



Designation: F 2102 – 01^{ε1}

Standard Guide for Evaluating the Extent of Oxidation in Ultra-High-Molecular- Weight Polyethylene Fabricated Forms Intended for Surgical Implants¹

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^{ε1} NOTE—Section 4.1.1 was editorially corrected in October 2001.

1. Scope

1.1 This guide describes a method for the measurement of the relative extent of oxidation present in ultra-high-molecular-weight polyethylene (UHMWPE) intended for use in medical implants. The material is analyzed by infrared spectroscopy. The intensity (area) of the carbonyl absorptions ($>C=O$) centered near 1720 cm^{-1} is related to the amount of chemically bound oxygen present in the material. Other forms of chemically bound oxygen (C-O-C, C-O-O-C, C-O-H, and so forth) are not captured by this method.

1.2 Although this method may 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 may be important contributors to these materials' characteristics.

1.3 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 centered near 1370 cm^{-1} to normalize for the sample's thickness, has been validated by an Interlaboratory Study (ILS) conducted according to Practice E 691.

1.4 The following precautionary caveat pertains only to the test method portion, Section 5, of this specification: *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory requirements prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method²

¹ This guide is under the jurisdiction of ASTM Committee F04 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.15 on Material Test Methods.

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² *Annual Book of ASTM Standards*, Vol 14.02.

3. Terminology

3.1 Definitions:

3.1.1 *bulk oxidation index (BOI)*—a sample's bulk oxidation index (BOI) is the average of the oxidation indices collected over a $500\text{-}\mu\text{m}$ section at the center of the sample.

3.1.1.1 *Discussion*—Typically, this is a plateau region with the smallest oxidation indices.

3.1.1.2 *Discussion*—For samples less than about 8 to 10 mm thick, this central region may display the sample's highest oxidation indices, depending on its state of oxidation.

3.1.2 *depth locator (DL)*—a measurement of the distance from the articular surface, or surface of interest, that a spectrum was collected and a corresponding OI calculated.

3.1.3 *oxidation index (OI)*—an oxidation index (OI) is defined as the ratio of the area of the absorption peak(s) between 1650 and 1850 cm^{-1} to the area of the absorption peak(s) between 1330 and 1396 cm^{-1} , as shown in Fig. 1.

3.1.4 *oxidation index profile*—an oxidation index profile is the graphical representation of variation of the sample's oxidation index with distance from its articular surface or the surface of interest. This is a plot of an OI versus DL. Typically, the graph will show the profile through the entire thickness of the sample.

3.1.5 *surface oxidation index (SOI)*—a sample's surface oxidation index (SOI) is the average of the oxidation indices from the sample's articular surface, or the surface of interest, to a depth of 3-mm subsurface.

4. Apparatus

4.1 Infrared Spectrometer:

4.1.1 A calibrated infrared spectrometer capable of recording a transmission absorption spectrum over the range of about 1200 to about 2000 cm^{-1} using about $200\text{-}\mu\text{m}$ -thick films at a resolution of 4 cm^{-1} and an aperture of about 200 by $200\text{ }\mu\text{m}$.

4.1.1.1 Other modes of collection (that is, percent reflection, attenuated total reflection (ATR), and so forth) and aperture and increment sizes may be used to generate the sample's absorption spectrum provided they can be demonstrated to

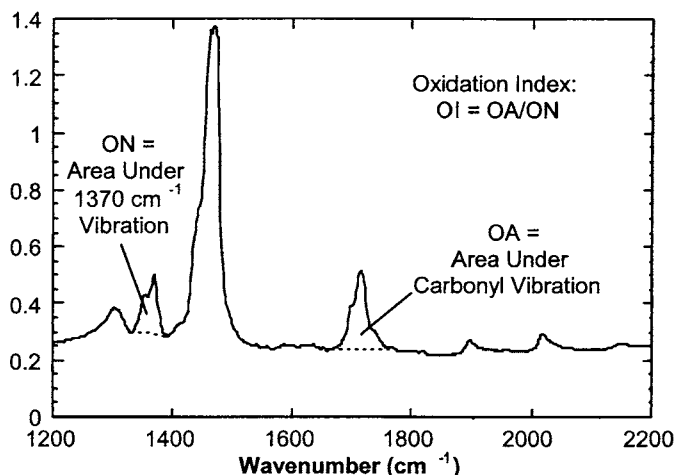


FIG. 1 Typical FTIR Spectra of Oxidized UHMWPE, Showing the Definition of an Area-Based Oxidation Index Based on Normalization Using the 1370-cm⁻¹ Peak

produce equivalent results. Too large an aperture can result in a loss of profile accuracy.

4.1.1.2 When a Fourier Transform Infrared (FTIR) spectrometer is used, a minimum of 32 scans shall be collected per spectrum.

4.1.1.3 The FTIR instrument and sample compartment should be purged with a moisture- and carbon-dioxide-free inert gas (for example, nitrogen, helium, or argon) to minimize spectral interference from these components.

4.2 Specimen Holder:

4.2.1 Equipment capable of accurately positioning the sample under the orifice in increments at the scale of the aperture dimensions.

4.3 Microtome:

4.3.1 Equipment capable of producing about 200-µm-thick slices (films) of a sample perpendicular to the articular surface or the surface of interest.

5. Procedure

5.1 Preparation of the Infrared Spectrometer:

5.1.1 Prepare the infrared spectrometer for collection of a transmission absorption spectrum from a thin film of the UHMWPE sample according to the manufacturer’s recommendations and the conditions described in Section 4 above.

5.1.2 Collect the sequence of spectra per 5.2 and 5.3.

5.2 Preparation of Test Specimen:

5.2.1 Using a microtome, or other appropriate device, prepare a thin slice of the sample about 200 µm thick.

5.2.2 The slice shall typically be taken near the center of the sample’s articular surface or the surface of interest.

5.2.3 The orientation of the slice shall typically be perpendicular to the articular surface or the surface of interest.

5.3 Configuration of the Test Specimen in the Spectrometer:

5.3.1 The test film (slice) shall be first configured in the spectrometer (after an appropriate background spectrum has been collected) such that the aperture is positioned over the first 200 µm of the film starting at the surface of interest.

5.3.2 Subsequent spectra shall be collected sequentially at increments matching the aperture size (that is, about 200 µm)

from the articular surface, or surface of interest, across the width of the film to the opposite surface.

5.3.2.1 Larger increments may be used; however, too large an increment size may result in a loss of profile accuracy.

6. Calculations

6.1 Oxidation Peak Area:

6.1.1 For each absorbance spectrum calculate the total area of the peak absorptions between 1650 and 1850 cm⁻¹ (see Fig. 1).

6.1.1.1 This is the area below the sample’s absorption curve and above the straight line baseline drawn between the same starting and ending points, namely, 1650 and 1850 cm⁻¹.

6.2 Normalization Peak Area:

6.2.1 For each absorbance spectrum, calculate the total area of the peak absorptions between 1330 and 1396 cm⁻¹ (see Fig. 1).

6.2.1.1 This is the area below the sample’s absorption curve and above the straight line baseline drawn between the same starting and ending points, namely, 1330 and 1396 cm⁻¹.

6.3 Oxidation Index (OI):

6.3.1 For each absorbance spectrum, calculate its OI by dividing the area of its oxidation peak (6.1) by the area of its normalization peak (6.2), as shown in Fig. 1.

6.4 Oxidation Index Depth Locator (DL):

6.4.1 Calculate the distance from the articular surface, or surface of interest (DL), for each spectrum and its corresponding OI from the following equation.

$$DL = 0.5(A) + n(S)$$

where:

A = the size of the aperture in micrometres in the step direction,

n = the number of steps (increments) the aperture had been moved from its initial location at the articular surface or surface of interest, and

S = the step (increment) size in micrometres.

6.5 Sample’s Oxidation Index Profile:

6.5.1 Construct a plot of a sample’s oxidation indices (OI) versus their corresponding depth locators (DL).

6.6 Surface Oxidation Index (SOI):

6.6.1 Calculate a sample’s SOI by calculating the average of the sample’s oxidation indices (OI) with depth locator (DL) values between 0 and 3000.

6.7 Bulk Oxidation Index (BOI):

6.7.1 Calculate a sample’s BOI by calculating the average of the sample’s oxidation indices (OI) corresponding to the center 500 mm of material.

7. Report

7.1 The report shall contain at least the following experimental details and results:

7.1.1 Material Information:

7.1.1.1 Resin type and resin lot number.

7.1.1.2 Consolidation method and manufacturer and manufacturer lot number.

7.1.1.3 Any special postconsolidation treatments, for example, HIPing, annealing, sterilization, cross-linking, stabilization, accelerated aging, and storage conditions.