INTERNATIONAL STANDARD

**ISO 6721-11**

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# Plastics — Determination of dynamic mechanical properties —

## Part 11: Glass transition temperature

*Plastiques — Détermination des propriétés mécaniques dynamiques —*

*Partie 11: Température de transition vitreuse*



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# Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 6721-11 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 2, *Mechanical properties*.

ISO 6721 consists of the following parts, under the general title *Plastics — Determination of dynamic mechanical properties*:

— *Part 1: General principles*

— *Part 2: Torsion-pendulum method*

— *Part 3: Flexural vibration — Resonance-curve method*

— *Part 4: Tensile vibration — Non-resonance method*

— *Part 5: Flexural vibration — Non-resonance method*

— *Part 6: Shear vibration — Non-resonance method*

— *Part 7: Torsional vibration — Non-resonance method*

— *Part 8: Longitudinal and shear vibration — Wave-propagation method*

— *Part 9: Tensile vibration — Sonic-pulse propagation method*

— *Part 10: Complex shear viscosity using a parallel-plate oscillatory rheometer*

— *Part 11: Glass transition temperature*

— *Part 12: Compressive vibration — Non-resonance method*

## Introduction

This part of ISO 6721 covers the use of dynamic mechanical analysis (DMA) procedures, in the temperature scanning mode, to determine a value for the glass transition temperature of plastics. It provides an alternative procedure to the use of differential scanning calorimetry (DSC) (see ISO 11357-2) for this measurement.

DMA is used to determine the variation of the storage modulus, loss modulus and tan delta as a function of temperature and frequency. From these data, a value for the glass transition is determined. Many types of commercial equipment are available that use this technique and, in principle, it applies to all the loading modes described in ISO 6721-1.

The procedures minimize errors due to thermal lag of the specimen, which varies with the heating rate used, through assuming the specimen temperature is given by the measured oven temperature[1)]. This eliminates the need for the temperature of the specimen to be measured directly by, for example, a thermocouple embedded in the specimen.



1) See SIMS G.D., GNANIAH S.J.P., *Calibration Procedures for Increased Confidence in DMA Measurements*, ICCM 11, Edinburgh, July 2009.

# Plastics — Determination of dynamic mechanical properties —

# Part 11: Glass transition temperature

**WARNING — The use of this part of ISO 6721 may involve hazardous materials, operations and equipment. The document does not purport to address all of the safety problems associated with its use. It is the responsibility of the user to establish appropriate health and safety practices and to determine the applicability of regulatory limitations prior to its use.**

## 1 Scope

This part of ISO 6721 specifies methods for determining a value of the glass transition temperature ($T_g$) from the dynamic mechanical properties measured during a linear temperature scan under heating conditions. The glass transition temperature is an indicator of the transition from a glassy state to a rubbery state.

Usually referred to as dynamic mechanical analysis (DMA), the methods and their associated procedures can be applied to unreinforced and filled polymers, foams, rubbers, adhesives and fibre-reinforced plastics/composites. Different modes (e.g. flexure, compression, tension) of dynamic mechanical analysis can be applied, as appropriate, to the form of the source material.



NOTE For tests undertaken in the flexure or torsion mode, an additional procedure is included to identify the severity of the influences of thermal lag on the measured data (see Annex B).



## 2 Normative references

The following referenced documents are indispensable for the application 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 6721-1:2011, *Plastics — Determination of dynamic mechanical properties — Part 1: General principles*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 6721-1 and the following apply.

**3.1**
**glass transition temperature**
$T_g$
temperature of the point of inflection of the decrease in the storage modulus curve corresponding to the transition

NOTE 1 This temperature often agrees with the temperature at the peak of the loss modulus data.

NOTE 2 It is expressed in degrees Celsius (°C).

NOTE 3 See Figure 1, data point 1.

**3.2**
**temperature at onset**
$T_{onset}$
temperature corresponding to the onset of the transition from glassy state, as defined by the intercept of two tangents in the storage modulus curve

NOTE 1 The first tangent is extrapolated from a linear portion of the curve prior to the transition, and the second tangent is extrapolated from the point of inflection of the decrease in the curve corresponding to the glass-rubber transition .

NOTE 2 It is expressed in degrees Celsius (°C).

NOTE 3 See Figure 1, data point 5.

**3.3**
**temperature at peak of loss modulus data**
$T_{loss}$
temperature of the peak of the loss modulus curve

NOTE 1 It is expressed in degrees Celsius (°C).

NOTE 2 See Figure 1, data point 2.

**3.4**
**temperature at peak of tan delta data**
$T_{tan\ delta}$
temperature of the peak in the tan delta curve



NOTE 1 It is expressed in degrees Celsius (°C).

NOTE 2 See Figure 1, data point 3.



**3.5**
**reference glass transition temperature**
$T_{g(0)}$
value of the extrapolated temperature at 0 °C/min heating rate that is used for specification and contract requirements

NOTE 1 It is expressed in degrees Celsius (°C).

NOTE 2 See Figure 2.

**3.6**
**QA glass transition temperature**
$T_{g(n)}$
value taken from the calibration curve at $n$ °C/min heating rate that is used for quality assurance purposes, by agreement, with heating rate dependent equipment (i.e. not the extrapolated $T_{g(0)}$ value]

NOTE 1 It is expressed in degrees Celsius (°C).

NOTE 2 See 9.3.2.

## 4 Principle

A specimen of known geometry is placed or held in a suitable mechanical loading system in an enclosed temperature chamber, or oven, that can be heated at a controlled rate. The specimen is mechanically oscillated at a fixed frequency, and changes in the viscoelastic response of the material are monitored and recorded as a function of the test temperature. The dynamic properties (storage modulus, loss modulus and tan delta) are determined from the load and displacement data recorded throughout the test (see ISO 6721-1). The glass transition temperature ($T_g$) is determined as the point of inflection in the storage modulus vs. the temperature plot. The test procedure described minimizes errors due to the thermal lag, which varies with the heating rate used, of the specimen temperature through assuming the specimen temperature is given by the measured oven temperature.

# 5 Equipment

## 5.1 Dynamic mechanical analyser

The test equipment shall be capable of heating at rates from 1 °C/min to 10 °C/min over the required temperature range and mechanically oscillating the specimen at the reference frequency of 1 Hz. The equipment should be capable of applying the temperature ramp profile to within ±5 % of the required heating rate.

The instrument shall continuously monitor and record the load applied to the specimen, and the corresponding displacement as a function of the measured temperature, in order to determine the storage modulus, loss modulus and tan delta. The load and displacement capabilities of the equipment shall be sufficient for the specimens tested.

The equipment shall be calibrated, as required by the equipment user manual — see Annex A.

## 5.2 Devices for measuring test specimen dimensions

These shall be in accordance with ISO 6721-1:2011, 5.6.

# 6 Test specimen

## 6.1 General

The test specimen shall be in accordance with ISO 6721-1:2011, 6.1.



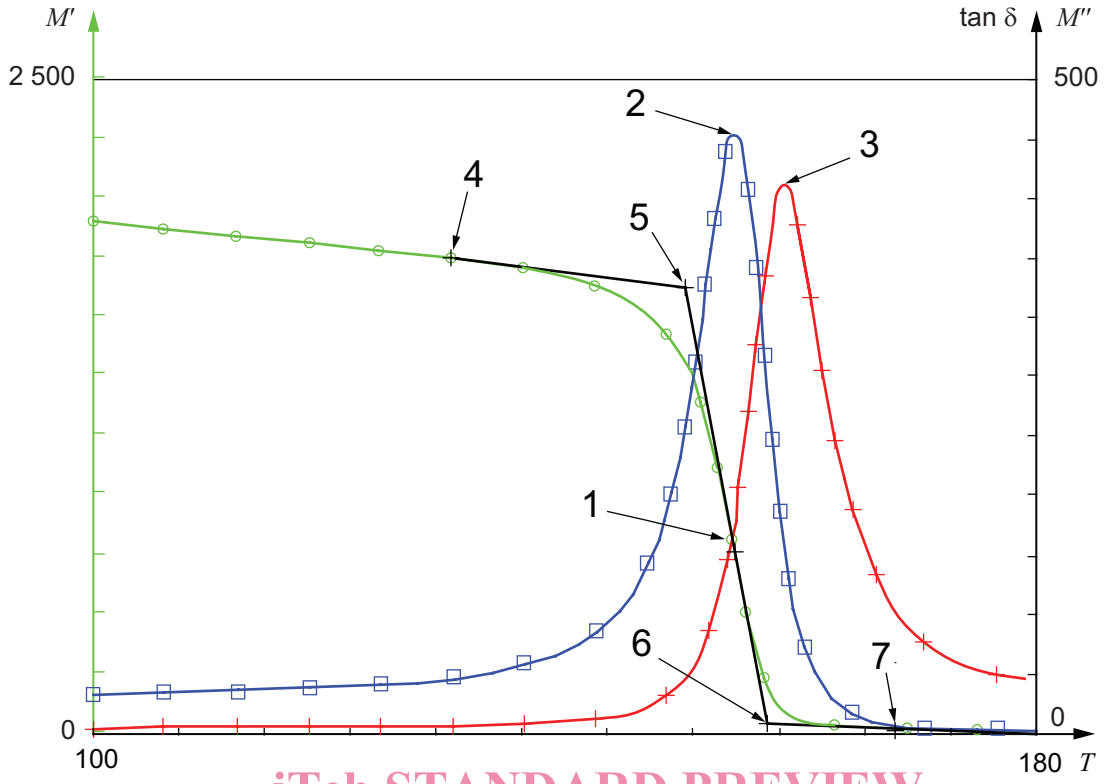
## 6.2 Shape and dimensions

The dimensions of the specimen shall be as required by the equipment for the selected test mode.



## 6.3 Preparation

The preparation of the test specimen shall be in accordance with ISO 6721-1:2011, 6.3.






**Key**

| | | | |
|---|---|---|---|
| 1 | inflection point (storage modulus) (= $T_g$) | $T$ | temperature, °C |
| 2 | peak (loss modulus) (= $T_{loss}$) | $M'$ | storage modulus, MPa |
| 3 | peak (tan delta) (= $T_{tan\ delta}$) | $M''$ | loss modulus, MPa |
| 4 | start point (storage modulus) | tan $\delta$ | tan delta |
| 5 | onset (storage modulus)(= $T_{onset}$) | | |
| 6 | endset (storage modulus) | | |
| 7 | end point (storage modulus) | | |



**Figure 1 — Plot of dynamic mechanical data against temperature**

## 7 Number of specimens

This shall be in accordance with ISO 6721-1:2011, Clause 7.

Prepare additional specimens (at least three) to assess the heating rate dependency of the method according to Clause 9.2

## 8 Conditioning

This shall be in accordance with ISO 6721-1:2011, Clause 8.

# 9 Test procedure

## 9.1 Test atmosphere

This shall be in accordance with ISO 6721:2011, 9.1

NOTE Measurements can be undertaken under static air conditions or an inert atmosphere. However, it is important that the calibration and the specimen tests be performed under identical conditions.

## 9.2 Assessment of heating rate dependence

### 9.2.1 Heating rate dependence — Procedure

Calibrate the instrument in accordance with Annex A. Position the temperature sensor in the instrument as closely as possible to the sample under test, but ensuring it is not touching it. The position of the sensor shall remain undisturbed for subsequent specimen tests. If moved, recalibration may be necessary (see Annex A).

Undertake tests according to Method A (see 9.3.1) to assess the heating rate dependence of the material/equipment.

### 9.2.2 Heating rate dependence — Results

If the temperature at the inflection points is shown to vary by more than ±2 °C between the different heating rates, use Method A (see 9.3.1).



NOTE For this case, a quality assurance procedure to reduce the testing time is also available (see 9.3.2).



If the results are shown to vary by less than ±2 °C between the different heating rates, use Method B (see 9.3.3).

## 9.3 Operation



### 9.3.1 Method A — Rate-dependent results

Mount the specimen into the instrument.

Apply a constant rate temperature scan from at least 50 °C below to 50 °C above the transition region(s) of interest at heating rates of 3 °C/min, 5 °C/min and 10 °C/min. Use a new specimen for each heating rate.

The reference test frequency of 1 Hz shall be used.

The load/displacement on the specimen shall be selected so that the specimen deformation is within the elastic range of the material being tested. The applied level shall remain constant to within ±10 % of the initial value applied.

Record the load and displacement data as a function of temperature, so that the storage modulus, loss modulus and tan delta can be calculated and plotted against temperature (see Figure 1). Determine the temperature at the inflection point for the storage modulus curve (see Figure 1, data point 1) at each heating rate.

Plot the temperature of the inflection points as a function of heating rate, as shown in Figure 2. Extrapolate the data to meet the $y$-axis at 0 °C/min using a linear fit. Report the extrapolated value to 0 °C/min as $T_{g(0)}$. These data form the “calibration curve” shown in Figure 2.

NOTE The determination of the extrapolation value can be aided by an additional scan at 1 °C/min, but care is needed if the material state (e.g. degree of cure) changes during the scan.