BRITISH STANDARD

BS EN
61006:2004

Electrical insulating materials — Methods of test for the determination of the glass transition temperature

The European Standard EN 61006:2004 has the status of a British Standard

ICS 17.220.99; 29.035.01

National foreword

This British Standard is the official English language version of EN 61006:2004. It is identical with IEC 61006:2004. It supersedes BS EN 61006:1993 which is withdrawn.

The UK participation in its preparation was entrusted by Technical Committee GEL/15, Insulating material, to Subcommittee GEL/15/5, Methods of test, which has the responsibility to:

— aid enquirers to understand the text;

— present to the responsible international/European committee any enquiries on the interpretation, or proposals for change, and keep the UK interests informed;

— monitor related international and European developments and promulgate them in the UK.

A list of organizations represented on this subcommittee can be obtained on request to its secretary.

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EUROPEAN STANDARD **EN 61006**

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English version

Electrical insulating materials – Methods of test for the determination of the glass transition temperature
(IEC 61006:2004)

Matériaux isolants électriques – Méthodes d'essai pour la détermination de la température de transition vitreuse (CEI 61006:2004)

Elektroisolierstoffe – Prüfverfahren zur Bestimmung der Glasübergangstemperatur (IEC 61006:2004)

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CENELEC
European Committee for Electrotechnical Standardization
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Foreword

The text of document 15E/222/FDIS, future edition 2 of IEC 61006, prepared by SC 15E, Methods of test, of IEC TC 15, Insulating materials, was submitted to the IEC-CENELEC parallel vote and was approved by CENELEC as EN 61006 on 2004-03-01.

This European Standard supersedes EN 61006:1993

Changes from EN 61006:1993 are as follows:

– the standard has been completely revised from an editorial point of view and adapted to the state of the art;

– a figure to demonstrate the dynamic mechanical analysis has been introduced.

The following dates were fixed:

– latest date by which the EN has to be implemented at national level by publication of an identical national standard or by endorsement	(dop)	2004-12-01
– latest date by which the national standards conflicting with the EN have to be withdrawn	(dow)	2007-03-01

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Endorsement notice

The text of the International Standard IEC 61006:2004 was approved by CENELEC as a European Standard without any modification.

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CONTENTS

ELECTRICAL INSULATING MATERIALS – METHODS OF TEST FOR THE DETERMINATION OF THE GLASS TRANSITION TEMPERATURE

1 Scope

This International Standard specifies procedures for test methods for the determination of the glass transition temperature of solid electrical insulating materials. They are applicable to amorphous materials or to partially crystalline materials containing amorphous regions which are stable and do not undergo decomposition or sublimation in the glass transition region.

2 Terms and definitions

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

2.1
glass transition
physical change in an amorphous material or in amorphous regions of a partially crystalline material from (or to) a viscous or rubbery condition to (or from) a hard one

NOTE The glass transition generally occurs over a relatively narrow temperature region and is similar to the solidification of a liquid to a glass state; it is not a first order transition. Not only do hardness and brittleness undergo rapid changes in this temperature region, but other properties such as thermal expansion and heat capacity also change rapidly. This phenomenon is also referred to as a second order transition, rubber transition or rubbery transition. Where more than one amorphous transition occurs in a material, the one associated with changes in the segmental motions of the molecular backbone or accompanied by the largest change in properties is usually considered to be the glass transition. Blends of amorphous materials may have more than one glass transition, each associated with a separate component of the blend.

2.2
glass transition temperature
T_g
midpoint of a temperature range over which the glass transition takes place

NOTE 1 The glass transition temperature can be determined readily only by observing the temperature range in which a significant change takes place in some specific electrical, mechanical, thermal, or other physical property. Moreover, the observed temperature can vary significantly depending on the property chosen for observation and on details of the experimental technique (e.g., heating rate, frequency of test). Therefore, the observed T_g should be considered only an approximate value, valid only for that particular technique and test conditions.

NOTE 2 For the purpose of test method C (see Clause 7), the temperature of the peak of the mechanical dissipation factor curve accompanying the glass transition is taken to be the glass transition temperature.

2.3
differential scanning calorimetry
DSC
technique in which the difference in heat flow energy inputs into a tested material and a reference material is measured as a function of temperature while the tested material and the reference material are subjected to a controlled temperature programme

NOTE The record is the differential scanning calorimetric or DSC curve.

2.4
differential thermal analysis
DTA
technique in which the temperature difference between a tested material and a reference material is measured as a function of temperature while the common environment of the tested material and the reference material is subjected to a controlled temperature programme

NOTE 1 The record is the differential thermal analysis or DTA curve.

NOTE 2 There are four characteristic transition points associated with a glass transition (see Figure 1).

- Extrapolated onset temperature (T_f) in °C – The point of intersection of the tangent drawn at the point of greatest slope on the transition curve with the extrapolated baseline prior to the transition.
- Extrapolated endset temperature (T_e) in °C – The point of intersection of the tangent drawn at the point of greatest slope on the transition curve with the extrapolated baseline following the transition.
- Midpoint temperature (T_m) in °C – The point on the thermal curve corresponding to half the heat flow difference between the extrapolated onset and extrapolated endset.
- Inflection temperature (T_i) in °C – The point on the thermal curve corresponding to the peak of the first derivative (with respect to temperature) of the parent thermal curve. This point corresponds to the inflection point of the parent thermal curve.

Two additional transition points are sometimes identified and are defined.

- Temperature of first deviation (T_o) in °C – The point of first detectable deviation from the extrapolated baseline prior to the transition.
- Temperature of return-to-baseline (T_r) in °C – The point of last deviation from the extrapolated baseline beyond transition.

For the purpose of this standard T_m will be taken as the glass transition temperature T_g which usually corresponds more closely to the transition determined by the dilatometric and other methods.

NOTE 3 Other temperatures than those previously defined can be used for specification purposes as established by individual contract.

2.5
thermodilatometry
technique in which a dimension of a test specimen under negligible load is measured as a function of temperature whilst the substance is subjected to a controlled temperature programme

2.6
thermomechanical analysis
TMA
technique in which a deformation of a test specimen under non-oscillatory load is measured as a function of temperature whilst the test specimen is subjected to a controlled temperature programme

2.7
dynamic mechanical analysis
DMA
technique in which either the storage elastic or loss modulus, or both, of a substance under oscillatory load or deformation is measured as a function of temperature, frequency or time, or combination thereof

2.8
complex storage modulus
complex quantity equal to the ratio of mechanical stress to mechanical strain under sinusoidal conditions

2.9
elastic (storage) modulus
the mathematically real part of the complex storage modulus. A quantitative measurement of elastic properties defined as the ratio of the stress in-phase with oscillating strain to the magnitude of the strain

2.10
loss modulus
the mathematically imaginary part of the complex storage modulus. A quantitative measure of energy dissipation defined as the ratio of stress 90° out of phase with oscillating strain to the magnitude of the strain

2.11
mechanical dissipation factor
the ratio of the loss modulus to the storage elastic modulus

NOTE If, for instance, a material is subjected to forced, sinusoidally oscillating, linear strain ε of constant amplitude, then the mechanical stress σ in the material is determined by

$$\sigma = \underline{E}\ \varepsilon = (E' + i\ E'')\ \varepsilon$$

where

$\underline{E}$ is the complex storage modulus;

E' is the storage modulus (in this case the elastic modulus);

E'' is the loss modulus;

i is the square root of negative one.

The mechanical dissipation factor is equal to E''/E'.

3 Significance of a method

The glass transition temperature is highly dependent on the thermal history of the material structure to be tested.

For amorphous and semi-crystalline materials the determination of the glass transition temperature may provide important information about their thermal history, processing conditions, stability, progress of chemical reactions, and mechanical and electrical behaviour.

The glass transition temperature may be used, for example, as an indication of the degree of cure of thermoset materials. The glass transition temperature of thermoset materials normally increases with increasing cure. Its determination is useful for quality assurance, specification compliance and research.

4 Test methods

This standard describes three methods for the determination of the glass transition temperature. They are based on commercially available instruments, capable to operate in a typical temperature range of –100 °C to +500 °C.

One method may be more effective in the delineation of the transition than the others depending on the specific material composition, structure and physical state, etc.

Selection of the method should therefore be made according to practical criteria.

NOTE The glass transition takes place over a temperature range and is known to be affected by time dependent phenomena, such as the rate of heating (cooling). For these reasons only data gathered at the same heating rate should be compared.

Care should be taken in comparing the glass transition temperature reported by one technique with that of another.

5 Method A: By differential scanning calorimetry (DSC) or differential thermal analysis (DTA)

5.1 General

a) Differential scanning calorimetry or differential thermal analysis provides a rapid method for determining changes in heat capacity in a material.

b) The glass transition is indicated by an endothermic shift in the differential heat flow resulting from a change of the heat capacity of the material.

5.2 Interferences

An increase or decrease in heating rate from those specified may alter the results.

The presence of additives and/or impurities will affect the transition, particularly if an impurity tends to form solid solutions, or to be miscible in the post-transition phase. If particle size has an effect upon the transition temperature determined, the samples to be compared should be of approximately the same particle size. The loss of volatile components (e.g., water) during the measuring process may affect the results.

In some cases the material of the test specimens may react with air during the temperature cycle, causing an incorrect transition to be measured.

Where it has been shown that this effect is present, provision shall be made for running the test under vacuum or an inert gas blanket. Since some materials degrade near the transition region, care should be used to distinguish between degradation and transition.

Since milligram quantities of material are used, it is essential to ensure that test materials are homogeneous, representative and of similar mass and shape.

5.3 Apparatus

A differential scanning calorimeter (DSC) is used or a differential thermal analyzer (DTA) capable of heating (cooling) at rates up to at least (20 ± 1) K/min, and of automatically recording differential heat flow or differential temperature between the tested material and a reference material, to the required sensitivity and precision.

NOTE DSC is the main used one.

Aluminium or other metal pans of high thermal conductivity are used as test specimen holders.

For ease of operation an inert reference material with a heat capacity approximately equivalent to the test specimen may be used (e.g. aluminium oxide).

Nitrogen of 99,9 % purity or other inert gas supply is used for blanketing the test specimen. If oxidative reactions are excluded, air can also be used. The pressure of the selected gas shall be constant.

The dew-point of the selected gas shall be below the lowest operating temperature.

5.4 Calibration

Following the instrument manufacturer's procedure calibrate the temperature axis of the instrument by using one or more of the standard reference materials given below. Reference materials shall have a minimum purity of 99,9 % and shall be selected according to the temperature range of interest. Calibration against these materials shall employ the same heating rate, purge gas and purge gas flow rate to be used for the test specimens.

For many test measurements, the following melting-point reference materials may be used:

Reference material	*Melting point* (°C)	
Mercury	–38,9	[1][1]
Gallium	+29,8	
Indium	+156,6	[1]
Tin	+232,0	[1]
Lead	+327,5	[1]
Zinc	+419,6	[1]

NOTE The extrapolated onset (see 2.4) should be used as the melting-point temperature, as reported above, in differential scanning calorimetry, and the melting endothermic peak should be used as the melting-point temperature in differential thermal analysis in cases where the temperature sensor is located inside the test specimen.

5.5 Precautions

This standard may involve the use of hazardous materials, operations and equipment. It is the responsibility of whoever uses this standard to establish appropriate safety practices and to determine the applicability of regulatory limitations prior to use.

5.6 Test specimens

- Powdered or granular test specimens – Avoid grinding if a preliminary thermal cycle as outlined in 5.7. is not performed. Grinding or similar techniques for size reduction often introduce thermal effects because of friction or orientation, or both, and thereby change the thermal history of the test specimen.
- Moulded or pelleted test specimens – Cut the test specimens with a microtome, razor blade, hypodermic punch, paper punch or cork borer (size No. 2 or 3) to appropriate size, in thickness or diameter and length that will best fit the test specimen holder and will approximate the desired mass in the subsequent procedure.
- Film or sheet test specimens – For films thicker than 0,04 mm. For thinner films, cut slivers to fit in the test specimen holder or punch disks, if circular test specimen pans are used.
- Liquid test specimens – Catalyzed liquid thermosetting resin can be directly cured in the test pan.

NOTE Any mechanical or thermal pre-treatment should be reported.

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1 Figures in square brackets refer to the bibliography.

5.7 Procedure

a) Use a test specimen of appropriate mass for the material to be tested. In most cases 10 mg to 20 mg is satisfactory. An amount of reference material with a heat capacity closely matched to that of the test specimen shall be used.

b) Initiate flow of purge gas according to 5.3. Perform and record an initial thermal cycle up to a temperature high enough to erase previous thermal history, testing at a rate of (20 ± 1) K/min.

c) Hold temperature until a steady state is achieved (usually 5 min to 10 min).

d) Quench cool at a minimum rate of 20 K/min to well below the transition temperature of interest, usually 50 K below.

e) Hold temperature until a steady state is achieved (usually 5 min to 10 min).

f) Reheat at a maximum rate of 20 K/min and record the heating curve until all desired transitions have been completed.

Heating rates shall be reported.

NOTE The recommended reheating rate is 10 K/min in accordance with ISO/FDIS 11403-2 [2]. Increasing the heating rate produces greater base line shifts thereby improving detectability. In the case of DSC, the signal is directly proportional to the heating rate for heat capacity measurements.

g) Measure the mid-point temperature T_m (°C) and record it as T_g.

h) Determination should be made on a minimum of three test specimens and T_g reported as the mean of the determinations.

5.8 Test report

The report shall include the following.

– Complete identification and description of the material tested, including source and manufacturer's code.

– Description of instrument used for the test.

– Form of test specimen, method of preparation and any pre-treatment.

– Statement of the dimensions, geometry and material of the test specimen holder and the heating and cooling rates.

– Description of temperature calibration procedure.

– Identification of the measurement atmosphere, gas pressure and flow rate, purity and composition, including humidity, if applicable.

– The measured T_g.

– Any side reactions (for example cross linking, thermal degradation, oxidation) shall also be reported and the reaction identified, if possible.

6 Method B: By thermomechanical analysis (TMA)

6.1 General

This method covers the determination of the glass transition temperature of materials using the following techniques:

– Method B1 – Thermodilatometry (expansion mode)

  This method is applicable to materials that exhibit sufficient rigidity over the test temperature range, so that no significant indentation or compression of the test specimen by the sensing probe occurs.

– Method B2 – Thermomechanical (penetration mode)

  This method is applicable to materials that exhibit sufficient change in hardness over the test temperature range, so that significant indentation of the test specimen by the sensing probe occurs. It may not be applicable to highly filled systems.

Both techniques use a thermomechanical analyser or similar device to determine the movement of a probe positioned on a test specimen when the material is subjected to a constant heating rate.

The movement of the probe is recorded as a function of temperature.

The change in slope of the probe displacement curves in expansion mode (B1) or penetration (B2) is used to determine the transition temperature.

Thermodilatometry (Method B1) provides a rapid method to measure the change in dimension of a test specimen under negligible load as a function of temperature whilst the test specimen is subjected to a controlled temperature programme.

A discontinuity in the coefficient of thermal expansion is associated with the glass transition.

The penetration technique (Method B2) monitors instead the change in hardness of a test specimen under load as a function of temperature whilst the test specimen is subjected to a controlled temperature programme.

A discontinuity in the probe displacement versus temperature curve is associated with the glass transition.

6.2 Apparatus

Apparatus is composed with a test specimen holder into which the specimen can be placed. Changes in the length or in the compressive modulus of the specimen are sensed by the movement of a probe.

The shape and size of the probe shall be such that the load applied to the test specimen by the probe shall cause neither significant indentation nor significant compression of the test specimen (Method B1) or shall cause indentation of the test specimen (Method B2) within the temperature range of interest.

For Method B1, flat, circular probes whose diameter is 2 mm to 5 mm are used.

For Method B2, hemispherical probes of similar or lower diameter are required.

The following means are used.

- Means for sensing movement of the probe resulting from changes in length or modulus of the test specimen and for translating these movements into a signal suitable for input to a recorder or data processing system. The sensing element should be capable of producing an electrical output of at least 1 mV/μm of probe movement to a sensitivity of ±50 nm with provision for less sensitive ranges when needed.
- A means of recording changes in test specimen length (±50 nm) or probe position as a function of test specimen temperature (±0,1 K). X-Y or strip chart recorders that have sensitivities of 1 μm of probe deflection per centimetre of chart deflection or greater are acceptable. Instruments with digital and data processing require an appropriate plotter or printer plotter.
- A means for uniformly heating the test specimen at a predetermined rate over the temperature range of interest. Provisions should be made for pre-cooling the furnace and test specimen where near ambient or sub-ambient temperature measurements are to be made. Heating and cooling rates of up to at least 10 K/min are required.
- Means for measuring the temperature of the test specimen.
- A means of purging the test specimen environment with a dry inert gas, such as nitrogen or helium (the latter preferred due to its higher thermal conductivity). The dew-point of the selected gas shall be below the lowest operating temperature.

6.3 Calibration

Using the same heating rate, purge gas and purge gas flow rate used for test specimens, calibrate the instrument temperature axis using one or more >99,9 % pure standard reference materials, covering the temperature range of interest.

Temperature calibration to ±1,0 K should be achieved by observing the penetration extrapolated onset by a 50 mN loaded probe on a crystalline specimen of the reference material when heated through its melting-point at the same rate as the test specimen. The following reference materials may be used.

Reference material	*Melting point* (°C)	
Mercury	–38,9	[1]
Gallium	+29,8	
Indium	+156,6	[1]
Tin	+232,0	[1]
Lead	+327,5	[1]
Zinc	+419,6	[1]

The probe displacement measuring and recording system should be calibrated using the procedure recommended by the instrument manufacturer using standard test specimens of known thickness. For the purpose of the procedure, standard thicknesses of 300 μm to 600 μm are recommended.

6.4 Precautions

This standard may involve the use of hazardous materials, operations and equipment. It is the responsibility of whoever uses this standard to establish appropriate safety practices and to determine the applicability of regulatory limitations prior to use.

6.5 Test specimens

Test specimens can be analysed as received or after pre-treatment. If some conditioning is applied to the test specimen prior to the analysis, this treatment shall be noted in the report.

Preferred test specimen thickness is 1 mm to 3 mm. Thicknesses outside this range may be used but shall be reported. A thickness of less than 5 µm is not recommended. Test specimens with smooth parallel surfaces are preferred.

6.6 Procedure

Place a test specimen of 1 mm to 3 mm thickness in the test specimen holder under the probe. The test specimen temperature sensor is placed in contact with the test specimen or as near to the test specimen as possible (whichever is recommended by the instrument manufacturer). Select probe according to 6.2.

Soft materials can incur indentation above the glass transition temperature T_g. In such a case, if operating in expansion mode (Method B1), a supplementary thin metal disc (e.g. aluminium) may be required to be placed between the probe and the upper surface of the test specimen to effectively increase the probe diameter and thereby avoid undue penetration.

Move the furnace to enclose the test specimen holder. Start the dry inert purge gas before cooling or heating the test specimen. If measurements near ambient or sub-ambient temperatures are to be made, cool the test specimen and furnace to at least 30 K below the lowest temperature of interest. The refrigerant used for cooling should not come into direct contact with the test specimen if it is not identical with the purge gas.

Method B1: Apply a force of 5 mN to 10 mN to the sensing probe to ensure that the probe is in contact with the test specimen. Other loads may be used but shall be noted in the report.

Method B2: Apply a load of 50 mN to 1 000 mN to the sensing probe.

Select an appropriate sensitivity setting on the recorder.

NOTE Pre-analysis on a similar test specimen may be necessary to provide this information.

Heat the test specimen at a constant heating rate of (10 ± 1) K/min over the desired temperature range. Other heating rates may be used but shall be noted in the report.

An abrupt change in the slope of the displacement curve indicates a transition of the material from one state to another. The projected temperature from the intersection of the extrapolated linear portions of the curve (see Figures 2 or 3) is used as the transition temperature.

If residual stresses are evident (a sudden irreversible deflection near the glass transition), the heating should be stopped about 20 K above this temperature. The temperature is then returned to the initial conditions and the run repeated. The glass transition determined on this second run is reported along with the supplied heat treatment (see Figure 4).

Determination should be made on a minimum of three test specimens and T_g reported as the mean of the determinations. Results obtained by retesting some test specimens shall not be treated as an independent test of a new test specimen.

6.7 Calculations

Determine the glass transition temperature as follows.

a) Construct a tangent to the expansion or to the penetration curve below the transition temperature.

b) Construct a tangent to the expansion or to the penetration curve above the transition temperature.

c) The temperature at which these lines intersect is reported as the glass transition temperature (T_g).

6.8 Test report

The report shall include the following.

– Designation of the material including the name of the manufacturer and information on lot number and chemical composition when known.

– Method of test, test specimen preparation including any mechanical, thermal or environmental exposure.

– Test specimen orientation with respect to the original part or the direction of the oriented fibre fillers if a composite material is tested.

– Dimensions of the test specimen.

– Glass transition temperature (T_g).

– Description of the thermomechanical analytical apparatus used.

– Purge gas, flow rate and cooling medium if used.

– Details of probe shape and load used.

– Probe displacement curves obtained.

– Heating rate.

7 Method C: By dynamic mechanical analysis (DMA)

7.1 General

This method covers the determination of the glass transition temperature of solid electrical insulating materials using dynamic mechanical analyzers.

A test specimen of known geometry is placed in mechanical oscillation either at fixed or natural resonant frequencies. The elastic modulus mechanical dissipation factor of the test specimen is measured as a function of temperature (either isothermally or with variable temperature). A plot of the mechanical dissipation factor is indicative of the visco-elastic characteristics of the test specimen. Rapid changes in visco-elastic properties at a particular temperature are normally referred to as transition regions.

NOTE The particular method for measurement of the mechanical dissipation factor depends upon the operating principle of the instrument used.

These methods are typically intended for materials having an elastic modulus in the range of 0,5 MPa to 100 GPa. This modulus range may be extended depending upon the instrumentation used.

7.2 Interferences

Increase or decrease in heating rate from those specified may alter results.

The transition temperature is a function of the experimental frequency; therefore the frequency of oscillation must always be specified. (The transition temperature increases with increasing frequency.) Extrapolation to a reference frequency may be accomplished using a pre-determined frequency shift-factor. For the purpose of this test, the transition temperature shall be measured (or reported) at 1 Hz.

Care should be taken so that buckling of the clamped test specimen due to thermal expansion does not occur during the test.

7.3 Methods and apparatus

7.3.1 Apparatus

The function of the apparatus is to hold a test specimen of uniform cross-section, so that the test specimen acts as the elastic and dissipative element in a mechanically oscillated system.

Instruments of this type are commonly called dynamic mechanical analyzers.

7.3.2 Methods

- Resonant systems
  - Freely decaying torsional oscillation
  - Forced, constant amplitude oscillation
  - Flexural oscillation
- Non-resonant systems, fixed frequency
  - Forced, constant amplitude, torsional oscillation
  - Forced, constant amplitude, tensile oscillation
  - Forced, constant amplitude, compressive oscillation
  - Forced, constant amplitude, flexural oscillation

NOTE For resonant systems the frequency of oscillation is a function of the properties of the test specimen and is dependent on temperature.

7.3.3 Composition of the apparatus

All apparatus shall comprise the following.

– Clamps – A clamping arrangement that permits gripping of the test specimen without slipping.

– Oscillatory deformation (strain) – A device for applying an oscillatory deformation (strain) to the test specimen. The deformation (strain) may be applied and then released, as in free-vibration devices, or continuously applied, as in forced-vibration devices.

- Detectors – A device or devices for determining dependent and independent experimental parameters such as force (stress), deformation (strain), frequency of oscillation, and temperature. Temperature should be measurable with an accuracy of 0,5 K, frequency to ±1 % and force to ±1 %.
- Temperature controller and oven – A device for controlling the test specimen temperature either by heating (in steps or ramps), cooling (in steps or ramps) or maintaining a constant temperature environment.
- Dry nitrogen or other inert gas supply for purging purposes.
- Calipers or other length-measuring device capable of measuring to an accuracy of ±1 % of the test specimen dimension.

7.4 Calibration

- Ice water 0,0 °C
- Indium 156,6 °C

7.4.1 Temperature

Using the same heating rate, ramp temperature schedule or isothermal temperature used for test specimens, calibrate the instrument temperature axis, using the instrument manufacturer's procedures with either or both of the above mentioned substances.

7.4.2 Other parameters

For other parameters used in the determination of modulus and dissipation factor, calibration shall be conducted in accordance with the manufacturer's recommendation.

7.5 Precautions

Toxic or corrosive effluents, or both, may be released when heating the test specimen near its decomposition point and can be harmful to personnel or to the apparatus.

7.6 Test specimens

The size or shape of the test specimens shall be in accordance with the instrument manufacturer's recommendation.

When small quantities of test specimens are used, it is essential that the test specimens be representative of the material.

Due to the numerous types of dynamic mechanical instruments, test specimen size is not fixed by this practice. In many cases, a test specimen of 0,75 mm × 10 mm × 50 mm is found to be usable and convenient.

NOTE It is important to select a test specimen size consistent with the modulus of the material under test and capabilities of the measuring apparatus. For example, thick test specimens may be suitable for measurement on materials of low modulus while thin test specimens may be required for materials of high modulus.

7.7 Procedure

- Measure the length, width and thickness of the test specimen to an accuracy of ±1 %.
- Maximum strain amplitude shall be within the linear visco-elastic range of the material.

NOTE 1 Strains of less than 1 % are recommended.

– Select the test frequency as close to 1 Hz as is practical. The test frequency may either be fixed or variable depending upon the test apparatus.

NOTE 2 For the purpose of this test, analytical results should be reported at 1 Hz.

– Vary the temperature of the test specimen from the lowest to the highest temperature of interest while measuring its elastic and damping properties.

NOTE 3 Preferably, tests conducted over a temperature range should be performed in incremental steps or at a rate slow enough to allow temperature equilibrium throughout the entire test specimen. The time to reach equilibrium will depend upon the mass of the particular test specimen and the gripping arrangement. Temperature programme rates of 1 K/min to 2 K/min or 2 K to 5 K step intervals held for 3 min to 5 min have been found suitable. The effect of heating rate may be observed by running test specimens at two or more rates and comparing the results obtained.

NOTE 4 The accuracy required of the temperature measurement will depend upon the rate of change of mechanical dissipation factor with the temperature of the test specimen being investigated. In transition regions, experience has indicated that the test specimen temperature should be read to ±0,5 K.

– Unless otherwise specified, three test specimens shall be tested.

7.8 Calculations

Calculate the mechanical dissipation factor profile using the equations provided by the instrument manufacturer's operating manual. Plot the mechanical dissipation factor profile as a function of the test specimen temperature.

Use the average measured values of the test specimen length, width and thickness.

Select the temperature at the maximum of the mechanical dissipation factor profile as the glass transition temperature (T_g) (see Figure 5).

NOTE 1 The glass transition takes place over a temperature range and is known to be affected by time dependent phenomena, such as the rate of heating and the frequency of oscillation. For these reasons only data gathered at the same temperature programme and frequency of oscillation can be compared.

For the purpose of this test, 1 Hz is taken as the reference frequency.

NOTE 2 Comparison of the value of T_o to that obtained at other frequencies may be accomplished through the use of a predetermined frequency shift factor k.

$$\Theta_r = \Theta_o - k\,(\lg f_o - \lg f_r) = \Theta_o - k \lg (f_o / f_r)$$

where

Θ_r is the glass transition temperature at f_r ;

Θ_o is the glass transition temperature as measured at the observed frequency;

k is the frequency shift factor;

f_o is the observed frequency (Hz) of oscillation;

f_r is the reference frequency (Hz).

EXAMPLE:

If the frequency shift factor is 8 K per decade of frequency (k = 8 K) and the observed temperature is Θ_o = 100 °C with an observed frequency f_o = 15 Hz, then at f_r = 1 Hz:

Θ_r = [100 – 8 lg (15/1)] °C

= (100 – 8 × 1,18) °C

= 90,6 °C

Calculate Θ_r at 1 Hz and report it as T_g.

7.9 Test report

The report shall include the following.

- Complete identification and description of the material tested including source and manufacturer's code.
- Identification of the test method, e.g., freely decaying torsional oscillation (see 7.2).
- Dimensions of the test specimen.
- Description of the calibration procedure.
- Identification of the measurement atmosphere by gas composition, purity and rate used.
- Details of the test specimen conditioning prior to test. If some heat treatment is applied to the specimen to obtain this preferred analytical form, this treatment should be noted in the report.
- Temperature programme including initial and final temperatures as well as rate of linear temperature change or size and duration of temperature steps.
- Original experimental data.
- Number of test specimens tested.
- Equations used to obtain the mechanical dissipation factor profile.
- Plot of the mechanical dissipation factor versus test specimen temperature.
- The mechanical dissipation factor should be plotted on the ordinate with upward deflections as increase in damping. The ordinate should be clearly labelled with title and units of measurement.
- The temperature should be plotted on the abscissa increasing from the left to the right. The abscissa should be clearly labelled with title and units of measurements.
- Mean value of T_g at 1 Hz.

  For measurements made at some frequency other than 1 Hz, report the original observed temperature (θ_o), the frequency of oscillation (f_o) and the frequency shift factor used (k).

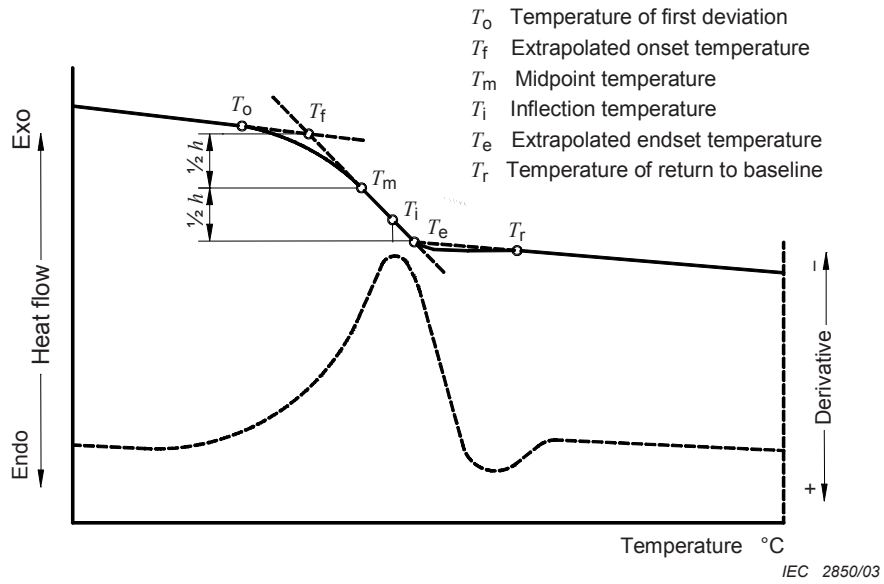

Figure 1 – Differential scanning calometry (DSC): characteristic transition points associated with glass transition

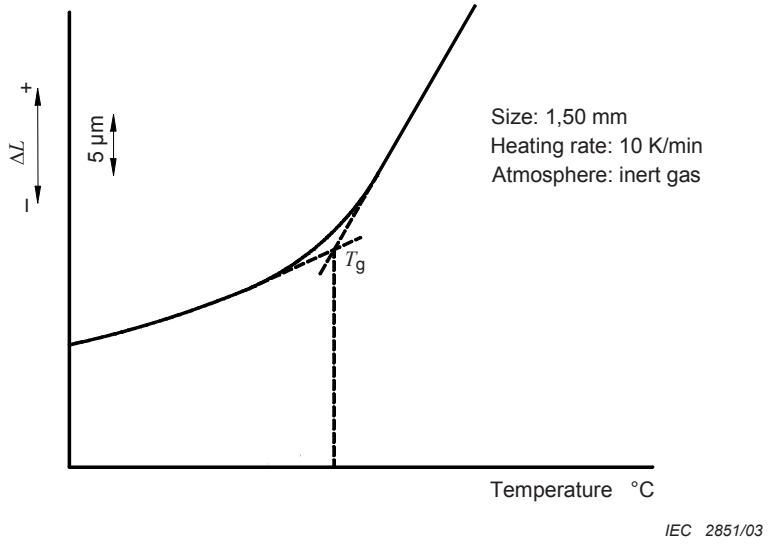

Figure 2 – Thermomechanical analysis (TMA) (Expansion mode): determination of glass transition temperature T_g

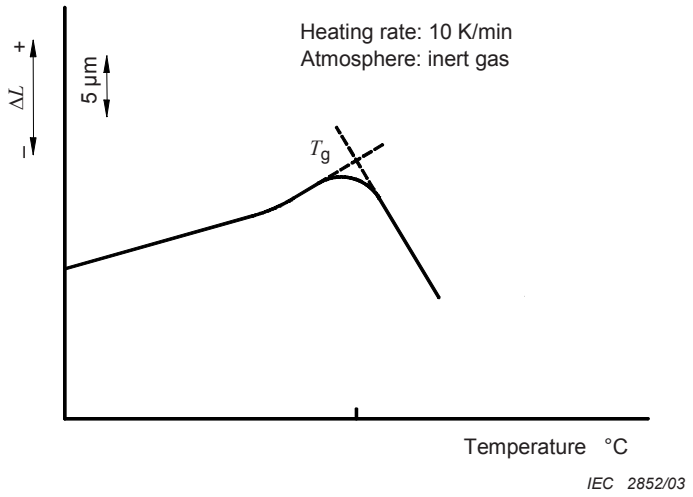

Figure 3 – Thermomechanical analysis (TMA) (Penetration mode): determination of the glass transition temperature T_g

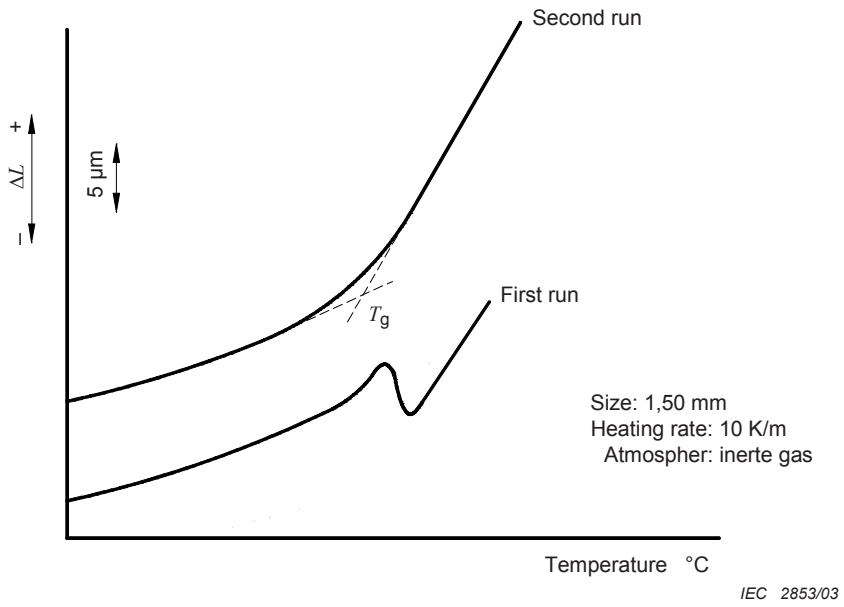

Figure 4 – Thermomechanical analysis (TMA) (Expansion mode): determination of the glass transition temperature (second run)

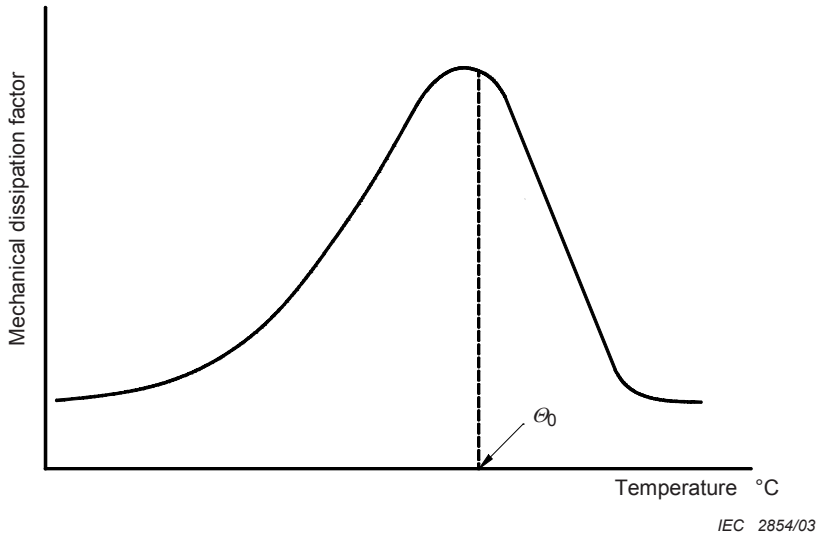

Figure 5 – Typical mechanical dissipation factor profile

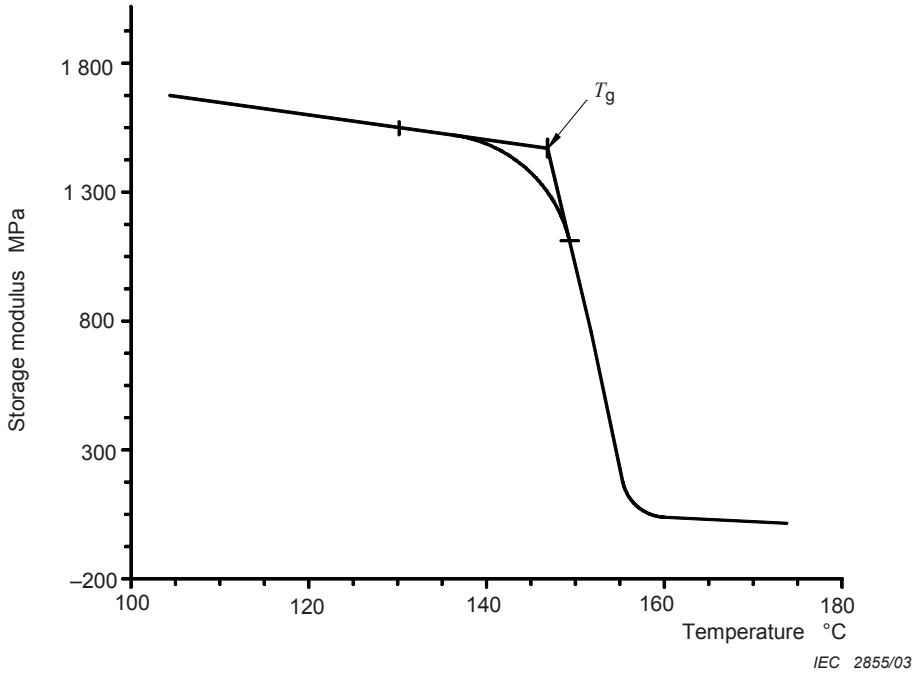

Figure 6 – Dynamic mechanical analysis (DMA): determination of the glass transition temperature T_g

Annex A
(informative)

Graphical evaluation

Figure 6 is a plot of storage modulus versus temperature. The changes in the curve indicate the glass transition region. The softening of the storage modulus (i.e. indication of the material to store energy) can be watched. A decrease in the storage modulus indicates a decrease in the stiffness of the specimen. The decrease occurs because the specimen softens from its glassy state. At higher temperatures, the storage modulus finally reaches a minimum value. T_g can be graphically evaluated from the abscissa value of the intersection of the two tangents on the curve.

The methodology may be applied to a wide variety of thermosets, thermoplastics and composites.

Bibliography

[1] Rossini, F.D., *Pure and Applied Chemistry*, volume 22, 1970, page 557.

[2] ISO/FDIS 11403-2:—, *Plastics – Acquisition and presentation of comparable multipoint data – Part 2: Thermal and processing properties* (revision of ISO 11403-2:1995)

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