Designation: ~~E2224 – 23~~ E2224 – 23a

An American National Standard

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 *Units*—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,~~is intended for use by competent forensic science practitioners with the requisite formal education, discipline-specific training (see Practice E2917 ~~and abilities.~~), and demonstrated proficiency to perform forensic casework.*

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.*

[1] This guide is under the jurisdiction of ASTM Committee E30 on Forensic Sciences and is the direct responsibility of Subcommittee E30.01 on Criminalistics.

Current edition approved ~~April 1, 2023~~May 1, 2023. Published May 2023. Originally approved in 2002. Last previous edition approved in ~~2022~~2023 as ~~E2224 – 22.~~E2224 – 23. DOI: ~~10.1520/E2224-23.~~10.1520/E2224-23A.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D123 Terminology Relating to Textiles
E131 Terminology Relating to Molecular Spectroscopy
E620 Practice for Reporting Opinions of Scientific or Technical Experts
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
E2917 Practice for Forensic Science Practitioner Training, Continuing Education, and Professional Development Programs

2.2 *Other Documents:*

ISO 17025 Testing and calibration laboratories[3]

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 (for example, vegetable, rayon/Lyocell).

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**).[4]

3.2.7 *exclusionary difference*—a difference in one or more characteristics between compared items that is sufficient to determine that the compared items did not originate from the same source, are not the same source, or do not share the same composition or classification.

3.2.7.1 *Discussion*—

What is sufficient depends on the performance and limitations of the method used on the material in question. **[OSAC (2Preferred)Definition]**

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

3.2.8.1 *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 (**23**).

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

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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] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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

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.

3.2.10.1 *Discussion*—

Acrylic, nylon, polyester, olefin, urethane, and polyvinyl are examples of fiber synthesized from chemical compounds. Cellulose-based fibers, such as acetate and rayons, and alginate fibers are examples of modified or transformed polymers. **D123**

3.2.11 *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}).

3.2.12 *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).

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

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 can 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 proceed to IR spectroscopy as the next step in the analytical scheme, as applicable.

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**5). 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.

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.

6.2.1 Useful sample preparation accessories include, but are not limited to, forceps, sample supports, IR windows, presses, dies, rollers, scalpels, and tungsten probes.

6.3 The quantity of fiber used and the number of fiber samples required will differ according to the following:

6.3.1 Specific technique and sample preparation,

6.3.2 Sample composition (for example, fabrics comprised of multiple fiber types),

6.3.3 Condition of the sample, and

6.3.4 Other case-dependent analytical conditions, concerns, or both.

6.4 When necessary to ensure reproducibility and evaluate intra-sample variations, repeat analysis of samples is recommended, if possible. The number of replicates is dependent on factors such as sample size, composition, and condition, and is evaluated on a case-by-case basis.

6.5 IR analysis can be performed using either transmittance or reflectance. These measurements can be taken with a variety of equipment configurations and accessories, the most common being the use of a diamond compression cell, ATR or an IR microscope.

6.5.1 ATR, also known as internal reflection spectroscopy (IRS), is a rapid sampling method that enables the examiner to collect IR spectra from manufactured textile fibers with minimal sample preparation.

6.6 Fiber samples being compared shall be prepared and analyzed in the same manner. Generally, fibers should be flattened prior to analysis. The sample needs to be thin enough not to over-absorb and to provide sufficient surface area for analysis in order to obtain the best quality spectra. Flattening the fiber alters the morphology, and therefore, the minimum length of fiber necessary for the analysis should be used. Flattening the fibers can alter the crystalline/amorphous structure of the fiber and result in minor differences in peak frequencies and intensities. This should be taken into consideration when making spectral comparisons (**56**). Leaving the fiber un-flattened, while allowing crystallinity-sensitive bands to be observed unaltered, results in distortion of peak heights due to variable pathlengths (**67**). In certain situations, a combination of both approaches is advisable.

6.6.1 Fibers analyzed by means of ATR generally do not require sample prep (for example, flattening) prior to coming into contact with the ATR crystal. However, because ATR is a surface technique, contaminants on the surface of the fiber can make a more significant contribution to the spectrum as compared to using a transmission method.

6.7 The flattened fiber can be mounted across an aperture, on an IR window, or between IR windows. The choice of window material should not reduce the effective spectral range of the detector being used. Common IR window materials used for this purpose include, but are not limited to, potassium bromide (KBr), cesium iodide (CsI), barium fluoride (BaF_2), zinc selenide (ZnSe), and diamond. When the fiber is mounted between two IR windows, a small KBr crystal should be placed next to the fiber. The background spectrum should be acquired through this crystal to avoid interference fringes that would arise if the spectrum were acquired through the air gap between the two IR windows.

6.8 When several fibers are mounted on or in a single mount, they should be physically separated so that their positions can be unambiguously documented for later retrieval, reanalysis, or both, and to prevent spectral contamination from stray light that might pass through another fiber.

7. Analysis

7.1 *Equipment:*

7.1.1 A mid-infrared spectrometer (FTIR is the current standard, but dispersive IR is not excluded) and an IR microscope that is compatible with the spectrometer or diamond compression cell with beam condenser are recommended (~~7~~8). The lower frequency cutoff varies with the microscope detector used, and should be no higher than 750 cm^{-1}.

7.2 *Instrument Parameters:*

7.2.1 All spectrometer and microscope components should be turned on and allowed to reach thermal stability prior to commencement of performance verification and operational runs. It should be noted that some FTIR instruments are designed to work best when left on or in the standby mode 24 hours a day. Refer to the manufacturer's guidelines for the optimum performance of their instruments.

7.2.2 It is essential that instrument performance be evaluated routinely (for example, once a month or before use, if used less frequently), in a comprehensive manner, and according to the laboratory's operation manual or the manufacturer's guidelines.

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

7.2.4 Sample and background spectra shall be run under the same instrument conditions. A background spectrum refers to a reference absorption spectrum, which includes the absorbance contributions of all system components except the sample of interest. Instrument parameters include resolution, number of scans, and the size of the apertures. The order in which the spectra are obtained is determined by instrument capabilities, policy, or analyst discretion.

7.2.5 Depending on instrument capabilities, 16 to 256 scans are typically collected at a resolution of 4 cm^{-1} or less.

7.2.6 *Microscope Parameters:*

7.2.6.1 The apertures that control the areas (fields) of sample illumination and detector measurement in an IR microscope can be of fixed or variable size, and can be either rectangular or circular in shape. Variable rectangular apertures are recommended because they can be more closely matched to the fiber shape. Light throughput, stray light reduction, and aperture focus in the sample image plane are some of the considerations in selecting aperture parameters and positioning. Fiber width, length, flatness, and linearity will usually limit the size of the illumination and detector apertures used for analysis. In general, the aperture boundaries should lie within the edges of the fiber.

7.2.6.2 The objective, condenser, or both, should be optimized, if possible, for any IR window in the beam path. This compensation reduces spherical aberration and permits more accurate focus.

7.2.6.3 To reduce potential polarization effects, the fibers being compared should be oriented in the same direction.

7.2.6.4 Focus as close to the center of the sample volume as possible and center on the optical axis of the system. Focus and re-center the condenser as necessary. This is best accomplished using a circular field aperture.

7.2.6.5 Adjust the detector measurement aperture width to just slightly less than the width of the fiber, but preferably not less than 10 µm (89). The aperture length can vary with sample geometry, but not be so great as to allow the detector to be saturated when acquiring a background spectrum. Adjust the illuminating field aperture so that the image of its edges coincides with those of the detector measurement aperture. The size and position of the apertures should not vary between sample and background data acquisition for a given analysis.

7.3 The quality of a spectrum is dependent on the focus, size of the apertures, thickness of the fiber, nature of the fiber, presence