 50 ml/min to 100 ml/min. However, other atmospheres may be used by agreement between the interested parties.

Raise the temperature of the specimen at a constant rate of not more than 5 °C/min.

Record the TMA curve in the penetration mode for the overall process.

8 Expression of results

The penetration temperature T_p is determined as the point of intersection of the tangents to the TMA curve (see Figure 1).

If the TMA curve indicates that the change takes place in more than one stage, the penetration temperatures (T_{p1}, T_{p2}…) for each of the stages shall be determined.

NOTE T_p may be used as a measure of the softening temperature.

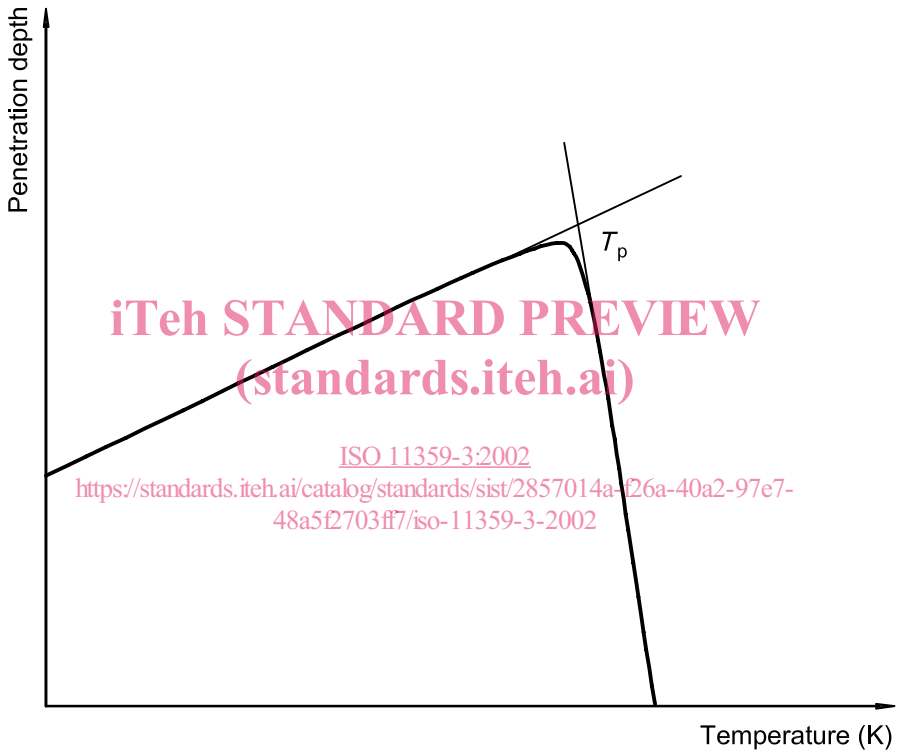

Figure 1 — Determination of penetration temperature

Calculate the mean of at least two measurements determined as above and report the result to the nearest whole number. In cases where the TMA curve indicates two or more stages, calculate mean values for each stage.

9 Precision

The precision will, in general, be dependent on the nature of the sample. Accordingly, the reader is recommended to refer to reference [1] in the Bibliography.

10 Test report

The test report shall include the following, as required:

a) all details necessary for complete identification of the sample;

b) the manufacturer and type of TMA apparatus used;

c) the shape and dimensions of the penetration probe;

d) the shape and dimensions of the test specimen;

e) details of the conditioning of the test specimen;

f) the heating rate used;

g) the test atmosphere and gas flow rate;

h) the materials used for temperature calibration and their melting points;

i) the result of the test, i.e. the mean penetration temperature T_p;

j) details of any operation not specified in this part of ISO 11359 and/or agreed on between the interested parties;

k) the date of the test.

Bibliography

[1] T. TAKAHASHI, M. SERIZAWA, T. OKINO and T. KANEKO, A round-robin test of the softening temperature of plastics by thermomechanical analysis, *Thermochimica Acta*, 147 (1989), pp. 387-399

[2] International Confederation for Thermal Analysis (ICTA), *For Better Thermal Analysis*, 3rd Edition, 1991