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An American National Standard

Standard Guide for Fourier Transform Infrared Spectroscopy in Forensic Tape Examinations[1]

This standard is issued under the fixed designation E3085; 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 Infrared spectroscopy (IR) is a valuable method for the identification and comparison of pressure sensitive tapes (**1-20**).[2] This guide provides basic recommendations and information about infrared spectrometers and accessories, with an emphasis on sampling techniques specific to pressure sensitive tape examinations. The particular method(s) employed by each examiner or laboratory will depend upon available equipment, examiner training, sample suitability, and sample size.

1.2 This guide is intended for examiners with a basic knowledge of the theory and proficiency in the use of infrared spectroscopy as well as experience in the handling and forensic examination of pressure sensitive tapes. Further, this guide is to be used in conjunction with a broader analytical scheme (**21-23**).

1.3 Disclaimer: This guide offers a set of instructions for performing one or more specific operations. This standard cannot replace knowledge, skill, or the ability acquired through appropriate education, training, and experience and should be used in conjunction with sound professional judgment.

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:*[3]

E131 Terminology Relating to Molecular Spectroscopy

E573 Practices for Internal Reflection Spectroscopy

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

E1492 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory

E2224 Guide for Forensic Analysis of Fibers by Infrared Spectroscopy

3. Terminology

3.1 *Definitions*—For terms relevant to molecular spectroscopy, refer to Terminology E131.

3.1.1 *background, n*—the signal produced by the entire analytical system apart from the material of interest.

3.1.2 *elastomer, n*—the polymeric backbone of a pressure sensitive adhesive imparting elastic properties, for example, rubber.

3.1.3 *fiber reinforcement, n*—the fabric portion of some pressure sensitive adhesive tapes; also referred to as *scrim*.

3.1.4 *filler/extender, n*—an inorganic material that is added to a tape to modify a physical property or reduce cost.

3.1.5 *low e-glass, n*—a dichroic mirror that is coated with an IR reflective surface.

3.1.5.1 *Discussion*—Such glass is suitable for use as a sample support when performing IR reflection techniques.

3.1.6 *meaningful difference, n*—a feature or property of a sample that does not fall within the variation exhibited by the comparison sample, considering the limitations of the sample or technique, and therefore indicates the two samples do not share a common origin.

3.1.6.1 *Discussion*—The use of this term does not imply the formal application of statistics.

3.1.7 *pressure-sensitive adhesive, n*—a viscoelastic material which, in solvent-free form, remains tacky and will adhere instantaneously to most solid surfaces with the application of very slight pressure.

3.1.8 *pressure-sensitive adhesive (PSA) tape, n*—a combination of a pressure sensitive adhesive with a continuous flexible backing (for example, cloth, paper, metal, or plastic) or with a backing and release liner.

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[2] The boldface numbers in parentheses refer to a list of references at the end of this standard.

[3] 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.

3.1.8.1 *Discussion*—Use of the word "tape" in this guide refers to PSA tapes and their components.

3.1.9 *release coat, n*—an inert material with a low surface energy, applied to a backing film on the side opposite the adhesive, that provides ease of unwind and prevents delamination or tearing.

3.1.10 *tackifier, n*—solid resins added to the adhesive base polymer to impart the necessary tack and adhesion.

4. Summary of Guide

4.1 This guideline covers the analysis of tape backings and adhesives by infrared spectroscopy. It can be applied to a wide range of infrared spectrometers and accessory configurations.

4.2 For the infrared analysis of the fiber reinforcement, refer to Guide E2224.

5. Significance and Use

5.1 This guide is designed to assist an examiner in the selection of appropriate sample preparation methods for the analysis, comparison, and identification of pressure sensitive adhesive (PSA) tapes. If no meaningful differences are noted between the known and unknown samples regarding physical appearance or measurements, then IR spectroscopy should be the next step in the analytical scheme.

5.2 Infrared spectroscopy can provide molecular information regarding major organic and inorganic components. For various reasons, components in lesser amounts are typically more difficult to identify unequivocally. Reasons for this include interference of the absorption bands of the major components with the less intense bands of minor components and sensitivity issues whereby the minor components are present at concentrations below the detection limits of the instrument.

5.3 Infrared spectroscopy can be used to obtain spectra for elucidation of the chemical composition of a tape and for comparison of two or more tape samples. When used for spectral comparisons, the objective is to determine whether any meaningful differences exist between the samples.

6. Sample Handling

6.1 The general collection, handling, and tracking of samples shall meet or exceed the requirements of Practice E1492.

6.2 The work area and tools used for the preparation of samples shall be free of any materials that could transfer to the sample.

6.3 When analyzing difficult samples (for example, adhesive residue, dirty samples, limited sample size, or inhomogeneous samples), care shall be taken in sampling each available tape layer (that is, film backing, adhesive, fiber reinforcement if present) as well as in selection of appropriate analytical conditions. An attempt shall be made to remove extraneous material from the specimen before analysis. In order to ensure reproducibility and evaluate intra-sample variations, repeat analysis of samples is recommended. The number of replicates is dependent on factors such as sample size and condition and is evaluated on a case-by-case basis.

6.4 If necessary, the tape backing can be cleaned with an appropriate solvent (for example, methanol or hexane). Alternatively, residue can be removed by gentle scraping of the surface. An adhesive sample can be obtained by exposing and sampling the underlying portion.

6.5 The infrared analysis of tapes can be carried out using either transmission or reflection techniques. These measurements can be taken with a variety of equipment configurations and accessories, the most common being the use of Attenuated Total Reflection (ATR) or an infrared microscope. However, the use of an ATR requires a larger sample size.

6.6 Attenuated Total Reflection (ATR), also known as Internal Reflection Spectroscopy (IRS), is described in Practices E573. For forensic tape analysis, it offers a rapid approach to sampling a tape backing and the adhesive as virtually no sample preparation is necessary. Single or multiple reflection elements may be used depending on the amount of area available for sampling. When only a small area is available, a single reflection element is desirable to avoid contamination.

6.7 Transmission microspectroscopy is possible by sampling a small portion of the tape component (backing, or adhesive) and analyzing it as a thin film.

6.8 A diamond anvil cell may be used in the bench with a beam condenser or placed under the microscope accessory to analyze both the backing and the adhesive portions of tape.

6.9 Tackifiers or plasticizers can be extracted from the adhesive or backing using a mild solvent such as hexane or acetone (**20**), for subsequent analysis as a thin film using transmission mode.

6.10 Samples being compared shall be prepared and analyzed in the same manner.

7. Analysis

7.1 A standard mid-IR range Fourier transform infrared (FTIR) spectrometer is acceptable to conduct the necessary analyses. Detector cutoff no higher than 750 cm^{-1} is recommended. A mid-infrared FTIR spectrometer with an extended range down to 200 cm^{-1} is advantageous for the classification and comparison of inorganic fillers and pigments.

7.2 *Instrument Parameters:*

7.2.1 *Performance and Verification*—It is essential that instrument performance and verification be evaluated routinely (for example, monthly or before use if used less frequently).

7.2.2 The preferred performance evaluation method is in accordance with Practice E1421. In brief, this includes evaluation of the system throughput, single-beam spectrum, 100% T line, and polystyrene reference spectrum.

7.2.3 Sample and background scans shall be run under the same instrument conditions (for example, aperture size).

7.2.4 Typically, 16 to 256 scans are collected at a resolution of 4 cm^{-1} or less.

7.2.5 When comparing spectra, the data shall be displayed in the same units (for example, Absorbance units, % Transmission, % Reflectance).