



Designation: ~~D5477 – 11~~ D5477 – 18

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

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 (ϵ) 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 ~~as follows:~~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~~ safety, health, and ~~health~~ 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:*²

[D883 Terminology Relating to Plastics](#)

[D1248 Specification for Polyethylene Plastics Extrusion Materials for Wire and Cable](#)

[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 some 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](#).

3.1 *Definitions:*

3.1.1 For definitions of the terms used in this practice, refer to Terminologies [D883](#) and [D1600](#).

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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](#)).

¹ 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 Dec. 1, 2011; Aug. 1, 2018. Published December 2011; August 2018. Originally published as D5477 – 93. Last previous edition approved in 2002/2011 as ~~D5477 – 02~~D5477 – 11, which was withdrawn in January 2011 and reinstated in December 2011. DOI: 10.1520/D5477-11; DOI: 10.1520/D5477-18.

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.

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

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 multilayer film.

5. Apparatus

- 5.1 *FT-IR Spectrophotometer*, with nominal 4-cm⁻¹ resolution (see Practices E168).
- 5.2 *Microsampling Accessory*, accommodated into the FT-IR for microscopic infrared and visible light analysis, with nominal 6.25-µm resolution ~~on~~ 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*, ~~which with temperature readout,~~ is accommodated into the optical ~~microscope;~~ microscope or microsampling accessory.
- 5.5 *Microtome*.
- 5.6 *Surlyn™ Ionomer*, 1.25-mm thick.⁴
- 5.7 *Cyano-Acrylate Adhesive*.
- 5.5 *Micrometer, Microtome*, capable of measuring to ±0.0025 mm (0.0001 in., 0.1 mil). <25 µm slices ±2.5 µm.

6. Material

- 6.1 *Cyano-Acrylate Adhesive*. 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 Use gloves when plaques are prepared using a heated press. Take care to avoid burns when handling microscopic slides with the hot plate.
- 7.1 The optical bench of the FT-IR spectrophotometer contains a laser. To avoid eye injury, do not stare directly into the laser beam.

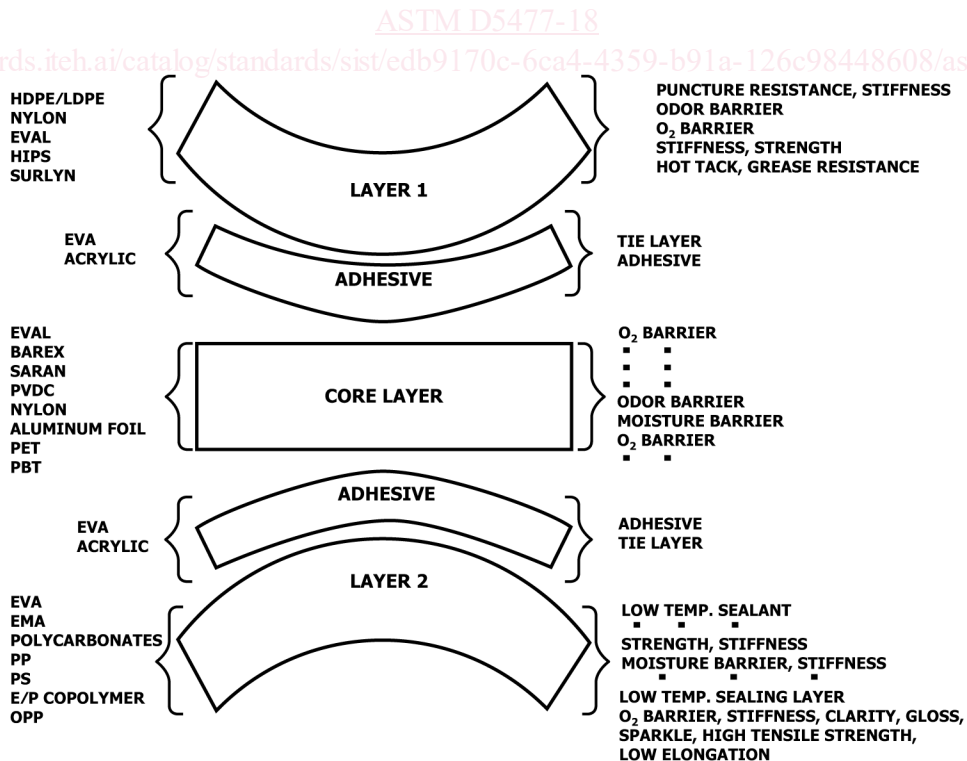


FIG. 1 Position and Function of Materials in a Typical Multilayer Films