SLOVENSKI STANDARD
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Proizvodi in sistemi za zaġļ ito in popravilo betonskih konstrukcij ï Preskusne metode ï Doloļ evanje temperature prehoda za steklasto stanje polimerov

Products and systems for the protection and repair of concrete structures - Test methods - Determination of glass transition temperatures of polymers

Produkte und Systeme für den Schutz und die Instandsetzung von Betontragwerken - Prüfverfahren - Bestimmung der Glasübergangstemperatur von Polymeren

Produits et systemes pour la protection et la réparation des structures en béton - Méthodes d'essais - Détermination de la température de transition vitreuse des polymeres

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Foreword

This document (EN 12614:2004) has been prepared by Technical Committee CEN /TC 104, "Concrete and related products", the secretariat of which is held by DIN.

It has been elaborated by Sub-Committee 8 "Products and systems for the protection and repair of concrete structures" (Secretariat AFNOR).

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2005, and conflicting national standards shall be withdrawn at the latest by April 2005.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

1 Scope

This document covers a test method for the determination of glass transition temperature (GTT) of polymers by differential scanning calorimetry (DSC) or differential thermal analysis (DTA).

This test method is applicable to polymers in granular form (below 60 mesh, < 250 µ, avoiding grinding if possible) or to any fabricated shape from which appropriate samples can be cut.

This test method is useful for specification acceptance.

This test method determines the structural behaviour of a polymer according to the variations of temperatures.

2 Terms and definitions

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

2.1
differential scanning calorimetry (DSC)
differential scanning calorimetry can be carried out according to two principles, depending on the method of measurement used

Power compensation differential scanning calorimetry records in function of time or temperature the required power to maintain a zero temperature difference between the polymer and an inert reference, when they are subjected to a controlled temperature program (Power-compensation DSC).

Heat-flux differential scanning calorimetry records in function of time or temperature the difference of heat-flux diffusing between the sample holder, the reference holder and the testing unit of the equipment (Heat-flux DSC).

For the two principles, the recording chart gives a DSC curve with, at the Y-axis, the heat flow and, at the X-axis, the temperature or time.

2.2
differential thermal analysis (DTA)
differential thermal analysis records the temperature difference between the polymer sample and an inert reference, while they are subjected to a controlled temperature program

The recording chart gives a DTA curve with, at the Y-axis, the temperature difference between the sample and the reference and, at the X-axis, the temperature or time.

3 Test principle

The test method consists of heating or cooling the sample at a controlled rate in a controlled atmosphere.

A suitable sensing device monitors continuously:

— temperature difference between the sample and the reference for differential thermal analysis;

— power or heat-flux changes for differential scanning calorimetry.

Glass transition of the sample is characterized on the recording chart by a change of the baseline during the heating or the cooling.

The test shall be carried out under an inert blanket (nitrogen) to avoid any reaction of the sample with air during the temperature cycle. The output of the inert gas shall be controlled by a flowmeter.

In addition, some polymers can react near the transition temperature; care shall be used to distinguish between reaction and transition.

4 General requirements for testing

4.1 Apparatus

4.1.1 Differential thermal analyzer or differential scanning calorimeter capable of heating or cooling at rates up to at least (10 ± 1) K/min and of automatically recording any difference in temperature (or difference in heat input) between the sample and a reference material, to the required sensitivity and precision. Increasing the heating rate has been found to produce sharper transition curves. For comparison the same heating rate shall be used.

4.1.2 Sample tubes or pans or other sample holders made of aluminium or other metal of high thermal conductivity shall be used, unless the product is aggressive toward this, in which cases borosilicate glass shall be used.

4.1.3 The measuring head shall be provided by a probe or thermocouple the reference temperature of which is obtained by putting one of the solderings in a stirred bath of ice/water or by an electronic device.

4.1.4 Reference material - glass beads, alumina powder, silicon carbide, or any material known to be unaffected by repeated heating - cooling and free from interfering transitions. The thermal diffusivity of the reference should be as close as possible to that of the sample.

4.1.5 Recording charts for temperature recording apparatus, with suitable graduations for measurement of either temperature differential or energy differential against temperature or time.

4.1.6 Nitrogen or other inert gas supply, for blanketing sample.

4.2 Calibration of the temperature scale of the apparatus

Using the same heating rate to be used for samples, calibrate the temperature scale of the apparatus with appropriate standard reference materials (Analytical Reagents) covering the temperature range of interest. For many commercial polymers, this range may be defined by the following substances (see Table 1).

Table 1 – Melting points of reference materials

Standard	Melting point ($°K$)
n-heptane	182,65
n-octane	216,35
n-decane	242,85
Water	273,15
Benzoïc acid	395,55
Indium	429,55
Tin	505,05
Lead	600,55
Zinc	692,65

When lead is used as a standard, a fresh sample should be used each time.

5 Preparation of sample

5.1 General

Sample shall be homogeneous and representative.

5.2 Powdered or granular sample

Avoid grinding if preliminary thermal cycles as outlined in 6.2 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 sample.

5.3 Moulded or pelleted samples

Cut the samples 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 sample holder and will approximate the desired weight in the subsequent procedure.

5.4 Film or sheet samples

For films thicker than 0,04 mm see 5.3. For thinner films, cut slivers to fit in the sample tubes or punch disks, if circular sample pans are used.

6 Test procedure

6.1 Sample weight

Use a sample weight appropriate for the material to be tested.

In most cases sample weights of 10 mg to 20 mg for DSC, 10 mg to 100 mg for DTA are satisfactory.

NOTE Since milligram quantities of sample are used it is essential to ensure that samples are homogeneous and representative.

6.2 Preliminary thermal cycle

The preliminary thermal cycle involves heating of the sample at a rate of 10 K/min under nitrogen inert gas with a controlled flow adapted to the apparatus, from ambient temperature to 30 K above the melting point or up to a temperature high enough to erase previous thermal history.

The time of exposure to high temperature shall be minimized, to avoid sublimation or decomposition of the sample.

The selection of this temperature and duration of maintaining are crucial in case of studies about tempering.

The preliminary thermal cycle may interfere with the transition of interest, causing an incorrect transition or eliminating a transition. Where it has been shown that this effect is present, omit the preliminary thermal cycle.

6.3 Quench cooling

Quench cool to 50 °K below the transition temperature of interest.

Hold temperature for 10 min.

6.4 Test thermal cycle

Repeat heating (6.2) at a rate of 10 °*K*/min, and record the heating curve until all desired transitions have been completed.

Measurements below ambient temperature may be carried out by cooling down the apparatus by the means of liquid nitrogen - as far as the apparatus is designed for this technique - followed by the same temperature cycle as described above.

6.5 Measurements

Measure defined temperature *Tf, Tg, Te, Tm* (see Figure 1):

where

Tf is extrapolated onset temperature, in °*K*;

Tg is glass transition temperature, in °*K*;

Te is extrapolated end temperature, in °*K*;

Tm is mid point temperature, in °*K*.

The temperature at which a tangent to the curve intercepts an extension of the base line on the low temperature side shall be designated *Tf,* and the temperature at which a tangent to the curve interceps on extension of the base line on the high temperature side shall be designated *Te*.

Tm is the mid point temperature, defined at $\frac{h}{2}$, *h* being the distance between *Tf* and *Te*, measured parallel to the ordinate.

The glass transition temperature, *Tg*, is defined equal to *Tm*. However for most applications the *Tf* temperature is more meaningful and may be used as *Tg* in place of the midpoint of the *Tg* temperature.

Devices which calculate the above given temperatures, automatically or semi automatically, after putting in the straight lines by the means of a computer should work in a comparable manner.