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Implants for surgery — Ultra-high-molecular-weight polyethylene —

Part 4: Oxidation index measurement method

Implants chirurgicaux — Polyéthylène à très haute masse moléculaire —

Partie 4: Méthode de mesurage de l'indice d'oxydation

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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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 (see 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 (see 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.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 150, *Implants for surgery*, Subcommittee SC 1, *Materials*.

This second edition cancels and replaces the first edition (ISO 5834-4:2005) which has been technically revised.

The main changes compared to the previous edition are as follows:

— test methods harmonized with respective ASTM standards;

— editorial updates in line with all other parts of the ISO 5834 series.

A list of all parts in the ISO 5834 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

This document describes a method for the measurement of the relative extent of oxidation present in ultra-high molecular weight polyethylene (UHMWPE) intended for use in surgical implants. The material is analysed by infrared spectroscopy. The intensity of the carbonyl absorptions (>C=O) centred near 1 720 cm^{-1} is related to the amount of chemically bound oxygen present in the material. Other forms of chemically bound oxygen (R_1OR_2, R_1OOR_2, ROH, etc.) are not detected by this method.

Although this method might give the investigator a means to compare the relative extent of carbonyl oxidation present in various UHMWPE samples, it is recognized that other forms of chemically bound oxygen can be important contributors to characteristics of these materials.

The applicability of the infrared method has been demonstrated by many literature reports. This particular method, using the intensity (area) of the C-H absorption centred near 1 370 cm^{-1} to normalize for the sample's thickness, has been validated by an interlaboratory study (ILS).

Implants for surgery — Ultra-high-molecular-weight polyethylene —

Part 4: Oxidation index measurement method

1 Scope

This document specifies a method for the measurement of the relative extent of oxidation present in ultra-high molecular weight polyethylene (UHMWPE).

It is applicable to ultra-high molecular weight polyethylene (UHMWPE) intended for use in surgical implants.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 5834-2, *Implants for surgery — Ultra-high molecular weight polyethylene — Part 2: Moulded forms*

ISO 11542-1, *Plastics — Ultra-high-molecular-weight polyethylene (PE-UHMW) moulding and extrusion materials — Part 1: Designation system and basis for specifications*

ISO 11542-2, *Plastics — Ultra-high-molecular-weight polyethylene (PE-UHMW) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 11542-1 and ISO 11542-2 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
aperture size
L_a
length and width of a rectangular aperture, or the diameter of a circular aperture used by an infrared spectrometer to make spectral measurements

3.2
bulk oxidation index
$I_{ox,b}$
<sample> mean of the oxidation indices collected over a range of about 0,5 mm near the centre of the sample's oxidation index profile

Note 1 to entry: Typically this is a plateau region with the smallest oxidation indices. For samples less than about 8 mm to 10 mm thick, this central region might display the sample's highest oxidation indices, depending on its state of oxidation.

3.3
depth locator
d_l
measurement of the mean distance from the articular surface, or surface of interest, from which a spectrum was collected and a corresponding I_{ox} calculated

3.4
increment size
L_i
distance between two adjacent locations on a test film where sequential infrared spectra are collected

Note 1 to entry: This distance is typically a constant for a given test specimen.

3.5
normalization peak area
A_{norm}
total area of the normalization peak(s) between 1 330 cm^{-1} and 1 396 cm^{-1}

Note 1 to entry: This area is computed as the area between the baseline and the spectral trace, as shown in Figure 1.

3.6
oxidation
incorporation of oxygen into another molecule (e.g. UHMWPE) by means of a chemical covalent bond

3.7
oxidation index
I_{ox}
ratio of the area of the absorption peak(s) between 1 650 cm^{-1} and 1 850 cm^{-1} (A_{ox}) to the area of the absorption peak(s) between 1 330 cm^{-1} and 1 396 cm^{-1} (A_{norm})

Note 1 to entry: See Figure 1.

3.8
oxidation index profile
graphical representation of variation of the sample's oxidation index with distance from its articular surface or the surface of interest

Note 1 to entry: This is a plot of I_{ox} against d_l. Typically the graph will show the profile through the entire thickness of the sample.

3.9
oxidation peak area
A_{ox}
total area of the absorption peak(s) between 1 650 cm^{-1} and 1 850 cm^{-1}

Note 1 to entry: This area is computed as the area between the baseline and the spectral trace, as shown in Figure 1.

3.10
surface oxidation index
*I*ox,s
<sample> mean of the oxidation indices from the sample's articular surface, or the surface of interest, to a depth of 3 mm subsurface

4 Test articles

The test articles shall be made from UHMWPE moulded material and classified as Type 1, Type 2 or Type 3 in accordance with ISO 5834-2.

NOTE The UHMWPE finished products for this application are not equipped with light stabilizers and therefore need to be protected against UV influence.

5 Materials and apparatus

5.1 Materials

The test articles for oxidation index measurements shall be made from UHMWPE moulded forms in accordance with the requirements of ISO 5834-2.

5.2 Apparatus

5.2.1 Infrared spectrometer, calibrated, capable of recording a transmission absorption spectrum over the range of about 1 200 cm^{-1} to about 2 000 cm^{-1} using 150 µm to 250 µm thick films at a resolution of 4 cm^{-1} and an aperture of about 0,2 mm × 0,2 mm. An increment size of 0,2 mm is recommended.

Other modes of collection [i.e. percent reflection, attenuated total reflection (ATR), etc.], and aperture and increment sizes may be used to generate the sample's absorption spectrum provided they can be demonstrated to produce equivalent results. Too large an aperture can result in a loss of profile accuracy.

When a Fourier transform infrared (FTIR) spectrometer is used, a minimum of 32 scans shall be collected per spectrum unless the reproducibility of the results can be justified as described in ASTM F2102, in which case a minimum of 8 scans shall be collected. The FTIR instrument and sample compartment should be purged with a moisture- and carbon-dioxide-free inert gas (e.g. nitrogen, helium, or argon) to minimize spectral interference from these components.

5.2.2 Specimen holder, consisting of equipment capable of accurately positioning the sample under the aperture.

5.2.3 Microtome, consisting of equipment capable of producing 150 µm to 250 µm thick slices (films) of a sample perpendicular to the articular surface or the surface of interest.

6 Significance and use

The methods described in this document may be used to measure the oxidation indices of UHMWPE components under real-time conditions such as shelf ageing and after implantation and accelerated oxidative challenges.