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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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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 (see www.iso.org/directives).

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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

$I_{\mathrm{ox,b}}$

<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.

7 Procedure

7.1 Preparation of test specimens

Using a microtome, or other appropriate device, prepare a thin slice of the sample 150 µm to 250 µm thick. The slice shall typically be taken near the centre of the sample's articular surface or the surface of interest. The orientation of the slice shall typically be perpendicular to the articular surface or the surface of interest.

If the samples have been taken from retrieved devices or in-vitro specimens which have been exposed to lipids, the retrieved devices shall be submerged in a reagent (heptane or hexane) to extract lipids from the polymer that interfere with the carbonyl peak absorptions.

7.2 Configuration of test specimen in the spectrometer

The test film (slice) shall be first configured in the spectrometer (after an appropriate background spectrum has been collected) so that the aperture is positioned over the first 200 µm of the film starting at the articular surface. Subsequent spectra shall be collected sequentially at about 200 µm increments from the articular surface across the width of the film to the opposite surface. Larger increment sizes may be used, however too large an increment size can result in a loss of profile accuracy.

7.3 Preparation of the infrared spectrometer

Prepare the infrared spectrometer for collection of a transmission absorption spectrum according to the manufacturer's recommendations and the conditions described in 5.2.1. Collect the sequence of spectra according to 7.2.

8 Calculations

8.1 General

The results obtained from the following calculations can be useful for describing the oxidation characteristics of a sample or comparing the oxidation characteristics of one sample with another.

8.2 Oxidation peak area

For each absorbance spectrum, calculate the total area of the peak absorptions between 1 650 cm^{-1} and 1 850 cm^{-1} (Figure 1, A_{ox}). This is the area below the sample's absorption curve and above the straight baseline drawn between the same starting and ending points, namely: 1 650 cm^{-1} and 1 850 cm^{-1}.

NOTE For samples that are significantly oxidized, their carbonyl absorption peak(s) is typically very intense and broad. For such samples, a starting and ending wavenumber for the absorption peak(s) and its baseline can be as wide as 1 650 cm^{-1} to 1 850 cm^{-1}. For samples displaying very small levels of oxidation, their carbonyl absorption peak(s) is typically very weak and narrow in comparison to highly oxidized samples. For such samples, a starting and ending wavenumber for the absorption peak(s) and its baseline can be closer to 1 680 cm^{-1} to 1 765 cm^{-1}.

8.3 Normalization peak area

For each absorbance spectrum, calculate the total area of the peak absorptions between 1 330 cm^{-1} and 1 396 cm^{-1} (Figure 1, A_{norm}). This is the area below the sample's absorption curve and above the straight baseline drawn between the same starting and ending points, namely: 1 330 cm^{-1} and 1 396 cm^{-1}.

8.4 Oxidation index

For each absorbance spectrum, calculate its I_{ox} by dividing the area of its oxidation peak (8.2) by the area of its normalization peak (8.3), as shown in Figure 1.

8.5 Depth locator

Calculate the mean distance from the articular surface (d_l) for each spectrum and its corresponding I_ox from the following formula:

$$d_\mathrm{l} = 0{,}5L_\mathrm{a} + nL_\mathrm{i}$$

where

L_a is the size of the aperture in micrometers in the step direction;

n is the number of steps (increments) by which the aperture is moved from its initial location at the articular surface;

L_i is the step (increment) size in micrometers.

NOTE The absorbance recorded by the instrument corresponds to the area of the sample (aperture) being illuminated by the IR beam. The $0{,}5L_\mathrm{a}$ factor in the equation allows for the calculation of the position of the centre of the aperture relative to the starting point or edge of the sample film.

8.6 Sample's surface oxidation index

Calculate a sample's $I_\mathrm{ox,s}$ by calculating the mean average of the sample's oxidation indices (I_ox) with depth locator (d_l) values between 0 mm and 3 mm.

NOTE 1 The first 3 mm of material subsurface to an articular surface typically experiences the greatest degree of real time oxidation and the greatest stresses during real time use. The $I_\mathrm{ox,s}$ is one way of representing the sample's oxidative state in this important region.

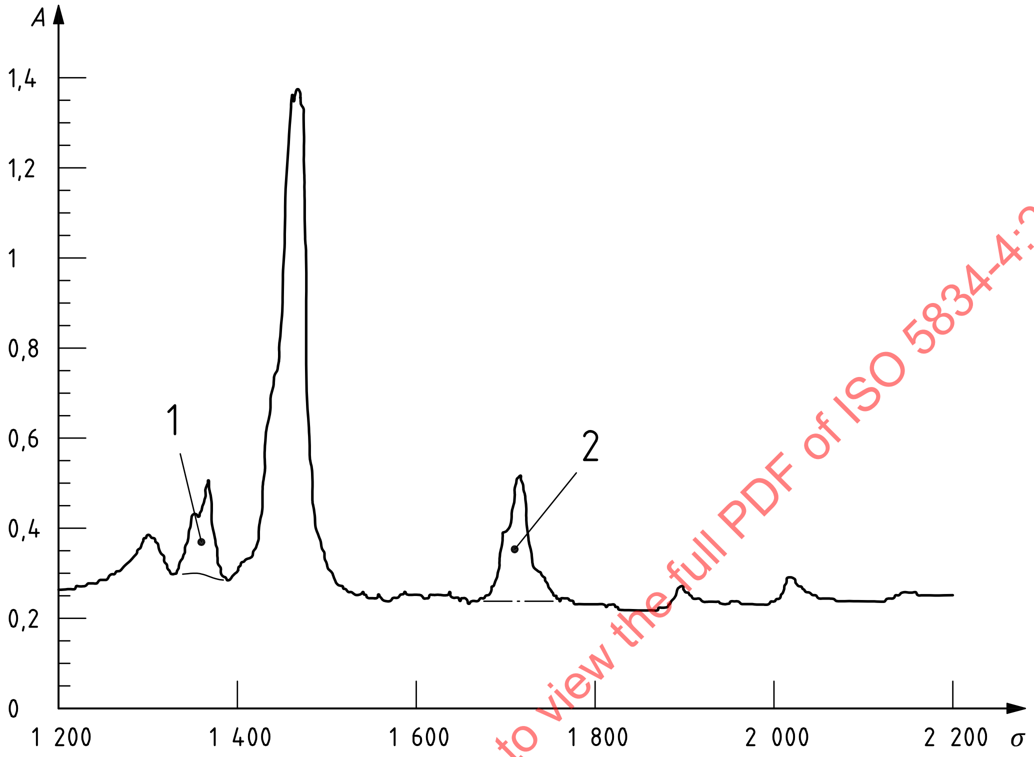
For samples demonstrating low levels of oxidation, the carbonyl region baseline wavelength should be established between 1 680 cm^{-1} and 1 765 cm^{-1}.

NOTE 2 There is no consensus on the definition of low levels of oxidation the reduction of the baseline in the carbonyl region can be required.

NOTE 3 A round robin results for virgin UHMWPE can be found in ASTM F2102:2013.

8.7 Sample's bulk oxidation index

Calculate a sample's $I_{ox,b}$ by calculating the mean of the sample's oxidation indices (I_{ox}) corresponding to about the centre 0,5 mm of material.



Key

A absorbance

σ wavenumber, cm^{-1}

1 normalization peak area, A_{norm} (1 370 cm^{-1})

2 oxidation peak area, A_{ox}

I_{ox} = A_{ox}/A_{norm}

Figure 1 — Typical FTIR spectra of oxidized UHMWPE, showing the definition of an area-based oxidation index based on normalization using the 1 370 cm^{-1} peak

8.8 Sample's oxidation index profile

Construct a plot of a sample's oxidation indices (I_{ox}, y-axis) versus their corresponding depth locators (d_l, x-axis).

9 Reports

9.1 General

It is important that details regarding the preparation of the test samples, the prior history of the material and the spectrometer operating parameters be recorded. Report the following minimum information in 9.2 to 9.8.