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

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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).

7.3 *Main Bench – Transmission:*

7.3.1 Transmission methods generally require more extensive sample preparation. The sample shall be thin enough not to over-absorb. For transmission data viewed in % transmittance, spectral peaks optimally should not fall below 10 % T. For spectra displayed in absorbance, the maximum absorbance optimally should be 1.0 or less. This typically requires a sample thickness of approximately 5–10 µm.

7.3.2 Sample preparation techniques that may be employed for transmission analysis in the main bench include backing or adhesive pressed in a diamond cell, a thin backing sample stretched over an aperture, or adhesive deposited onto an alkali halide pellet (for example, KBr, NaCl, AgCl).

7.4 *Main Bench – ATR:*

7.4.1 ATR methods lend themselves to conducting the examination of the tape intact. Since ATR is a surface technique it is necessary to remove any extraneous material from the area to be examined.

7.4.2 ATR is also useful in the analysis of duct tape backings for layer structure evaluation as a complementary technique to manual cross section. The adhesive is removed with an appropriate solvent (for example, hexane), and the backing is analyzed on both sides. Additional analysis may be conducted on middle layers as desired. The spectra are then compared.

7.5 *Microscope Accessory:*

7.5.1 The use of a microscope accessory is preferred for very small samples. It is important to note that there is a tradeoff between sensitivity and spectral range with the MCT detectors. The low energy cut off for most detectors is in the range of 700–450 cm^{-1}. The smallest apertures particularly limit the energy from the longer wavelengths (smaller wavenumbers) reaching the detector due to diffraction. Heterogeneity issues are also more pronounced when using very small apertures.

7.5.2 The microscope attachment permits the analysis of multiple samples placed on an appropriate support material. The method affords the advantage of viewing the sample optically and choosing the most appropriate area for analysis.

7.5.3 Spectral measurements using an FTIR microscope can be obtained in transmission, reflection, or ATR mode.

7.6 *Microscope Accessory – Transmission:*

7.6.1 Transmission measurements are commonly used because they generate spectra with fewer artifacts than other sampling modes. However, transmission methods generally entail more sample preparation than reflection techniques. The tape sample shall be rendered thin enough not to over-absorb. Samples can be placed directly over a small aperture for analysis or placed on an appropriate alkali halide plate. This typically requires a sample thickness of approximately 3–5 µm.

7.6.2 A diamond cell can also be used as a sample support medium with the FTIR microscope. The adhesive can be smeared on one of the diamond faces. A thin peel of a tape backing is placed onto one of the diamond faces, the second diamond is positioned on top, and pressure is applied. One diamond is typically removed prior to analysis once the sample has been compressed. This leaves the thin compressed film adhering to one of the diamond faces.

7.7 *Microscope Accessory – Reflection:*

7.7.1 If samples are analyzed directly on an infrared light reflecting surface (for example, low e-glass or gold mirror), the reflection mode can be used to produce spectra mimicking double-pass transmission spectra. The technique is sometimes referred to as “transflection” or “reflection/absorption.” Samples need to be approximately half the thickness of an optimum transmission sample.

7.7.2 The FTIR microscope can also be used in the specular reflection mode; however, it is not useful for tape unless the surface of the sample is highly reflective.

7.8 *Microscope Accessory – ATR:*

7.8.1 ATR objectives are available for infrared microscopes. Applying consistent pressure to each sample can mitigate spectral variations. Intra-sample variations can result from sample heterogeneity; therefore, multiple samplings shall be conducted as feasible.

8. Classification, Comparison, and Interpretation

8.1 Classification of commonly encountered tape components is based on the interpretation of characteristic infrared absorption bands.

8.1.1 Depending on the condition of the tape and the concentration of the material, tape components can contain the following:

8.1.1.1 *Backing*—Polymer film, plasticizers, fillers, extenders, flame retardants.

8.1.1.2 *Adhesive*—Elastomer, tackifiers, fillers, extenders.

8.1.1.3 *Release Coating.*

8.1.1.4 *Fiber Reinforcement.*

8.2 Classification of a tape component may be achieved by evaluating the absorption bands present in the spectrum with respect to band position, band shape, and band intensity (**12**). After evaluating the absorption bands, interpretation and classification of a spectrum can be accomplished through comparison of the collected data to spectra of reference materials, use of flow charts, and published findings. Spectral libraries of known materials can also be used to characterize the components present in the tape.

8.3 Tape component classification can be hindered by the presence of certain pigments. An alternative analytical technique, such as pyrolysis-gas chromatography (Py-GC), may be used to elucidate the classification (**6, 7**).

8.4 Component classification can be difficult in contaminated tapes. Contributions from any materials co-mingled with the adhesive shall be considered.

8.5 Comparison of known and questioned evidence can be conducted with both spectra displayed in transmittance or absorbance, although certain information can be more readily observed in one form or the other.

8.5.1 There are a number of factors to consider when assessing whether or not spectra can be distinguished from one another: the presence or absence of absorption bands, their positions, shapes, and relative intensities.

8.5.1.1 For spectra that cannot be distinguished from one another, characteristic absorption bands observed in one spectrum are also present in the comparison spectrum. The position of the absorption bands should have reasonable agreement with each other and is somewhat dependent on the shape of the absorption band. The positions of corresponding peaks in two or more spectra should be within ± 5 cm^{-1}. Additionally, the absorption bands should have comparable relative intensities and shapes for the spectra being compared.

8.5.1.2 If subtle differences are noted between questioned and known items, where possible, collect additional spectra to demonstrate whether the differences are repeatable and therefore meaningful. The number of additional spectra collected is predicated by several things: the amount of sample present, the hetero-/homogeneity of the material, typical spectral variation observed in similar materials, etc. Therefore, the number of replicates shall be determined on a case-by-case basis.

8.6 Spectra are dissimilar if they contain one or more meaningful differences (for example, absence or presence of constituents, reproducible intensity differences).

8.7 Spectra cannot be distinguished if they contain no meaningful differences (for example, comparable constituents, reproducible intensities).

8.8 A spectral comparison is inconclusive if no determination can be made as to whether observed differences are meaningful (for example, peaks are not well resolved, sample condition).

9. Documentation

9.1 When making comparisons of tape samples, the analyst's assessment of the IR spectra shall be documented.

9.2 For chemical identification of PSA tape components, the positions of the absorption bands according to wavelength or wavenumber and their relative intensities shall be compared to those of known reference spectra. It is desirable to supplement the identification by other methods such as polarized light microscopy (PLM), Py-GC, scanning electron microscopy with energy dispersive spectroscopy (SEM/EDS), X-ray fluorescence (XRF), or X-ray diffraction (XRD).

9.3 Case notes shall include a copy of the instrumental data that was used to reach a conclusion. All paper and electronic copies that are retained as part of the case file shall include a unique sample designation, the operator's name or initials, and the date of analysis.

9.4 A description of the evidence analyzed by IR, the method of sample preparation, the analytical instrumentation used, mode of operation (transmission, ATR, etc.), and its operating parameters shall be included in the case notes or in the procedural manuals.

10. Keywords

10.1 adhesive; analysis; backing; fiber reinforcement ; forensic examinations; infrared spectroscopy; pressure sensitive adhesive (PSA) tape

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