INTERNATIONAL STANDARD

ISO 11359-3

Second edition
2019-02

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 11359-3:2019(E)

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Published in Switzerland

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 11359-3:2002), which has been technically revised. The main changes compared to the previous edition are as follows:

— the normative references have been revised;

— the specification of penetration probes have been revised.

A list of all parts in the ISO 11359 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Plastics — Thermomechanical analysis (TMA) —

Part 3: Determination of penetration temperature

1 Scope

This document specifies a method for the determination of the penetration temperature of thermoplastics using thermomechanical analysis (TMA).

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

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 472, *Plastics — Vocabulary*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472, ISO 11359-1 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

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

3.2
penetration temperature
temperature at which an abrupt probe displacement takes place during *penetration mode* (3.1)

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.

The apparatus shall, in addition, be capable of applying the specified constant force 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. Preferably, the probe shall be cylindrical in shape with a flat tip. The nominal diameter of the tip shall be (0,5 ± 0,05) mm or (1,0 ± 0,12) mm and its length not less than 1 mm. The surface of the penetration probe shall be parallel to that of the test specimen.

If a flat surface of the test specimen or parallel alignment with the probe surface cannot be achieved, a spherical probe may be used.

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.

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 or use one of the test atmospheres specified in ISO 291.

7 Procedure

7.1 Calibration of apparatus

See ISO 11359-1.

7.2 Measurement

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 ± 0,01) N 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 flow rate 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 K/min.

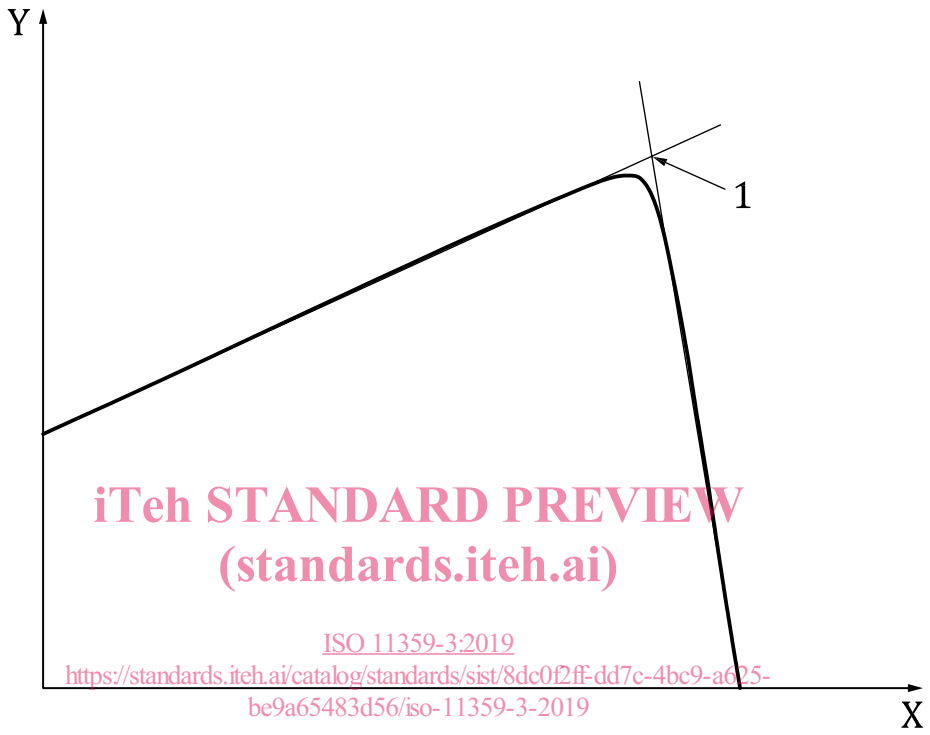
Record the TMA curve in the penetration mode for the overall process.

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}, etc.) for each of the stages shall be determined.

NOTE T_p can be used as a measure of the softening point.



Key

Y	penetration depth	X	temperature
1	penetration temperature, *T*p		

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 user is recommended to refer to Reference [1].

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 document and/or agreed on between the interested parties;

k) the date of the test.

Bibliography

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

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