



Standard Test Method for Evaluating Trans-Vinylene Yield in Irradiated Ultra-High Molecular Weight Polyethylene Fabricated Forms Intended for Surgical Implants by Infrared Spectroscopy¹

This standard is issued under the fixed designation F2381; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method describes the measurement of the number of trans-vinylene groups in ultra-high molecular weight polyethylene (UHMWPE) intended for use in medical implants. The material is analyzed by infrared spectroscopy.

1.2 This test method is based on Guide [F2102](#).

1.3 The applicability of the infrared method has been demonstrated in other literature reports. This particular method, using the intensity (area) of the C-H absorption centered at 1370 cm^{-1} to normalize for the sample's thickness, will be validated by an Interlaboratory Study (ILS) conducted according to Practice [E691](#).

1.4 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.5 *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 limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:²

[E691](#) Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

[E1421](#) Practice for Describing and Measuring Performance of Fourier Transform Mid-Infrared (FT-MIR) Spectrometers: Level Zero and Level One Tests

[F2102](#) Guide for Evaluating the Extent of Oxidation in

¹ This test method 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 For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

Polyethylene Fabricated Forms Intended for Surgical Implants

3. Terminology

3.1 Definitions:

3.1.1 *trans-vinylene index (TVI)*—a trans-vinylene index is defined as the ratio of the area of the absorption peak centered near 965 cm^{-1} to the area of the absorption peak centered near 1370 cm^{-1} .

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 TVI calculated.

3.1.3 *trans-vinylene index profile*—a trans-vinylene index profile is defined as the graphical representation of variation of the sample's trans-vinylene index with distance from its articular surface or the surface of interest. This is a plot of TVI versus DL. Typically, the graph will show the profile through the entire thickness of the sample.

4. Significance and Use

4.1 Published literature shows that the yield of radiolytic reactions that occur during radiation treatment increases with radiation dose level. Measurement of the products of these reactions can be used as an internal dosimeter.

4.2 Trans-vinylene unsaturations are formed during ionization treatment by abstraction of a hydrogen molecule, and to a lesser extent by the recombination of two adjacent alkyl free radicals that reside on the same chain.

4.3 Previous work generated calibration curves of trans-vinylene absorption area as a function of absorbed radiation dose, yielding a linear relationship for both gamma- and electron beam-irradiated polyethylene.

4.4 This data can be used to determine received dose as a function of position, assuming a calibration curve (TVI versus radiation dose level) is known for the particular material and radiation conditions used, and can be used to determine uniformity of dose level in irradiated polyethylene.

5. Apparatus

5.1 Infrared Spectrometer:

5.1.1 A calibrated infrared spectrometer capable of recording a transmission absorption spectrum over a minimum range of 900 to about 2000 cm^{-1} using about 200 μm -thick films at a resolution of 4 cm^{-1} and an aperture of approximately 200 by 200 μm for a rectangular aperture, or 200 μm diameter for a circular aperture.

5.1.1.1 Other modes of collection (that is, reflection, attenuated total reflection (ATR), and so forth) and aperture and sampling step 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.

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

5.1.1.3 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.

5.2 *Specimen Holder*—Equipment, such as an x - y table, capable of accurately positioning the sample under the FTIR aperture with a minimum resolution at the scale of the aperture dimensions.

5.3 *Microtome*—Equipment capable of producing films of thickness 200 μm or less of a sample perpendicular to the articular surface or the surface of interest.

6. Sampling, Test Specimens, and Test Units

6.1 Using a microtome, or other appropriate device, prepare a thin slice of the sample about 200 μm thick. If the detected signal from the FTIR is too weak with this thickness, a thicker sample may be used.

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

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

6.4 Waviness in the infrared spectrum caused by Fourier rippling on the lower wavelengths can interfere with the trans-vinylene absorbance at 965 cm^{-1} . This rippling is caused by internal reflection of the infrared beam, and can be avoided by lightly rubbing the sample film against 400 grit sandpaper until the film becomes translucent. This roughening procedure should be done slowly to avoid heating the film, and should be performed until the area under a single peak due to Fourier rippling is less than 10 % of the area under the trans-vinylene peak.

7. Preparation of Apparatus

7.1 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, Practice E1421, and the conditions described in 5.1.

8. Procedure

8.1 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.

8.2 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.

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

9. Calculation of Results

9.1 *Trans-vinylene Peak Area*:

9.1.1 For each absorbance spectrum, calculate the total area of the absorption peak centered near 965 cm^{-1} (see Fig. 1).

9.1.2 This area is the area below the sample's absorbance curve and above the straight baseline drawn between the same starting and ending points used in 3.1.1.

9.2 *Normalization of Peak Area*:

9.2.1 For each absorbance spectrum, calculate the total area of the absorption peak centered near 1370 cm^{-1} (see Fig. 2).

9.2.2 This area is the area below the sample's absorbance curve and above the straight baseline drawn between the same starting and ending points used in 3.1.1.

9.3 *Trans-vinylene Index (TVI)*—For each absorbance spectrum, calculate its TVI by dividing the area of its trans-vinylene peak (see 9.1) by the area of its normalization peak (see 9.2).

9.4 *Depth Locator (DL)*—Calculate the distance from the articular surface, or surface of interest, termed the depth locator (DL), for each spectrum and its corresponding TVI from the following equation:

$$DL = 0.5(A) + n(S) \quad (1)$$

where:

- A = the size of the aperture in micrometers 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 micrometers.

10. Report

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

10.1.1 *Material Information*:

10.1.1.1 Resin type and resin lot number,

10.1.1.2 Consolidation method and manufacturer and manufacturer lot number, and

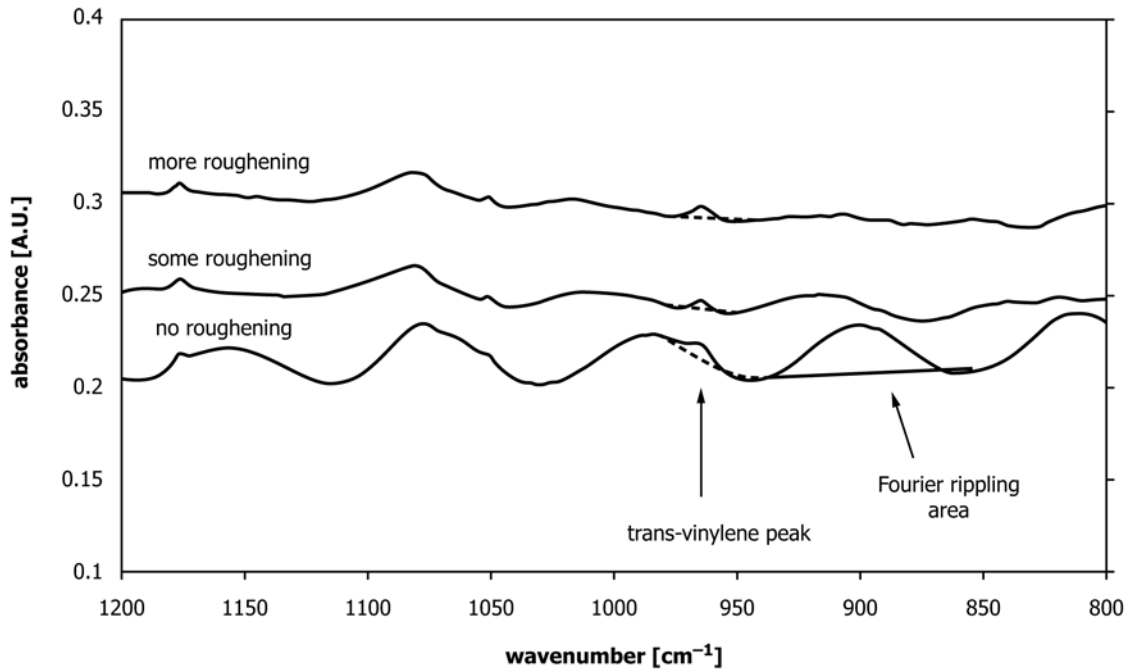
10.1.1.3 Any special post-consolidation treatments, for example, HIPing, annealing, sterilization, crosslinking, stabilization, accelerated aging, and storage conditions.

10.1.2 *Sample Information*:

10.1.2.1 Indicate whether the sample is an orthopedic implant or laboratory test specimen,

10.1.2.2 Articular surface or non-articular surface,

10.1.2.3 Test sample's original dimensions,



NOTE 1—Fourier rippling disappears with increased surface roughening. The original absorption peaks are unaffected by the surface roughening.

FIG. 1 FTIR Spectra of Thin UHMWPE Film Showing the Effects of Roughening on Fourier Rippling

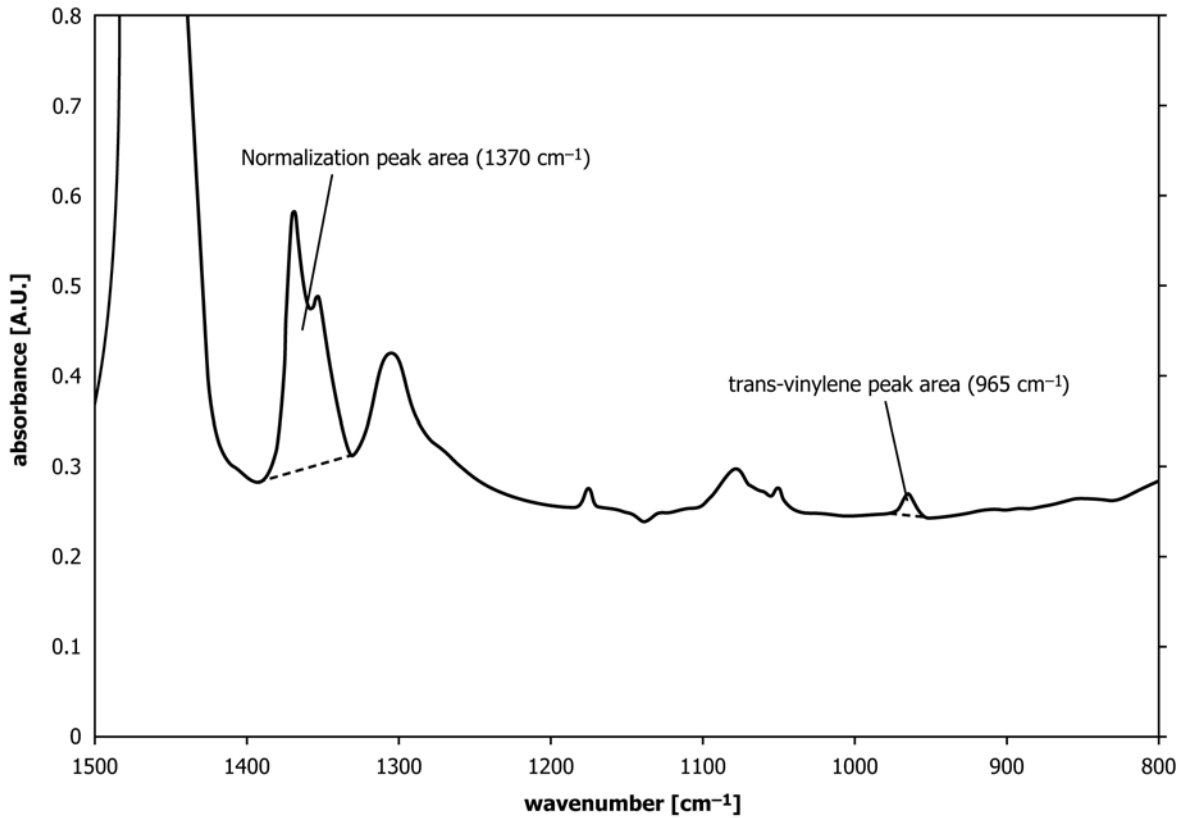


FIG. 2 Typical FTIR Spectrum of Radiation-Crosslinked UHMWPE, Showing the Definition of an Area-Based Trans-Vinylene Index Based on the Normalization Peak at 1370 cm^{-1}

10.1.2.4 Any special post-treatments of the original test sample, for example, annealing, sterilization, crosslinking, stabilization, accelerated aging, and storage conditions,

10.1.2.5 Test film thickness and total width, and

10.1.2.6 Any special post-treatments of the test films, for example, annealing, sterilization, crosslinking, stabilization, accelerated aging, and storage conditions.

10.1.3 *Spectrometer Information:*

10.1.3.1 Manufacturer and model number,

10.1.3.2 Analog or Fourier transform spectrometer, and

10.1.3.3 Aperture dimensions, profile step size, spectral resolution, and number of scans per spectrum.

10.1.4 *Data Analysis Information:*

10.1.4.1 Manual or by spectrometer's software algorithms.

11. Precision and Bias

11.1 Precision and bias data will be forthcoming following a round-robin test.

12. Keywords

12.1 crosslinking; FTIR (Fourier Transform Infrared); implant; trans-vinylene index; TVI; UHMWPE (ultra-high molecular weight polyethylene)

APPENDIX

(Nonmandatory Information)

X1. RATIONALE

X1.1 The extent of crosslinking present in orthopedic implant components made of UHMWPE has been shown to affect both mechanical properties and wear behavior. It is therefore important to have standard methods for assessing the degree of crosslinking of such materials.

X1.2 The method described herein is an adaptation of methods described in the literature, and of other ASTM standards.

X1.3 The intensity (area) of the trans-vinylene absorptions (-C=C-) centered near 965 cm^{-1} is related to the amount of crosslinking experienced by the material when exposed to ionizing radiation. The correlation between TVI and actual

received radiation dose depends on the nature of the irradiation conditions, for example, radiation source (gamma or electron beam), temperature, dose rate, and oxygen level. To determine radiation doses from TVI measurements, a calibration curve must be prepared for the specific radiation conditions used, using validated dosimeters (such as photochromic dyes) as an indicator of the delivered radiation dose.

X1.4 This test method is useful for assessing the uniformity of crosslinking in an irradiated component or pre-form.

X1.5 This test method does not specify desired levels of TVI to achieve optimal functional characteristics.

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