



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](#)

[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  $4\text{-cm}^{-1}$  resolution (see Practices [E168](#)).

5.2 *Microsampling Accessory*, accommodated into the FT-IR for microscopic infrared analysis, with nominal  $6.25\text{-}\mu\text{m}$  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  $<25\ \mu\text{m}$  slices  $\pm 2.5\ \mu\text{m}$ .

<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.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto: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

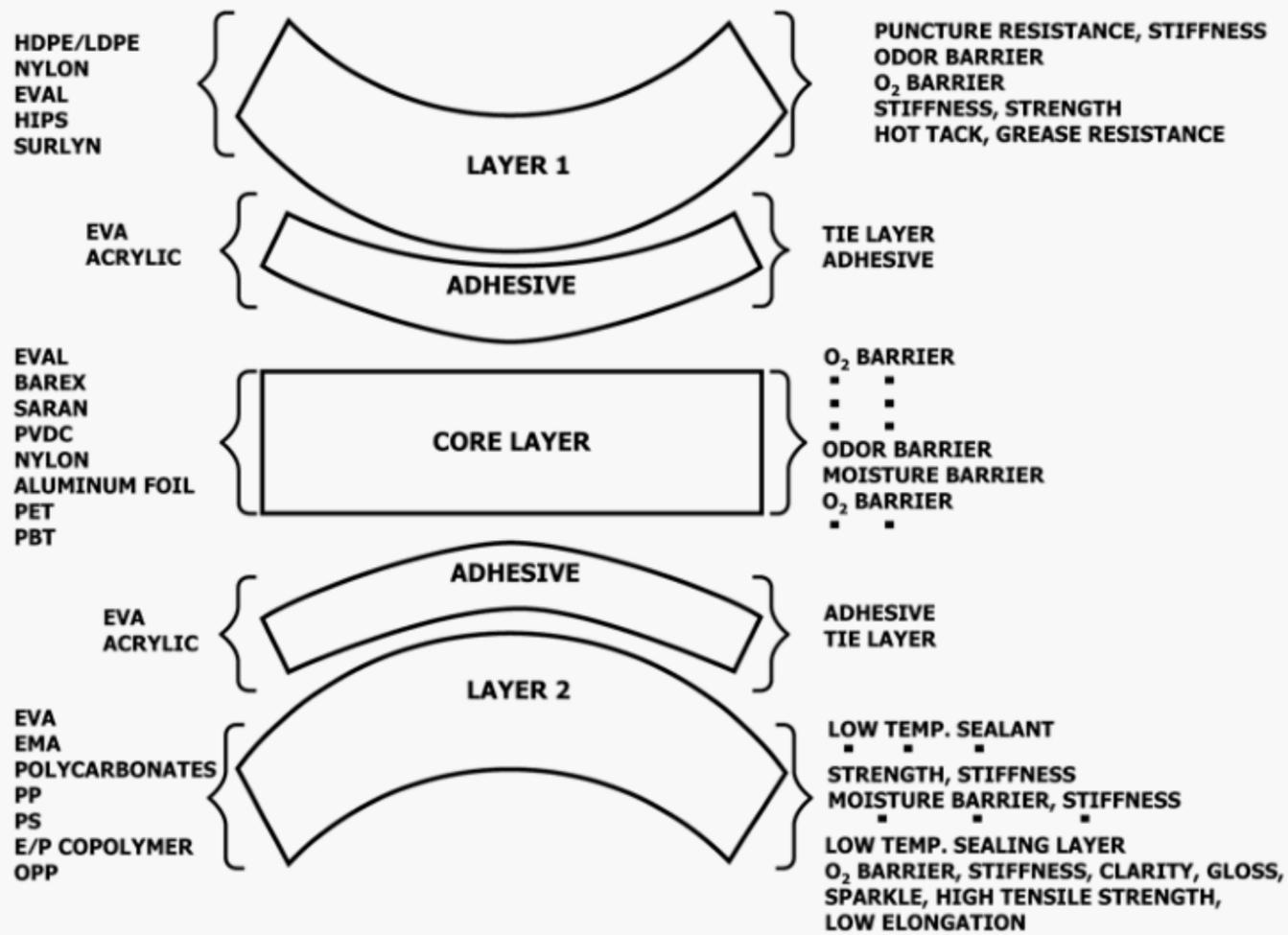


FIG. 1 Position and Function of Materials in a Typical Multilayer Film

## 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.

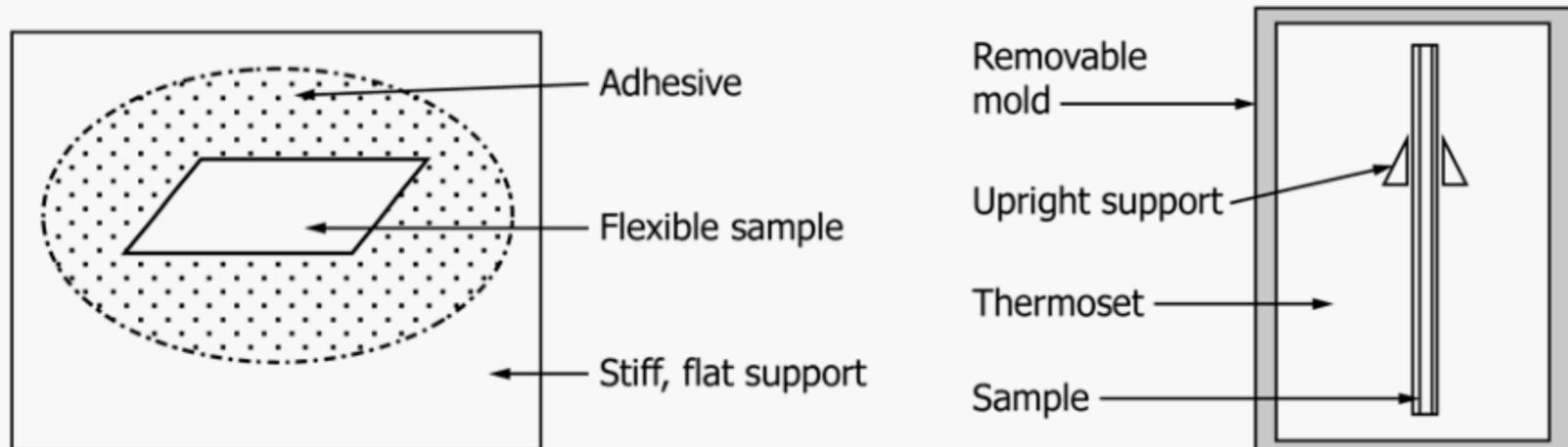


FIG. 2 Sandwich for Thin Films

9. Procedure

9.1 Optical Microscopy

9.1.1 The first step is to observe the sample visually in an optical microscope. The fundamentals of optical microscopy and sample preparation have been discussed in detail elsewhere.<sup>3</sup>

9.1.2 The key to optical microscopy analysis is the sample preparation. A 25 to 50- $\mu\text{m}$  thick section allows the visible and IR radiation to go through the section. A section with very few knife marks is required. A knife mark is a gouge created in the section by a defect in the microtome knife. Under cross-polarized light, knife marks will confuse and distort the boundaries of an inhomogeneity or layers in a multilayer specimen.

9.1.3 Once suitable sections have been collected, they are viewed in the optical microscope under cross-polarized light. In pigmented materials, it is also necessary to view the materials in uncrossed polarized light. Differences in contrast between inhomogeneities of layers develop in these situations due to differences in the intrinsic birefringence of the resins, thermal and stress history, and pigment concentration. The differences in contrast generally define material boundaries.

The areas of interest may then be photographed and measurements made to quantify the dimensions of inhomogeneities or layers.

9.1.4 Since differences in contrast may arise from factors other than chemical differences, it is necessary to conduct a hot-stage microscopic analysis. In the hot-stage, each of the different materials is located when their birefringence disappears in the microscope under cross-polarized light, in accordance with their melting temperature. The hot-stage is heated at a rate of up to 10°C/min. It is advisable to recrystallize the polymers and recheck the melting points since processing history may influence softening point upon first melting in the hot-stage.

9.2 Infrared Microscopy—

9.2.1 The infrared microscope permits the identification of inclusions and multilayer films down to diameters of 10  $\mu\text{m}$ .

9.2.2 This procedure uses the microscope to locate and analyze small areas of a specimen. The microscope mates with a FT-IR spectrometer. Infrared spectra may then be obtained for each small area of interest.

9.2.3 A schematic of an instrument capable of performing these tasks is shown in Fig. 3. This microscope accessory is very useful for locating and then identifying small inhomogeneities, such as a particle, oxidized zone, or layer in a plastic film. Its use is unique for the identification of the

<sup>3</sup> Chamot, E. M., and Mason, C. W., *Handbook of Chemical Microscopy*, John Wiley and Sons, Inc., New York, NY, 1985.

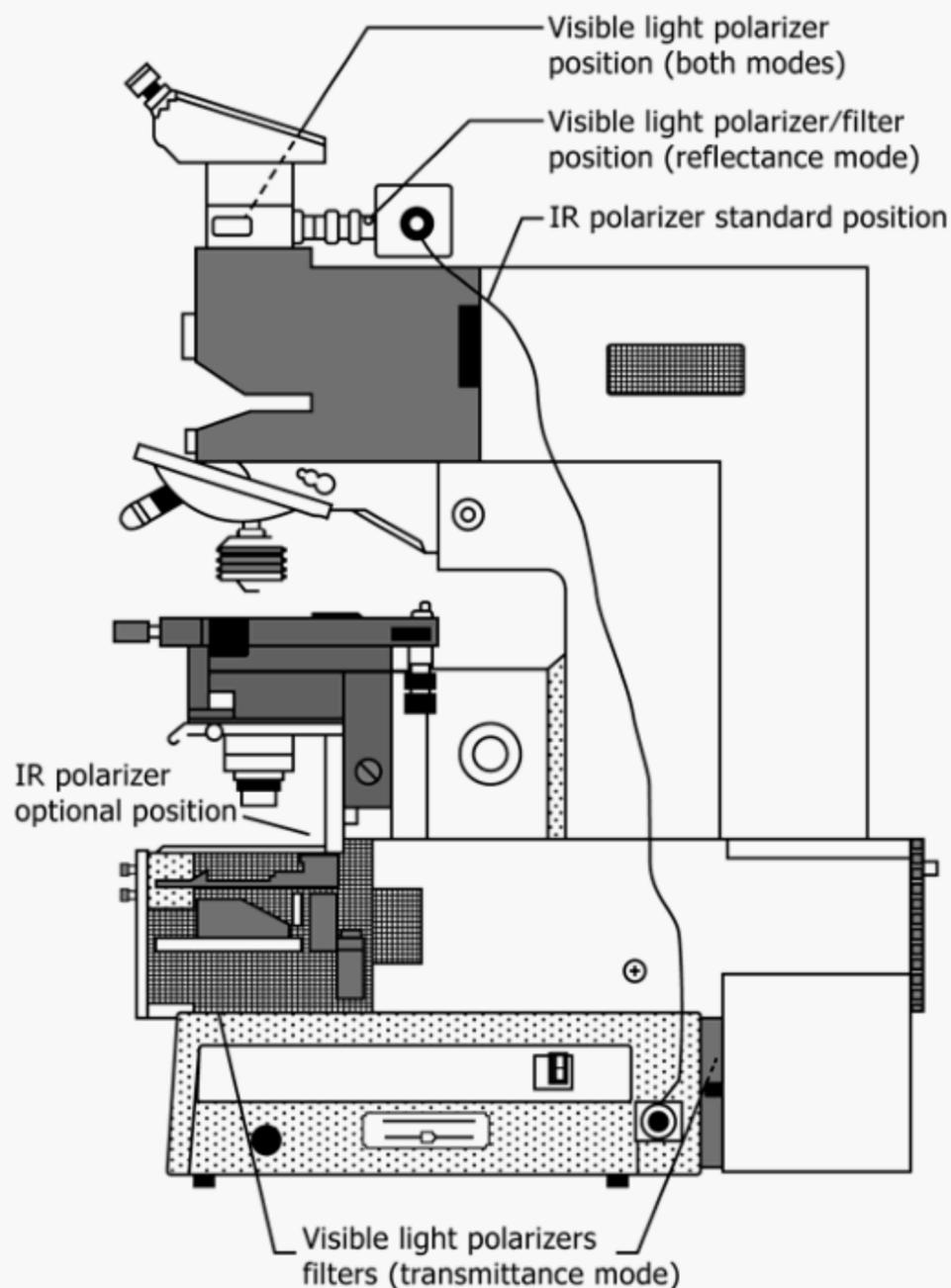


FIG. 3 Schematic of An Infrared Microsampling Accessory with Viable Lenses and Polarizers Built In

layers in a multilayer film or sheet. The area of interest is chemically identified by the interpretation of the infrared spectra obtained for that area.

9.2.4 The infrared microscope is focused on the area of interest within the section. This typically requires using apertures to isolate the area. All spectra are recorded at  $4\text{ cm}^{-1}$  resolution. Acceptable signal-to-noise ratios are obtained by the co-adding at least 100 spectra.

9.2.5 Many unresolved peaks in the spectrum indicate sections need to be thinner, assuming apertures, gain, and other parameters optimized.

9.2.6 A well-resolved spectrum, along with the estimated melting point, may now be used to aid in chemical identification.

## 10. Report

10.1 Report the following information:

- 10.1.1 Preparation method,
- 10.1.2 Material(s) identification,
- 10.1.3 Method(s) of determination,
- 10.1.4 The date of the test.

## 11. Keywords

11.1 Fourier transform infrared spectroscopy; microscopy; microsampling; cross-polarized

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