INTERNATIONAL STANDARD

ISO 11359-2

Second edition
2021-11

Plastics — Thermomechanical analysis (TMA) —

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

Plastiques — Analyse thermomécanique (TMA) — Partie 2: Détermination du coefficient de dilatation thermique linéique et de la température de transition vitreuse

Reference number
ISO 11359-2:2021(E)

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published 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.

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 second edition cancels and replaces the first edition (ISO 11359-2:1999), which has been technically revised.

The main changes compared to the previous edition are as follows:

— the testing procedure has been revised with regard to test load and purge gas conditions;

— the evaluation of determination of the mean coefficient of thermal expansion with reference specimen has been specified more precisely;

— the document has been editorial 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 2: Determination of coefficient of linear thermal expansion and glass transition temperature

1 Scope

This document specifies a test method, using thermodilatometry[1], for the determination of the coefficient of linear thermal expansion of plastics in a solid state by thermomechanical analysis (TMA). This document also specifies the determination of the glass transition temperature using TMA.

NOTE The coefficient of linear thermal expansion can be measured using various types of thermodilatometry apparatus. This document concerns only TMA apparatus.

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 https://www.electropedia.org/

3.1
differential coefficient of linear thermal expansion
α
expansion, normalised to the reference length L_0 at room temperature, for any of the three dimensions at temperature T and at constant pressure p, given in reciprocal kelvins, by the following formula:

$$\alpha = \frac{(\mathrm{d}L)_p}{(\mathrm{d}T)_p} \times \frac{1}{L_0} = \frac{(\mathrm{d}L/\mathrm{d}t)_p}{(\mathrm{d}T/\mathrm{d}t)_p} \times \frac{1}{L_0}$$

where

L_0 is the reference length at room temperature T_0, in the axis of measurement;

L is the length at temperature T, in the axis of measurement;

$\mathrm{d}L$ is the change in length over the time interval dt at constant pressure p;

$\mathrm{d}T$ is the change in temperature over the time interval dt at constant pressure p

3.2
mean coefficient of linear thermal expansion
$\bar{\alpha}$
expansion, normalised to the reference length L_0 at room temperature, for any of the three dimensions in a specified temperature interval ΔT at constant pressure, given in reciprocal kelvins, by the following formula:

$$\bar{\alpha} = \frac{\Delta L}{\Delta T} \times \frac{1}{L_0}$$

where

ΔL is the change in length of the test specimen between two temperatures T_1 and T_2;

L_0 is the reference length of the test specimen at room temperature in the axis of measurement;

ΔT is the change in temperature, equal to $T_2 - T_1$

Note 1 to entry: The determination is made over a temperature interval ΔT between T_1 and T_2. The representative temperature T_{rep} is given by

$$T_{rep} = \frac{T_1 + T_2}{2}$$

Note 2 to entry: By replacing the term “length” by “volume” in the formulae in 3.1 and 3.2, the coefficient of volumetric thermal expansion can be obtained.

4 Principle

The change in a dimension of a test specimen is measured as a function of temperature using a TMA apparatus to generate a TMA curve from which the coefficient of linear thermal expansion can be calculated.

5 Apparatus

The components of the TMA apparatus used for this document shall be as specified in ISO 11359-1 and shall also be capable of

a) operating in a compression mode or a tension mode or both, and

b) maintaining the specimen under a controlled atmosphere in accordance with ISO 291.

NOTE 1 Measurements on specimens of film or fibre are made in the tension mode.

NOTE 2 Typically, an atmosphere of dry air or an inert gas such as nitrogen is used.

6 Test specimens

6.1 Preparation

Test specimens shall be prepared in accordance with ISO 11359-1.

The standard test specimen is a rectangular specimen 5 mm to 10 mm in length and about 5 mm in width. However, specimens of other dimensions may be used by agreement between the interested

parties. The ends of the test specimen shall be parallel. If applicable, the orientation of the specimen with respect to the direction of production shall be recorded, i.e. machine direction, transverse direction or other.

Reference shall be given to the relevant material standards for the number of test specimens, but at least three specimens shall be prepared and tested from each sample.

6.2 Conditioning

Reference to the relevant material standards shall be given for the conditioning of specimens before measurement.

In order to eliminate any thermal-memory effects in the specimen, each specimen may be heated from the minimum measurement temperature (at least 50 °C below T_g) to the maximum temperature (at least 50 °C above T_g), and held at this latter temperature for at least 5 min. Subsequently, the specimen may be cooled to the minimum temperature at the same rate as that to be used for actual measurements.

NOTE 1 Heating the test specimen to temperatures of 50 °C above T_g can result in changes in molecular orientation and/or blend morphology, thereby changing the coefficient of linear thermal expansion in certain directions.

NOTE 2 If thermal-memory effects are observed despite preheating for 5 min at maximum temperature, the preheating time can be extended as long as no decomposition occurs.

7 Procedure

7.1 Calibration of apparatus

The apparatus shall be calibrated in accordance with ISO 11359-1. After cleaning the surfaces of the specimen, probe and sample holder, the specimen shall be placed on the sample holder with the probe as close as possible.

7.2 Determination

The unloaded probe shall be set on the upper surface of the specimen. A load shall be applied that is big enough to achieve a reliable transfer of the load to the specimen, but at the same time low enough to prevent a detectable compression of the specimen. Preferably, loads in the range between 1 mN and 50 mN should be used. Provided the effect on the measured value is small, other loads may be used.

When specimens made of film, fibre or soft material are tested, the determination shall be carried out in tension mode with both ends of the specimen gripped.

A constant gas flow shall be maintained around the specimen, preferably of dry inert gas or dry air, within a flow rate range of 0 ml/min to 100 ml/min. However, other atmospheres may be used by agreement between the interested parties.

The temperature of the specimen shall be increased at a constant rate of not more than 5 K/min.

The TMA curve for the test specimen shall be recorded, i.e. the change in length with increasing temperature.

NOTE 1 In order to check for anisotropy effects, specimens can be taken in different directions from the sample to be investigated.

Under the same conditions, the change in length of a reference specimen having a known mean coefficient of linear thermal expansion and about the same length as the test specimen may be measured.

NOTE 2 This is not necessary when the apparatus used measures the difference in length between the test specimen and a reference specimen directly.

8 Expression of results

8.1 Method of calculation

8.1.1 Differential coefficient of linear thermal expansion, α

The differential coefficient of linear thermal expansion α, in reciprocal kelvins (K^{-1}), at temperature T shall be obtained from the TMA curve using Formula (1)(see Figure 1):

$$\alpha = \frac{dL}{dT} \times \frac{1}{L_0} \tag{1}$$

where

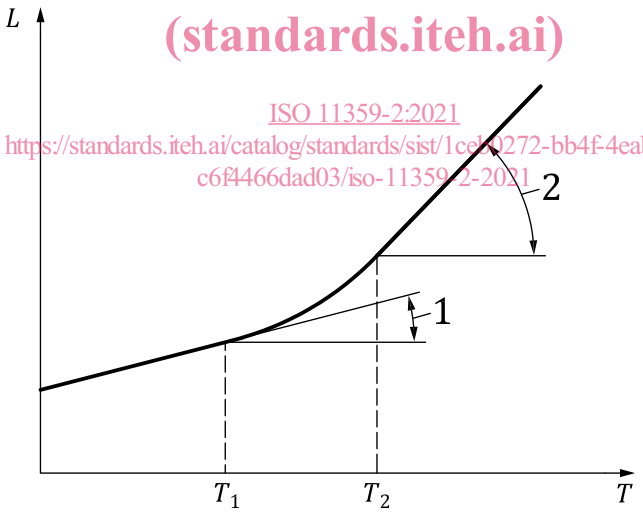
L_0 is the length of the specimen, in micrometres, at room temperature;

L is the length of the specimen, in micrometres, at temperature T;

T is the temperature, in kelvins.

The value of α shall be calculated to the nearest 10^{-7} K^{-1} for each specimen. The mean of these individual values shall be calculated and rounded to the nearest 10^{-6} K^{-1}.

In the case of a test specimen exhibiting a glass transition, the differential coefficient of thermal expansion shall be calculated before and after the glass transition.



Key

L	length of the specimen
T	temperature
T_1, T_2	selected temperatures for determination of the differential coefficient of linear thermal expansion
1	slope dL/dT at temperature T_1
2	slope dL/dT at temperature T_2

Figure 1 — Determination of differential coefficient of linear thermal expansion, α

8.1.2 Mean coefficient of linear thermal expansion, $\bar{\alpha}$

8.1.2.1 Method A: Determination without reference specimen

The mean coefficient of linear thermal expansion, $\bar{\alpha}$, in reciprocal kelvins (K^{-1}), between two temperatures T_1 and T_2 shall be obtained from the TMA curve using Formula (2) (see Figure 2):

$$\bar{\alpha} = \frac{\Delta L}{\Delta T} \times \frac{1}{L_0} \tag{2}$$

where

L_0 is the length of the specimen, in micrometres, at room temperature;

ΔL is the difference in length, in micrometres;

$\Delta T\,(=T_2 - T_1)$ is the temperature difference, in kelvins.

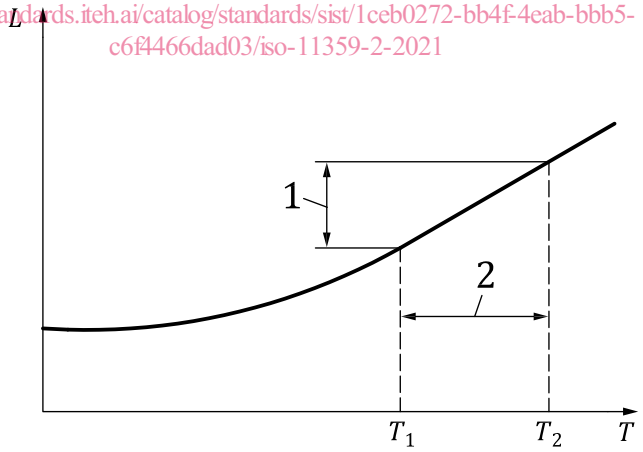
Two temperatures shall be selected and the difference in temperature ΔT shall be calculated.

The corresponding change in length ΔL shall be determined from the TMA curve.

The value of the mean coefficient of linear thermal expansion, $\bar{\alpha}$, shall be calculated to the nearest 10^{-7} K^{-1} for each specimen. The mean of these individual values shall be calculated and rounded to the nearest 10^{-6} K^{-1}.

Calculation of the corresponding representative temperature is specified in 8.1.4.

In the case of a test specimen exhibiting a glass transition, the coefficient of linear thermal expansion shall be calculated before and after the glass transition.



Key

L	length of the specimen
T	temperature
T_1, T_2	limits of the temperature range ΔT used for determination of the mean coefficient of linear thermal expansion
1	change of length ΔL between temperatures T_1 and T_2
2	change of temperature ΔT between temperatures T_1 and T_2

Figure 2 — Determination of mean coefficient of linear thermal expansion $\bar{\alpha}$

8.1.2.2 Method B: Determination with reference specimen

For this method, the length of the test specimen and the reference specimen L_0 shall be the same. The mean coefficient of linear thermal expansion $\bar{\alpha}$, in reciprocal kelvins (K^{-1}), between two temperatures T_1 and T_2 shall be obtained using Formula (3):

$$\bar{\alpha} = \frac{\Delta L_{Spm} - \Delta L_{Ref}}{L_0 \times (T_2 - T_1)} + \bar{\alpha}_{Ref} \tag{3}$$

where

$\bar{\alpha}$ is the mean coefficient of linear thermal expansion of the test specimen, in reciprocal kelvins (K^{-1})

L_0 is the length of the test specimen and reference specimen, in micrometres, at room temperature

T_1 is the lower end of the temperature range, in degrees Celsius, used to measure the mean coefficient of linear thermal expansion

T_2 is the higher end of the temperature range, in degrees Celsius, used to measure the mean coefficient of linear thermal expansion

ΔL_{Spm} is the difference in test specimen length, in micrometres, between T_1 and T_2, i.e. the measured value of [(length at T_2) – (length at T_1)]

ΔL_{Ref} is the difference in reference specimen length, in micrometres, between T_1 and T_2, i.e. the measured value of [(length at T_2) – (length at T_1)]

$\bar{\alpha}_{Ref}$ is the value of the mean coefficient of linear thermal expansion of the reference specimen, in reciprocal kelvins, between T_1 and T_2

The value of the mean coefficient of linear thermal expansion $\bar{\alpha}$ shall be calculated to the nearest 10^{-7} K^{-1} for each specimen. The mean of these individual values shall be calculated and rounded to the nearest 10^{-6} K^{-1}.

Calculation of the corresponding representative temperature is specified in 8.1.4.

In the case of a test specimen exhibiting a glass transition, the coefficient shall be calculated before and after the glass transition.

NOTE 1 Any material with known constant and reproducible coefficients of linear thermal expansion can be used for the reference specimen. Suitable examples include aluminium, gold, silica, sapphire, etc.

NOTE 2 Some instruments can measure the difference in length between the test specimen and the reference specimen $\Delta L_{Spm} - \Delta L_{Ref}$ directly.

8.1.3 Glass transition temperature

The glass transition temperature shall be determined as the point of intersection of the tangents to the TMA curve before and after the transition (see Figure 3).

NOTE The extrapolated glass transition onset temperature T_{eig} and the extrapolated glass transition end temperature T_{efg} can be determined from a differential TMA (DTMA) curve as the points of intersection of the tangent at the point of inflection with the extrapolated baseline before the glass transition and the extrapolated baseline after the glass transition, respectively. The width of the transition region is given by $T_{efg} - T_{eig}$.