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Methods of test for the determination of the glass transition temperature of electrical insulating materials (IEC 61006:1991)

Methods of test for the determination of the glass transition temperature of electrical insulating materials

Prüfmethoden zur Bestimmung der Glasübergangstemperatur von Elektroisolierstoffen

Méthodes d'essai pour la détermination de la température de transition vitreuse des matériaux isolants électriques

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(IEC 1006:1991)

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(CEI 1006:1991)

Prüfmethoden zur Bestimmung der Glasübergangstemperatur von Elektroisolierstoffen
(IEC 1006:1991)

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CENELEC

European Committee for Electrotechnical Standardization
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Europäisches Komitee für Elektrotechnische Normung

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FOREWORD

The CENELEC questionnaire procedure, performed for finding out whether or not the International Standard IEC 1006:1991 could be accepted without textual changes, has shown that no common modifications were necessary for the acceptance as European Standard.

The reference document was submitted to the CENELEC members for formal vote and was approved by CENELEC as EN 61006 on 9 March 1993.

The following dates were fixed:

- latest date of publication of an identical national standard	(dop)	1994-03-01
- latest date of withdrawal of conflicting national standards	(dow)	1994-03-01

ENDORSEMENT NOTICE

The text of the International Standard IEC 1006:1991 was approved by CENELEC as a European Standard without any modification.

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Methods of test for the determination of the glass transition temperature of electrical insulating materials

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INTERNATIONAL ELECTROTECHNICAL COMMISSION

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

FOREWORD

1) The formal decisions or agreements of the IEC on technical matters, prepared by Technical Committees on which all the National Committees having a special interest therein are represented, express, as nearly as possible, an international consensus of opinion on the subjects dealt with.

2) They have the form of recommendations for international use and they are accepted by the National Committees in that sense.

3) In order to promote international unification, the IEC expresses the wish that all National Committees should adopt the text of the IEC recommendation for their national rules in so far as national conditions will permit. Any divergence between the IEC recommendation and the corresponding national rules should, as far as possible, be clearly indicated in the latter.

PREFACE

This standard has been prepared by Sub-Committee 15A: Short-time tests, of IEC Technical Committee No. 15: Insulating materials.

The text of this standard is based on the following documents:

Six Months' Rule	Reports on Voting
15A(CO)56 15A(CO)60	15A(CO)58 15A(CO)63

Full information on the voting for the approval of this standard can be found in the Voting Reports indicated in the above table.

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

1 Scope

These methods of test cover procedures 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 Definitions

2.1 *Glass transition*

A 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 (Tg)*

The midpoint of the temperature range over which the glass transition takes place.

NOTE - The glass transition temperature can be determined readily only by observing the temperature at 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 *Tg* should be considered only an approximate value, valid only for that particular technique and test conditions.

3 Significance of the 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 provide 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 *Definitions*

5.2.1 *Differential scanning calorimetry* (DSC)

A technique in which the difference in 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. The record is the differential scanning calorimetric or DSC curve.

5.2.2 *Differential thermal analysis* (DTA)

A 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. The record is the differential thermal analysis or DTA curve.

NOTE - There are four characteristic transition points associated with a glass transition (see figure 1):

Extrapolated onset temperature (*Tf*) 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 (*Te*) 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 (*Tm*) in °C - The point on the thermal curve corresponding to half the heat flow difference between the extrapolated onset and extrapolated endset.

Inflection temperature (*Ti*) in °C - The point on the thermal curve corresponding to the peak of the first derivative (with respect to time) 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 (*To*) in °C - The point of first detectable deviation from the extrapolated baseline prior to the transition.

Temperature of return-to-baseline (*Tr*) in °C - The point of last deviation from the extrapolated baseline beyond transition.

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

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

5.3 *Interferences*

5.3.1 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 detected transition temperature, 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.