



Designation: D5477 – 18

# Standard Practice for Identification of Polymer Layers or Inclusions by Fourier Transform Infrared Microspectroscopy (FT-IR)<sup>1</sup>

This standard is issued under the fixed designation D5477; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This practice describes the techniques used for detecting two different polymer entities such as:

1.1.1 Abnormal specks or spots on a surface or in the film that are objectionable as defects and

1.1.2 Layers of different polymeric sheets commonly used as barrier films made by coextrusion.

1.2 This practice utilizes through-transmittance optical and infrared techniques.

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* Specific hazard statements are given in Section 7.

NOTE 1—There is no known ISO equivalent to this standard.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

## 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D883 Terminology Relating to Plastics

D1248 Specification for Polyethylene Plastics Extrusion Materials for Wire and Cable

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods.

Current edition approved Aug. 1, 2018. Published August 2018. Originally published as D5477 – 93. Last previous edition approved in 2011 as D5477 – 11. DOI: 10.1520/D5477-18.

<sup>2</sup> 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.

D1600 Terminology for Abbreviated Terms Relating to Plastics

E131 Terminology Relating to Molecular Spectroscopy

E168 Practices for General Techniques of Infrared Quantitative Analysis

E2015 Guide for Preparation of Plastics and Polymeric Specimens for Microstructural Examination

IEEE/ASTM SI-10 Standard for Use of the International System of Units (SI):The Modern Metric System

## 3. Terminology

3.1 *Definitions:*

3.1.1 For definitions of the terms used in this practice, refer to Terminologies D883 and D1600.

3.1.2 For units, symbols, and abbreviations used in this practice, refer to Terminology E131 or IEEE/ASTM SI-10.

## 4. Significance and Use

4.1 A speck will ultimately cause a failure to occur by virtue of its appearance in a film or by the decrease in electrical or mechanical properties in the polymer substrate (see Specification D1248).

4.2 The analysis of composite layers for barrier purposes by microscopic Fourier transform infrared spectroscopy (FT-IR) can indicate the adequacy of the barrier tape or indicate why a barrier may be defective (a missing layer or hole in the layer or poor coextrusion practice). Fig. 1 represents a typical multi-layer film.

## 5. Apparatus

5.1 *FT-IR Spectrophotometer*, with nominal 4-cm<sup>-1</sup> resolution (see Practices E168).

5.2 *Microsampling Accessory*, accommodated into the FT-IR for microscopic infrared analysis, with nominal 6.25- $\mu$ m resolution in the infrared mode.

5.3 *Optical Microscope*, equipped with cross-polarized light and phase contrast accessories. May be incorporated into the infrared microsampling accessory.

5.4 *Hot-Stage*, with temperature readout, is accommodated into the optical microscope or microsampling accessory.

5.5 *Microtome*, capable of <25  $\mu$ m slices  $\pm$ 2.5  $\mu$ m.

\*A Summary of Changes section appears at the end of this standard

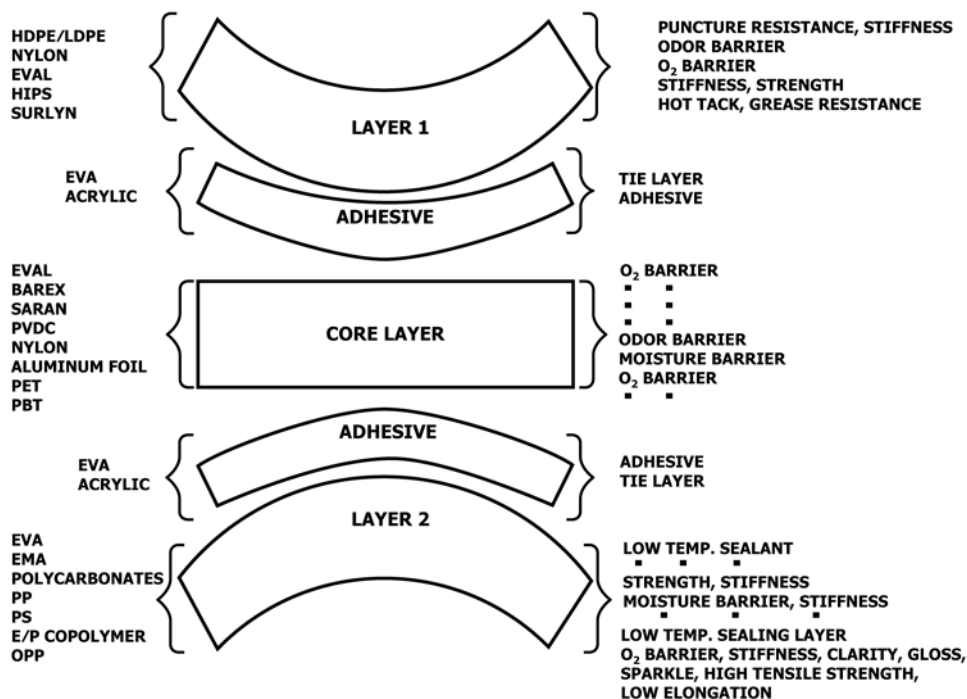


FIG. 1 Position and Function of Materials in a Typical Multilayer Film

## 6. Material

6.1 *Stiff plastic* at 25°C, 1.25 mm thick and large enough to hold sample (for example, ABS, boPET).

6.2 *Cyano-acrylate adhesive*.

6.3 *Thermoset material* for encapsulating sample (for example, 2-part epoxy, acrylic).

6.4 *Glass microscope slide* to support sample slice during inspection.

## 7. Hazards

7.1 The FT-IR spectrophotometer contains a laser. To avoid eye injury, do not stare directly into the laser beam.

7.2 Use gloves when samples are prepared. The cyano-acrylate adhesive will attach itself to the fingers and skin. Take care to prevent this from occurring.

7.3 Avoid burns when handling microscopic slides with the hot-stage.

## 8. Specimen Preparation

8.1 It is necessary to microtome a thin cross section at right angles to the surface of the film or sample in order to conveniently observe the individual layers or the interior of the speck.

8.2 Samples that do not deflect can be microtomed into the required thin sections as received.

8.3 Flexible samples must be supported during sectioning. Two common support techniques are shown, for flexible samples, in Fig. 2. On the left, a stiff, flat plastic is used for the support. A cyano-acrylate adhesive quickly bonds the flexible sample to the flat plastic support. On the right of Fig. 2, the sample is supported and cured inside a thermoset compound, such as a two-part epoxy. (See Guide E2015.)

8.4 The entire sandwich is then microtomed in a direction normal to the sample surface. Slice thickness typically of 25-50 μm provide satisfactory transmission of optical and infrared light.

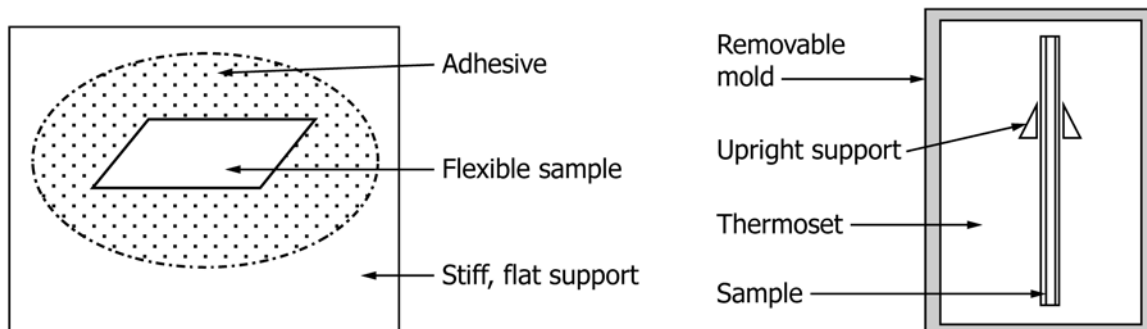


FIG. 2 Sandwich for Thin Films