DRAFT INTERNATIONAL STANDARD ISO/DIS 11359-1

ISO/TC **61**/SC **5** Secretariat: **DIN**

Voting begins on **2013-02-10** Voting terminates on **2013-05-10**

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

Plastics — Thermomechanical analysis (TMA) —

Part 1: General principles

Plastiques — Analyse thermomécanique (TMA) —

Partie 1: Principes généraux

[Revision of first edition (ISO 11359-1:1999)]

ICS 83.080.01

To expedite distribution, this document is circulated as received from the committee secretariat. ISO Central Secretariat work of editing and text composition will be undertaken at publication stage.

Pour accélérer la distribution, le présent document est distribué tel qu'il est parvenu du secrétariat du comité. Le travail de rédaction et de composition de texte sera effectué au Secrétariat central de l'ISO au stade de publication.

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 2.

The main task of technical committees is to prepare International Standards. 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 document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 11359-1 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 11359-1:1999), which has been technically revised.

The main changes are:

a) Clarification that deformations shall occur under constant load;

b) Inclusion of reference to ISO 472 in Definitions and cancellation of duplicate and trivial definitions;

c) Revision of apparatus requirements following guidelines specified in ISO 11357-1 and update of accuracy specifications;

d) Revision of specification of temperature calibration;

e) Revision of specification of displacement and sample length measurement.

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 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 under constant load as a function of temperature and/or time.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 11359-2, *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 Definitions

For the purposes of this document, the following terms and definitions apply.

3.1
Thermodilatometry
technique in which one dimension (or the volume) of a substance under negligible constant 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 one dimension is measured) and volume thermodilatometry (in which the volume is measured).

4 Principle

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

5 Apparatus

The components of a basic thermomechanical analyser consist of the following:

5.1 Temperature-programmable furnace, capable of:

a) generating constant heating or cooling rates for the intended measurements;

b) covering a temperature range in line with the experimental requirements;

c) maintaining the temperature variation at a given location to within ± 1 K over time;

d) measuring the temperature with an accuracy of ± 1 K over time;

e) maintaining a controlled purge gas atmosphere.

5.2 Displacement transducer, with an accuracy of ± 0,1 % or better.

5.3 Measurement probe, rod made of a material of low thermal-expansion coefficient (e.g. silica, ceramics, quartz glass, etc.), linked mechanically to the displacement 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 tarring.

5.4 Load application device, the load application device shall be suitable for the intended measurement, e.g. compression, penetration, tension, flexure, etc.

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.

5.5 Cooling device, capable of generating and maintaining controlled and reproducible low temperatures.

5.6 Inert or oxidising gas supply, type, purity and flow rates of purge gas shall be suitable for the intended measurements.

5.7 Micrometers or callipers, with an accuracy of ± 2 µm or better.

6 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 suitable for the intended measurement and specimen holder.

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).

NOTE Removal (smoothing) of the surface of specimens molded or formed from some materials may change the relative amount of oriented skin layers which, in turn, may affect the mechanical response.

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

Unless otherwise specified in the appropriate material standard, condition the specimen under one of the sets of conditions specified in ISO 291, if necessary.

A visual examination, by eye, shall be done to make sure that there are no imperfections or defects inside or on the surface, for instance bubbles, holes or scratches.

7 Procedure

7.1 Calibration

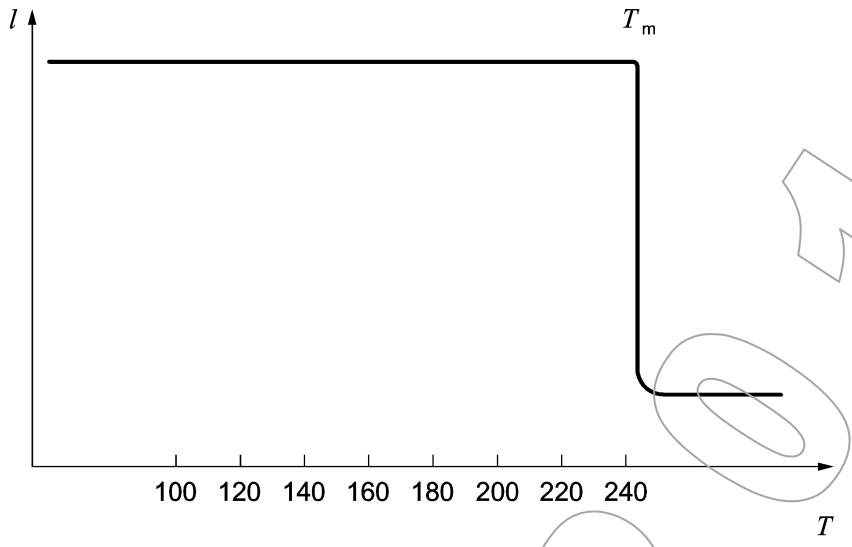
7.1.1 Furnace

Calibrate the furnace (5.1) using two or more metallic calibration materials covering the temperature range to be used for the test specimen. Calibration materials shall either be certified or chosen from those listed in Table 1 provided their purity is 99,99 % or better. The thickness of the specimens prepared from calibration materials shall be approximately 0,1 mm. Carry out the determinations of the melting point of the calibration 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 calibration material by the probe (5.3) occurs due to melting (see Figure 1). Determine the penetration temperature of the calibration material in accordance with ISO 11359-3.

Table 1 — Metallic calibration materials

Metal	Melting point[5, 6, 7] °C
Gallium	29,76
Indium	156,60
Tin	231,93
Lead	327,46
Zinc	419,53



Key

l	Displacement
T	Temperature
T_m	Melting point

Figure 1 — Determination of the melting point

7.1.2 Displacement transducer

Calibrate the displacement transducer (5.2) using a certified micrometer (5.7) or set of certified thickness gauges.

7.1.3 Load-application device

Calibrate the load-application device (5.4) by means of certified calibration masses or using a certified force gauge.

7.2 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 shall be corrected using the data obtained from the blank run.

7.3 Determination

Measure the length l_0 of the test specimen at one of the standard temperatures specified in ISO 291 using a micrometer or calliper (see 5.7).

Place the test specimen on the sample holder.

The details of the test conditions and procedures for TMA determinations are specified in ISO 11359-2 and ISO 11359-3. Select the temperature range, the heating and/or cooling rate and the load to be applied as given in those Standards, or as required in the relevant material standard.

Record the displacement as a function of temperature and/or time.

After completion of the run, cool down the specimen to room temperature.

8 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.