
Implants for surgery — Differential scanning calorimetry of poly ether ether ketone (PEEK) polymers and compounds for use in implantable medical devices

Implants chirurgicaux — Calorimétrie par balayage différentiel des polyéthercétones, polymères et composés pour l'utilisation dans les dispositifs médicaux implantables

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Contents

	Page
Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions	1
4 Apparatus and materials	2
4.1 Differential scanning calorimeter (DSC).....	2
4.2 Sample pans.....	3
4.3 Balance.....	3
4.4 Reference materials.....	3
4.5 Gas supply.....	3
5 Test specimens	3
6 Test conditions and conditioning of specimens	3
6.1 Test conditions.....	3
6.2 Conditioning of specimens.....	3
7 Instrument calibration	3
7.1 Temperature calibration.....	3
7.2 Energy calibration.....	3
8 Procedure	4
8.1 Baseline measurement.....	4
8.2 Sample preparation.....	4
8.3 Inserting the sample pans.....	4
8.4 Temperature scanning programme.....	4
8.5 Examining the specimen.....	4
8.6 Data analysis.....	5
9 Test report and typical properties	5
10 Precision	5

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. www.iso.org/directives

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The committee responsible for this document is ISO/TC 150, *Implants for surgery*, Subcommittee SC 1, *Materials*.

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Introduction

This International standard describes thermoanalytical differential scanning calorimetry (DSC) test methods for Poly Ether Ether Ketone (PEEK) which can be used for quality assurance purposes, for routine checks of raw PEEK materials and finished PEEK products or for the determination of comparable data needed for data sheets or databases.

The following definitions apply in understanding how to implement an International Standard and other normative ISO deliverables (TS, PAS, IWA):

- “shall” indicates a requirement;
- “should” indicates a recommendation;
- “may” is used to indicate that something is permitted;
- “can” is used to indicate that something is possible, for example, that an organization or individual is able to do something.

A requirement is defined as an “expression in the content of a document conveying criteria to be fulfilled if compliance with the document is to be claimed and from which no deviation is permitted.”

A recommendation is defined as an “expression in the content of a document conveying that among several possibilities one is recommended as particularly suitable, without mentioning or excluding others, or that a certain course of action is preferred but not necessarily required, or that (in the negative form) a certain possibility or course of action is deprecated but not prohibited.”

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Implants for surgery — Differential scanning calorimetry of poly ether ether ketone (PEEK) polymers and compounds for use in implantable medical devices

1 Scope

This International Standard specifies a method for the thermal analysis of Poly Ether Ether Ketone (PEEK) that is for use in the manufacture of implantable medical devices, using differential scanning calorimetry (DSC).

The transition temperatures to be determined are the glass transition temperature (T_g), the melting temperature (T_m) and the crystallization temperature on cooling (T_c).

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 11357-1:2009, *Plastics — Differential scanning calorimetry (DSC) — Part 1: General principles*

ISO 11357-2, *Plastics — Differential scanning calorimetry (DSC) — Part 2: Determination of glass transition temperature and glass transition step height*

ISO 11357-3, *Plastics — Differential scanning calorimetry (DSC) — Part 3: Determination of temperature and enthalpy of melting and crystallization*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11357-1 and the following apply.

3.1

glass transition

reversible change in an amorphous solid polymer or its amorphous regions in a semi-crystalline polymer from (or to) a viscous, rubbery condition to (or from) a hard and relatively brittle condition, which is indicated by a step change in the heat flow during heat-up

3.2

extrapolated onset temperature of glass transition

T_{ieg}

temperature at which the extrapolated initial baseline, on the low-temperature side of the curve is intersected by the tangent to the curve at the point of inflection

3.3

midpoint temperature of glass transition

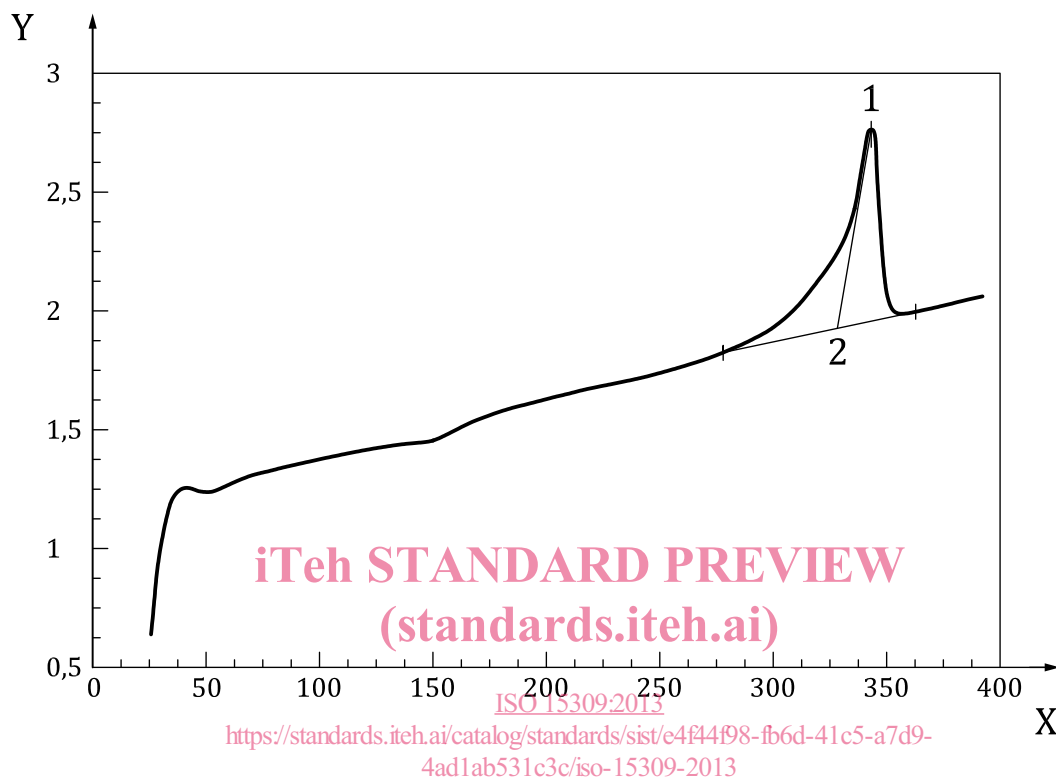
T_{mg}

temperature at which the curve is intersected by a line that is equidistant between the two extrapolated baselines

**3.4
melting temperature**

T_m
temperature at which transition between the fully or partially crystalline solid state becomes a liquid of variable viscosity, which is indicated by an endothermic peak in the DSC curve

Note 1 to entry: See [Figure 1](#).



Key

- X temperature, °C
- Y heat flow, W/g
- 1 endothermic peak: 343,84 °C
- 2 328,58 °C/49,13J/g

Figure 1 — Typical DSC Curve for PEEK (heating curve)

Note 2 to entry: The DSC trace should be free of transitions other than those listed in [Table 1](#).

**3.5
crystallization**

transition between the amorphous liquid state and the fully or partially crystalline solid state as indicated by an exothermic peak in the differential scanning calorimetry (DSC) curve

4 Apparatus and materials

4.1 Differential scanning calorimeter (DSC)

The main features of the instrument shall be as specified in ISO 11357-1:2009, 5.1.

4.2 Sample pans

Test material and reference pans shall be of equal size and mass, being manufactured from the same material. The material of construction shall be physically and chemically inert to PEEK up to a temperature of 500 °C.

EXAMPLE Aluminium.

4.3 Balance

The balance shall be capable of measuring the specimen, sample pan and reference pan mass to an accuracy of $\pm 0,01$ mg.

4.4 Reference materials

At least two certified reference materials that bracket the measured temperature range are required.

4.5 Gas supply

Analytical grade gases shall be used.

5 Test specimens

CAUTION — PEEK is a hard semi-crystalline polymer and care should be taken during any cutting operation in order to prevent injury.

The test specimens of PEEK polymer can be in the form of powder, granules or pellets, or it may be cut from sample pieces of moulded or extruded polymer. When cutting specimens care shall be taken to avoid heating or polymer re-orientation that could affect the properties. Ideally a sharp scalpel, snips or a microtone should be used.

6 Test conditions and conditioning of specimens

6.1 Test conditions

Testing shall be undertaken under ambient laboratory conditions. The DSC shall be switched on at least one hour before testing to allow for equilibration of the electronics and temperature.

6.2 Conditioning of specimens

Test sample shall be conditioned for a minimum of 4 h at a constant temperature between 18 °C and 28 °C.

7 Instrument calibration

7.1 Temperature calibration

Determine the transition temperatures for the two standard materials and measure the extrapolated onset temperature in accordance with ISO 11357-1. Compare the nominal values to the recorded values. The temperature calibration repeatability shall be better than 2 %.

7.2 Energy calibration

Determine the heat of fusion for the two standard materials and compare these values to the nominal values. The repeatability shall be within 2 %.