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Plastics — Thermomechanical analysis (TMA) —

Part 1: General principles

Plastiques — Analyse thermomécanique (TMA) —

Partie 1: Principes généraux

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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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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

For an explanation of 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 www.iso.org/iso/foreword.html.

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

This third edition cancels and replaces the second edition (ISO 11359-1:2014), which has been technically revised.

The main changes are as follows:

— the optional use of a cooling device has been added;

— the temperature calibration using the single sensor-DTA technique has been added;

— additional temperature calibration specimens have been specified for different deformation techniques;

— the list of temperature calibration substances has been extended;

— a correction method taking into account the influence of the change of length of the measuring probe has been added.

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 1: General principles

WARNING — The use of this document can involve hazardous materials, operations and equipment. It does not purport to address all of the safety or environmental problems associated with its use.

1 Scope

This document 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 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 11359-2, *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 document, the terms and definitions given in ISO 472 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 https://www.electropedia.org/

4 Principle

The deformation of a material under constant or variable 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 enclosure, capable of:

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

b) covering a temperature range in line with the experimental requirements, optionally, a suitable cooling device can be provided for measurements below ambient temperature;

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, 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 Length measurement devices, 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 can 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 moulded or formed from some materials can change the relative amount of oriented skin layers which, in turn, can 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 the location 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.

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

7 Procedure

7.1 Calibration

7.1.1 General

7.1.1.1 Temperature display

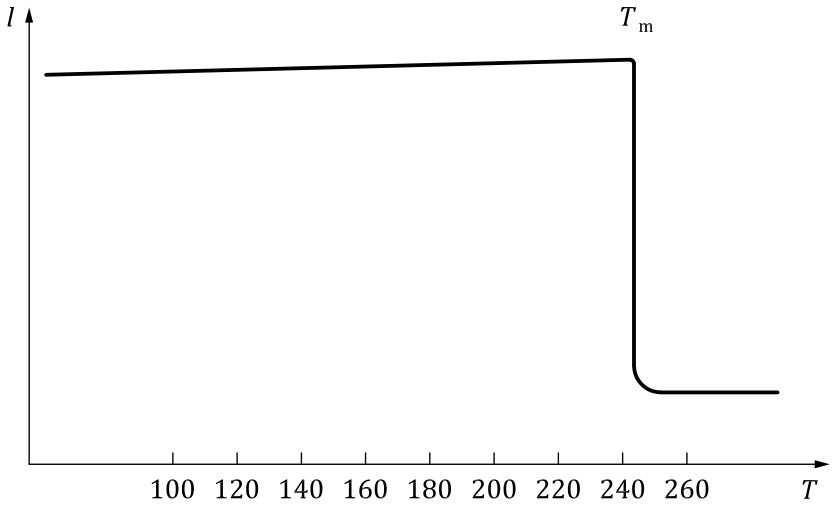
Prepare temperature calibration specimens suitable for the intended deformation mode.

Calibrate the temperature display (5.1) using two or more 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 suitable for the deformation mode and instrument used. 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 the temperature calibration specimen under the same load and heating rate using the relevant deformation mode as will be used for the test specimen, and plot the deformation against the temperature. From the sharp drop in the deformation curve (see, for example, the penetration temperature measured in Figure 1), the melting point of the calibration material can be determined.

Table 1 — Temperature calibration materials

Calibration material	Melting point[5][6] °C
Mercury	−38,83
Water	0,00
Gallium	29,76
Indium	156,60
Tin	231,93
Lead	327,46
Zinc	419,53



Key

l displacement

T Temperature, expressed in °C

T_m melting point (extrapolated onset temperature)

Figure 1 — Determination of the melting point

If the instrument is equipped with single sensor differential thermal analysis technology (SS-DTA)[7] [8], the determination of the melting point of the calibration materials may also be done using the single sensor differential thermal analysis signal.

7.1.1.2 Expansion or penetration

The temperature calibration in expansion or penetration mode may be done by placing a small sheet or drop of calibration material in a crucible and cover it with a disc-shaped plate slightly smaller than the inside diameter of the crucible. The covering disc shall be made of a material that is stable and chemically inert in the temperature range used for calibration, such as sapphire. For calibration materials with melting temperatures below ambient, the covering disc shall be made of material having a density lower than that of the calibration material, too, to prevent sinking, such as polyethylene or polypropylene.

Also, the crucible shall be made of a material that is stable and chemically inert to the sample at all temperatures under examination. Chemical inertness should be prioritised over homogeneity with the TMA sample holder and probe.

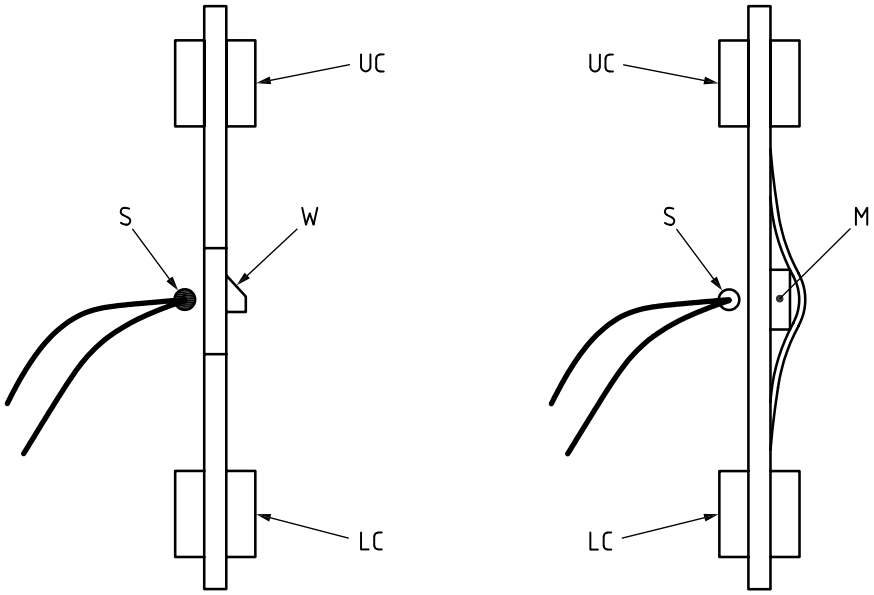
7.1.1.3 Flexure or torsion mode

The temperature calibration in flexure or torsion mode may be done using a temperature calibration specimen of bar geometry consisting of an envelope with a layer of calibration material, such as indium centrally encapsulated. Alternatively, PTFE tubes with embedded calibration materials, such as water, indium or tin, may be used.

NOTE Metals can be inserted in PTFE tubes in the form of wires of suitable diameter.

7.1.1.4 Tension mode

Temperature calibration specimens of water for the tension mode may be prepared by applying a drop of water onto a strip of paper or foil [see Figure 2 a)][9]. Calibration metals such as e.g. indium or tin can be encapsulated in aluminium foil [see Figure 2 b)][9].



a) Drop of water applied onto paper (or foil) strip **b) Calibration metal embedded in aluminium foil**

Key

UC upper clamps
LC lower clamps
S temperature sensor
W drop of water
M embedded metal

Figure 2 — Temperature calibration specimen in tension mode

These calibration specimens can be installed in the tension sample holder.

7.1.1.5 Shear and compression mode

Temperature calibration samples for shear and compression mode may be prepared similar to 7.1.1.4 and installed in the sample holder for the relevant deformation mode.

7.1.2 Displacement transducer

Calibrate the displacement transducer (5.2) using a certified length measurement device (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 Correction of the measured displacement

7.2.1 Blank correction

Due to the temperature deviation between the test chamber and the displacement transducer an inhomogeneous change of length of the measuring probe will occur. This shall be corrected for by

determination of the blank correction Δl_B measured using a specimen prepared from the measuring probe material or measured without specimen.

Carry out a blank run, recording the TMA curve under the same conditions as those to be used for the test specimen, but with a test specimen prepared from the measuring probe material. Alternatively, the blank run may be done without a test specimen.

The data obtained for the test specimen shall be corrected using the data obtained from the blank run using Formula (1):

$$\Delta l_P = \Delta l_M - \Delta l_B \quad (1)$$

where

Δl_P is the true change of length of the test specimen;

Δl_M is the measured change of length of the test specimen;

Δl_B is the blank correction determined with a test specimen prepared from the measuring probe material.

NOTE The blank correction can be done automatically by some instruments.

7.2.2 Correction for displacement of the measuring probe

For specimens with lower coefficients of thermal expansion and/or increased accuracy of displacement the displacement Δl_Q of that part of the measuring probe corresponding to the specimen length shall be taken into account.

Carry out a blank run as specified in 7.2.1 but using a specimen of the measuring probe having the same relevant dimensions as the test specimen.

Calculate the corrected change of length of the test specimen using Formula (2)

$$\Delta l_P = \Delta l_M - \Delta l_B + \Delta l_Q \quad (2)$$

where

Δl_P is the true change of length of the test specimen;

Δl_M is the measured change of length of the test specimen;

Δl_B is the blank correction determined with a specimen prepared from the measuring probe material;

Δl_Q is the true change of length of the measuring probe.

If the change of length of the measuring probe Δl_Q is not known, it may be determined using a reference specimen of known change of length Δl_{RT}. From the measured change of length, the displacement of the measuring probe can be calculated using Formula (3):

$$\Delta l_Q = \Delta l_{RT} + \Delta l_B - \Delta l_{RM} \quad (3)$$

where

Δl_Q is the true change of length of the measuring probe;

Δl_{RT} is the true change of length of the reference specimen;