

BS ISO 15309:2013



BSI Standards Publication

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

bsi.

...making excellence a habit.™

National foreword

This British Standard is the UK implementation of ISO 15309:2013.

The UK participation in its preparation was entrusted to Technical Committee CH/150/1, Materials for surgical implants.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2013. Published by BSI Standards Limited 2013

ISBN 978 0 580 74909 4

ICS 11.040.40

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 31 December 2013.

Amendments issued since publication

Date	Text affected
------	---------------

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 polyéthercétone, polymères et composés pour l'utilisation dans les dispositifs médicaux implantables





COPYRIGHT PROTECTED DOCUMENT

© ISO 2013

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

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

Published in Switzerland

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

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received. www.iso.org/patents

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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

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.”

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

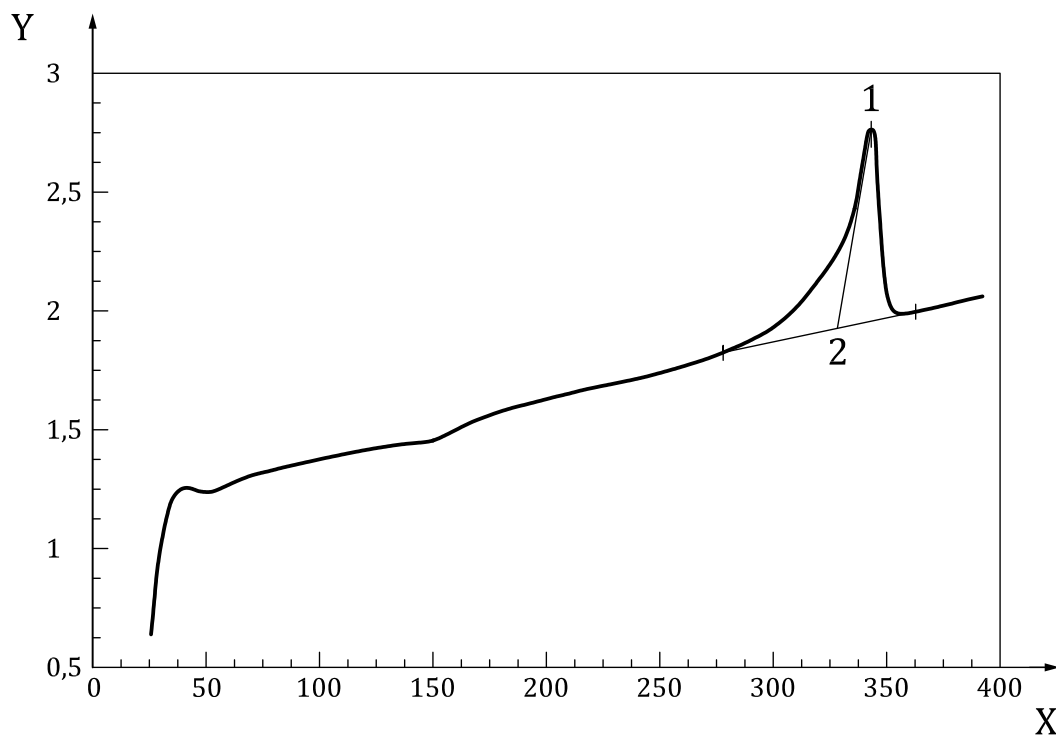
T_{mg}

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 microtome 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 %.

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. If 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.

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.

Table 1 — Typical range for temperature transitions

Parameter	Temperature transitions °C
T_g	125 to 165
T_m	320 to 360
T_c	260 to 320

British Standards Institution (BSI)

BSI is the national body responsible for preparing British Standards and other standards-related publications, information and services.

BSI is incorporated by Royal Charter. British Standards and other standardization products are published by BSI Standards Limited.

About us

We bring together business, industry, government, consumers, innovators and others to shape their combined experience and expertise into standards-based solutions.

The knowledge embodied in our standards has been carefully assembled in a dependable format and refined through our open consultation process. Organizations of all sizes and across all sectors choose standards to help them achieve their goals.

Information on standards

We can provide you with the knowledge that your organization needs to succeed. Find out more about British Standards by visiting our website at bsigroup.com/standards or contacting our Customer Services team or Knowledge Centre.

Buying standards

You can buy and download PDF versions of BSI publications, including British and adopted European and international standards, through our website at bsigroup.com/shop, where hard copies can also be purchased.

If you need international and foreign standards from other Standards Development Organizations, hard copies can be ordered from our Customer Services team.

Subscriptions

Our range of subscription services are designed to make using standards easier for you. For further information on our subscription products go to bsigroup.com/subscriptions.

With **British Standards Online (BSOL)** you'll have instant access to over 55,000 British and adopted European and international standards from your desktop. It's available 24/7 and is refreshed daily so you'll always be up to date.

You can keep in touch with standards developments and receive substantial discounts on the purchase price of standards, both in single copy and subscription format, by becoming a **BSI Subscribing Member**.

PLUS is an updating service exclusive to BSI Subscribing Members. You will automatically receive the latest hard copy of your standards when they're revised or replaced.

To find out more about becoming a BSI Subscribing Member and the benefits of membership, please visit bsigroup.com/shop.

With a **Multi-User Network Licence (MUNL)** you are able to host standards publications on your intranet. Licences can cover as few or as many users as you wish. With updates supplied as soon as they're available, you can be sure your documentation is current. For further information, email bsmusales@bsigroup.com.

BSI Group Headquarters

389 Chiswick High Road London W4 4AL UK

Revisions

Our British Standards and other publications are updated by amendment or revision.

We continually improve the quality of our products and services to benefit your business. If you find an inaccuracy or ambiguity within a British Standard or other BSI publication please inform the Knowledge Centre.

Copyright

All the data, software and documentation set out in all British Standards and other BSI publications are the property of and copyrighted by BSI, or some person or entity that owns copyright in the information used (such as the international standardization bodies) and has formally licensed such information to BSI for commercial publication and use. Except as permitted under the Copyright, Designs and Patents Act 1988 no extract may be reproduced, stored in a retrieval system or transmitted in any form or by any means – electronic, photocopying, recording or otherwise – without prior written permission from BSI. Details and advice can be obtained from the Copyright & Licensing Department.

Useful Contacts:

Customer Services

Tel: +44 845 086 9001

Email (orders): orders@bsigroup.com

Email (enquiries): cservices@bsigroup.com

Subscriptions

Tel: +44 845 086 9001

Email: subscriptions@bsigroup.com

Knowledge Centre

Tel: +44 20 8996 7004

Email: knowledgecentre@bsigroup.com

Copyright & Licensing

Tel: +44 20 8996 7070

Email: copyright@bsigroup.com



...making excellence a habit.™