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Standard Guide for Forensic Analysis of Fibers by Infrared Spectroscopy[1]

This standard is issued under the fixed designation E2224; 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 (IR) spectroscopy is a valuable method of fiber polymer identification and comparison in forensic examinations. The use of IR microscopes, coupled with Fourier transform infrared (FTIR) spectrometers, has greatly simplified the IR analysis of single fibers, thus making the technique feasible for routine use in the forensic laboratory. This guide provides basic recommendations and information about IR spectrometers and accessories, with an emphasis on sampling techniques specific to fiber 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 practice of IR spectroscopy, as well as experience in the handling and forensic examination of fibers. In addition, this guide is to be used in conjunction with a broader analytical scheme.

1.3 If polymer identification is not readily apparent from optical data alone, an additional method of analysis, such as microchemical tests, melting point, IR spectroscopy, Raman spectroscopy, or pyrolysis gas chromatography, should be used. An advantage of IR spectroscopy is that the instrumentation is readily available in most forensic laboratories and the technique is minimally destructive.

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 cannot replace knowledge, skills, or abilities acquired through education, training, and experience and is to be used in conjunction with professional judgment by individuals with such discipline-specific knowledge, skills, and abilities.*

1.6 *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.*

1.7 *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]

D123 Terminology Relating to Textiles
E131 Terminology Relating to Molecular Spectroscopy
E1421 Practice for Describing and Measuring Performance of Fourier Transform Mid-Infrared (FT-MIR) Spectrometers: Level Zero and Level One Tests
E1459 Guide for Physical Evidence Labeling and Related Documentation
E1492 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Science Laboratory
E2228 Guide for Microscopical Examination of Textile Fibers

3. Terminology

3.1 *Definitions*—For definitions of terms used in this guide, refer to Terminologies D123 and E131.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *aperture, n*—an opening in an optical system that controls the amount of light passing through a system.

3.2.2 *attenuated total reflection (ATR), n*—a method of spectrophotometric analysis based on the reflection of energy at the interface of two media which have different refractive indices and are in intimate contact with each other.

3.2.3 *background, n*—apparent absorption caused by anything other than the substance for which the analysis is being made. **E131**

3.2.4 *cellulosic fiber, n*—fiber composed of polymers formed from glucose subunits.

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[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.

3.2.5 *delustrant, n*—a pigment, usually titanium dioxide, used to dull the luster of a manufactured fiber. **E2228**

3.2.6 *diffraction, n*—phenomenon that arises as a result of passing radiation through the "lens" of the microspectrometer and past the edges of objects such as apertures and the specimen. It causes radiation to deviate from its usually straight line causing blurring of what should be sharp images (**1**).[3]

3.2.7 *generic class, n*—as used with textile fibers, a grouping having similar chemical compositions or specific chemical characteristics. **D123**

3.2.7.1 *Discussion*—A generic name applies to all members of a group and is not protected by trademark registration. Generic names for manufactured fibers include, for example, rayon, nylon, and polyester. Generic names used in the United States for manufactured fibers were established as part of the Textile Fiber Products Identification Act enacted by Congress in 1954 (**2**).

3.2.8 *interference fringes, n*—the pattern that results from constructive and destructive interference of light waves.

3.2.9 *man-made fiber, n*—a class name for various genera of filament, tow, or staple produced from fiber-forming substances which are chemically synthesized or modified.

3.2.10 *manufactured fiber, n*—a class name for various genera of filament, tow, or staple produced from fiber-forming substances which can be (*1*) polymers synthesized from chemical compounds, (*2*) modified or transformed natural polymers, or (*3*) glass.

3.2.11 *meaningful difference(s), 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. The use of this term does not imply the formal application of statistical tests.

3.2.11.1 *Discussion*—The evaluation of variation is typically based on the visual comparison of spectral data.

3.2.12 *mid-infrared, n*—pertaining to the IR region of the electromagnetic spectrum with wavelength range from approximately 2.5 to 25 µm (wavenumber range approximately 4000 to 400 cm^{-1}).

3.2.13 *sub-generic class, n*—a group of fibers within a generic class that share the same base-polymer composition; sub-generic names include, for example, nylon 6 and nylon 6,6.

4. Summary of Guide

4.1 This guide covers the collection and comparison of IR absorption spectra obtained from fibers and can be applied to a wide range of IR spectrometers and accessory configurations. This guide is not meant to be the first step in the process of a fiber examination (**3**).

4.2 This analytical guide focuses on the identification of manufactured textile fibers (with the exception of inorganic fibers). Although natural fibers may also be analyzed by IR spectroscopy, light microscopy is the primary method for the identification of natural fibers.

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 fibers using IR spectroscopy. IR spectroscopy may provide additional compositional information than is obtained using polarized light microscopy alone. The extent to which IR spectral comparison is conducted will vary with specific sample and case evaluations.

5.2 IR analysis should follow visible and fluorescence comparison microscopy, polarized light microscopy, and ultraviolet (UV)/visible spectroscopy. If no meaningful differences are noted between the known and unknown samples in optical properties, then IR spectroscopy may be the next step in the analytical scheme.

NOTE 1—IR analysis generally follows the aforementioned techniques since sample preparation (for example, flattening) irreversibly changes fiber morphology.

5.3 IR spectroscopy should be conducted before dye extraction for chromatography due to the semi-destructive nature of the extraction technique. Because of the large number of sub-generic classes, forensic examination of acrylic and modacrylic fibers is likely to benefit significantly from IR spectral analysis (**4**). Useful distinctions between subtypes of nylon and polyester fibers can also be made by IR spectroscopy.

5.4 IR spectroscopy can provide molecular information regarding major organic and inorganic components. Components in lesser amounts are typically more difficult to identify. 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.

5.5 Fiber samples are prepared and mounted for microscopical IR analysis by a variety of techniques. IR spectra of fibers are obtained using an IR spectrometer coupled with an IR microscope, ATR, or diamond compression cell with beam condenser.

5.6 IR spectroscopy can be used to obtain spectra for elucidation of the chemical composition of the fiber and for comparison of two or more fiber samples.

5.6.1 When used to characterize the fiber type, the spectrum can be compared to reference spectra obtained from authenticated samples or reference standards.

5.6.2 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 should meet or exceed the requirements of Practice E1492 and Guide E1459.

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