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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

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

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

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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

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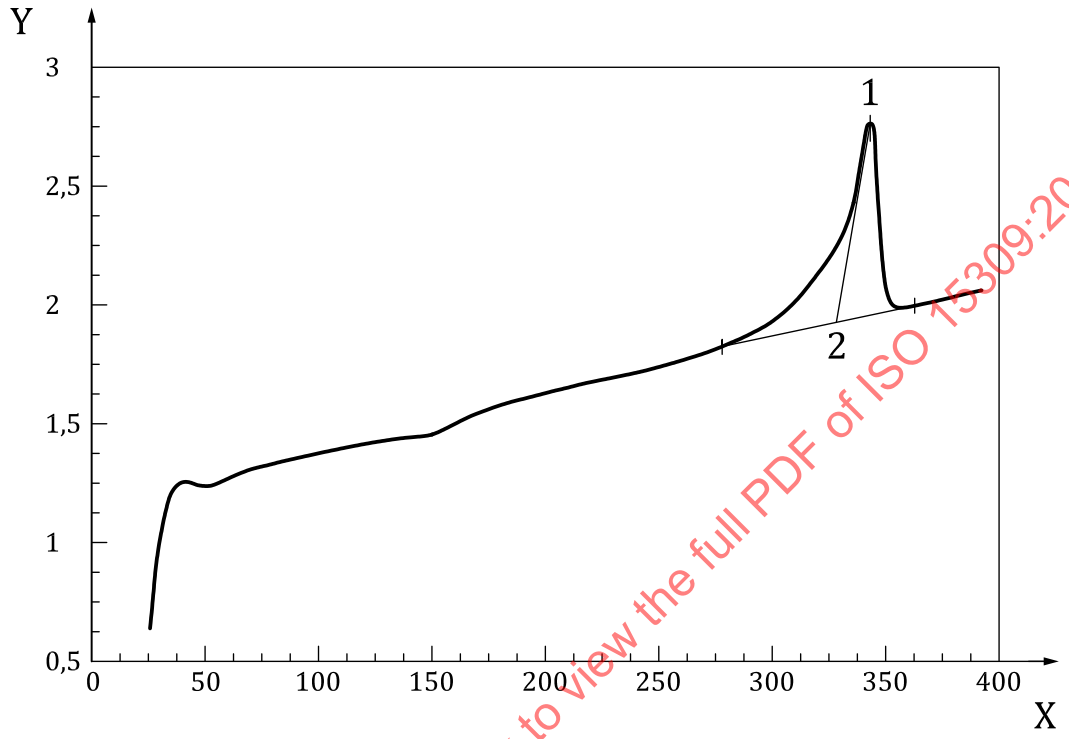
3.4
melting temperature
T_m
temperature at which transition between the fully or partially crystalline solid state becomes a liquid of variable viscosity, which is indicated by an endothermic peak in the DSC curve

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.

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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 ± 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 samples 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 %.

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8 Procedure

8.1 Baseline measurement

Place empty crucibles of the same nominal mass at the specimen and reference positions in the crucible holder. Adjust the experimental conditions to those which will be used for the actual measurement run. The recorded DSC curve (i.e. the instrument baseline) should be close to a straight line over the required temperature range. lf significant baseline curvature is observed, check the crucible holder for contamination.

NOTE With computer-controlled instruments, any remaining curvature can be corrected for by subtracting the instrument baseline from the DSC curve.

When a reasonably straight line cannot be obtained, record the DSC curve after confirming its repeatability.

8.2 Sample preparation

At least two specimens shall be prepared for each batch or sample under investigation using the following procedure.

— Weigh the sample pan and lid to the nearest 0,1 mg.

— Place the PEEK sample into the pan. The sample weight shall be adjusted so that it is between 5 mg and 15 mg. Record the specimen weight to the nearest 0,1 mg.

— Re-weigh the sealed sample pan and record the weigh to the nearest 0,1 mg.

— Seal the pan using a lid per the manufacturer's recommended procedure.

— Weigh and record the reference pan to the nearest 0,1 mg. Seal the pan using a lid per the manufacturer's recommended procedure.

— Re-weigh the sealed reference pan and record the weigh to the nearest 0,1 mg.

8.3 Inserting the sample pans

Use a suitable instrument (e.g. tweezers) to place the specimen and reference pans into the appropriate DSC furnace. Ensure that there is good, intimate contact between the base of the cell and the furnace. Close the instrument lid.

8.4 Temperature scanning programme

The following temperature program shall be used:

a) heat the specimen from 30 °C to 400 °C at a rate of 20 °C/min;

b) hold at 400 °C for 5 min;

c) cool from 400 °C to 30 °C at a set point rate of 20 °C/min;

d) re-heat from 30 °C to 400 °C at a rate of 20 °C/min.

8.5 Examining the specimen

Remove the specimen pan from the instrument and check if there has been any overflow or deformation of the pan. If so, separate the pan and lid to observe the residual specimen for evidence of volatilization or reaction.

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Due to the inherent stability of PEEK, and PEEK compounds, overflow from the pan and/or deformation of the pan should not occur.

Discard the result if any evidence of volatilization or reaction is observed in the batch of PEEK polymer or PEEK compound.

8.6 Data analysis

8.6.1 General

The data shall be processed according to the instrument manufacturer's instructions.

8.6.2 Melting temperature

The melting temperature shall be determined in accordance with ISO 11357-3 and recorded.

8.6.3 Crystallization temperature

The crystallization temperature on cooling shall be determined in accordance with ISO 11357-3 and recorded.

8.6.4 Glass transition temperature

The glass transition temperature shall be measured from the second heating cycle in accordance with ISO 11357-2. This can be recorded as either the T_{eig} or the T_{mg}.

9 Test report and typical properties

In addition to the information required in ISO 11357-1, the test report shall include the batch number or sample reference number.

The typical range into which the transitions fall is given in Table 1.

If any of the transitions fall outside of the expected range, then the batch of PEEK polymer, or PEEK compound material under test might not be suitable for surgical implant applications.

10 Precision

The precision of this method is presently unknown as inter-laboratory data are not available. Once inter-laboratory data are made available a precision statement can be added.

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