INTERNATIONAL STANDARD

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Plastics — Thermomechanical analysis (TMA) —

Part 1: General principles

Plastiques — Analyse thermomécanique (TMA) —
Partie 1: Principes généraux

Reference number
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Contents

International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet iso@iso.ch

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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.

International Standard ISO 11359-1 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee 5, *Physical-chemical properties.*

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

— *Part 1: General principles*

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

— *Part 3: Determination of penetration temperature*

Annex A of this part of ISO 11359 is given for information only.

Plastics — Thermomechanical analysis (TMA) —

Part 1:
General principles

1 Scope

This part of ISO 11359 specifies the general conditions for the thermomechanical analysis of thermoplastics and thermosetting materials, filled or unfilled, in the form of sheet or moulded parts.

Thermomechanical analysis consists of the determination of deformations of a test specimen as a function of temperature and/or time.

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

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

ISO 11359-3, *Plastics — Thermomechanical analysis (TMA) — Part 3: Determination of penetration temperature*.

3 Terms and definitions

For the purposes of this part of ISO 11359, the following terms and definitions apply.

3.1
thermomechanical analysis
TMA
technique in which the deformation of a test specimen under a non-oscillatory load is recorded as a function of temperature and/or time while the specimen is subjected to a controlled temperature programme

3.2
thermodilatometry
technique in which a dimension (or the volume) of a substance under negligible stress is measured as a function of temperature while the substance is subjected to a controlled temperature programme

NOTE A distinction is made between linear thermodilatometry (in which a dimension is measured) and volume thermodilatometry (in which the volume is measured).

3.3
TMA curve
curve obtained with a TMA apparatus, the curve being a recording of any measured dimension of the sample as a function of temperature or time

4 Principle

The deformation of a material under non-oscillatory stress is measured as a function of time at a constant temperature or as a function of temperature.

5 Materials

5.1 Pure metallic reference materials, with known melting points, for temperature calibration purposes.

Table 1 — Metallic reference materials (purity > 99,99 %)

Metal	Melting point[a] °C
Indium	156,6
Tin	231,9
Lead	327,5
Zinc	419,6

a Values from NIST reference material catalogue (see annex A).

6 Apparatus

The TMA apparatus shall have the following components:

6.1 Temperature-programmable furnace, with a test chamber capable of:

— running tests at constant heating or cooling rates from 1 K/min to 20 K/min or at constant temperature in the recommended range, 123 K (–150 °C) to 773 K (500 °C);

— maintaining the temperature of the test within an accuracy of ± 2 K;

— regulating the temperature with a resolution of at least 0,5 K;

— being swept by a gas flow.

6.2 Displacement transducer, with a detection limit of at least 10^{-1} µm.

6.3 Measurement probe: a rod made of a material with a low thermal-expansion coefficient (for instance, silica), linked mechanically to the transducer, with one end in contact with the test specimen, and with a shape suited to the type of measurement.

The apparatus shall include a means of compensating for the probe mass, either through calibration or taring.

6.4 Load-application device (compression, penetration, tension or flexure).

The magnitude of the load applied to the rod depends on the type of measurement required. It is necessary to determine the force actually applied to the specimen.

6.5 Cooling device, using liquid nitrogen, circulating refrigerant, ice or circulating water, and capable of producing a stable and reproducible temperature.

6.6 Inert or oxidizing gas supply device (flow rate about 10 ml/min to 100 ml/min).

6.7 Signal acquisition and/or recording equipment.

6.8 Micrometers or calipers.

7 Test specimens

Cut test specimens from a sheet or moulded article in such a way that any heating will not modify its structure. The test specimen may be of any shape but shall have a thickness of a few millimetres. Refer to the manufacturer's instructions regarding specimen size.

Ensure that the lower and upper surfaces of the specimen are parallel and smooth, smoothing down if necessary with abrasive paper (e.g. No. 200 grade).

If specimens are taken from a moulded article, report the procedure used, i.e. method, type of article and orientation of specimen.

Condition the specimen under one of the sets of conditions specified in ISO 291, if necessary.

A visual (naked eye) examination is recommended to make sure that there are no imperfections or defects inside or on the surface, for instance bubbles, holes or scratches.

8 Procedure

8.1 Calibration

8.1.1 Furnace

Calibrate the furnace (6.1) using two or more pure metallic reference materials (5.1) with known melting points (see Table 1) covering the temperature range to be used for the test specimen. The thickness of the metallic pieces made from the reference materials shall be approximately 0,1 mm. Carry out the determinations of the melting point of the reference materials under the same experimental conditions as those which will be used for the test specimen.

Measure, under the same load as will be used for the test specimen, the point at which penetration of the reference material by the probe (6.3) occurs due to melting (see Figure 1). Determine the penetration temperature of the reference material in accordance with ISO 11359-3.

8.1.2 Transducer

Calibrate the transducer (6.2) using a micrometer (6.8) or set of standard gauges of precisely known thickness. The precision of the micrometer or gauges shall be the same as that required for the precision of the displacement transducer.

8.1.3 Load-application device

Calibrate the load-application device (6.4) by means of the standard masses provided with each apparatus or using a certified force gauge. For details, refer to the manufacturer's instructions.

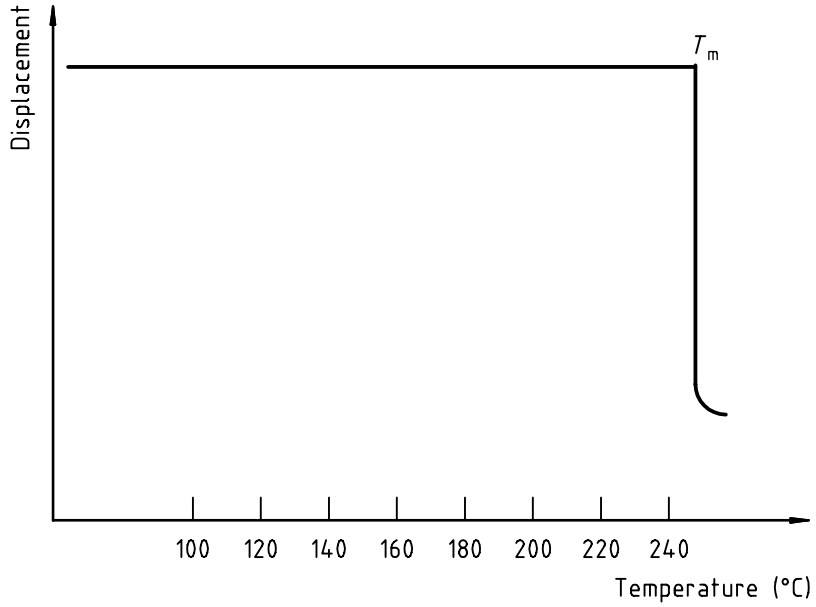

Figure 1 — Determination of the melting point

8.2 Blank run

Carry out a blank run, recording the TMA curve under the same conditions as those to be used for the test specimen, but without the test specimen.

The data obtained for the test specimen can then be corrected using the data obtained from the blank run.

8.3 Determination

Place the test specimen on the sample holder.

Place the temperature sensor as close as possible to the test specimen.

Measure the length L_0 of the test specimen at 23 °C using the displacement transducer.

Select the temperature range, the heating and/or cooling rate and the load to be applied, as given in ISO 11359-2 or ISO 11359-3 or as required in the relevant material standard.

Record the TMA curve as a function of temperature or time.

On completion of the run, allow the specimen to cool.

The details of the test conditions and procedures for each TMA test are specified in ISO 11359-2 and ISO 11359-3.

Compare the blank-run curve with that obtained for the test specimen and make any corrections necessary.

9 Test report

The test report shall include the following:

a) a reference to this part of ISO 11359;

b) all details necessary for complete identification of the material or product tested (lot number, etc.);

c) the type of test specimen used, its dimensions, the method of preparation and its orientation with reference to the sheet or article it was taken from;

d) details of the conditioning of the test specimen, if applicable;

e) the type of TMA equipment used;

f) the shape and dimensions of the probe;

g) the materials used for calibration purposes and the values obtained;

h) the experimental conditions used for the determination;

i) the results of the test, including the TMA curves obtained (if necessary);

j) the date(s) of testing.