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~~Designation:E2224–02~~ Designation: E2224 – 10

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) spectrophotometery is a valuable method of fiber polymer identification and comparison in forensic examinations. The use of IR microscopes coupled with Fourier transform infrared (FT-IR) spectrometers has greatly simplified the IR analysis of single fibers, thus making the technique feasible for routine use in the forensic laboratory.

1.2 This guideline is intended to assist individuals and laboratories that conduct forensic fiber examinations and comparisons in the effective application of infrared spectroscopy to the analysis of fiber evidence. Although this guide is intended to be applied to the analysis of single fibers, many of its suggestions are applicable to the infrared analysis of small particles in general.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D123 Terminology Relating to Textiles

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

3. Terminology

~~3.1~~

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

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *absorbance (A)*—the logarithm to the base 10 of the reciprocal of the transmittance, (T)~~; $A = \log_{10}(1/T) = -\log_{10}T$.~~ ~~3.2~~):

$$A = \log_{10}(1/T) = -\log_{10}T$$

3.2.2 *absorption band*—a region of the absorption spectrum in which the absorbance passes through a maximum.

~~3.3~~

3.2.3 *absorption spectrum*—a plot, or other representation, of absorbance, or any function of absorbance, against wavelength, or any function of wavelength.

~~3.4~~

3.2.4 *absorptivity (a)*—absorbance divided by the product of the sample pathlength (b) and the concentration of the absorbing substance (c)~~; $a = A/bc$~~ ~~3.5~~):

$$a = A/bc$$

3.2.5 *attenuated total reflection (ATR)*—reflection that occurs when an absorbing coupling mechanism acts in the process of total internal reflection to make the reflectance less than unity.

~~3.6~~

3.2.6 *background*—apparent absorption caused by anything other than the substance for which the analysis is being made.

~~3.7~~

3.2.7 *cellulosic fiber*—fiber composed of polymers formed from glucose subunits.

~~3.8~~

[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 July 10, 2002. Published September 2002. DOI: 10.1520/E2224-02.~~

Current edition approved Sept. 15, 2010. Published October 2010. Originally approved in 2002. Last previous edition approved in 2002 as E2224 – 02. DOI: 10.1520/E2224-10.

[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.

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3.2.8 *far-infrared*—pertaining to the infrared region of the electromagnetic spectrum with wavelength range from approximately 25 to 300 µm (wavenumber range 400 to 30 cm^{-1}).

~~3.9~~

3.2.9 *Fourier transform*—a mathematical operation that converts a function of one independent variable to one of a different independent variable.

3.2.9.1 *Discussion*—In FT-IR spectroscopy, the Fourier transform converts a time function (the interferogram) to a frequency function (the infrared absorption spectrum). Spectral data are collected through the use of an interferometer, which replaces the monochrometer found in the dispersive infrared spectrometer.

3.2.10 *Fourier transform infrared (FT-IR) spectrometry*—a form of infrared spectrometry in which an interferogram is obtained; this interferogram is then subjected to a Fourier transformation to obtain an amplitude-wavenumber (or wavelength) spectrum.

~~3.11~~

3.2.11 *generic class*—a group of fibers having similar (but not necessarily identical) chemical ~~composition. A~~composition; a generic name applies to all members of a group and is not protected by trademark registration.

3.2.11.1 *Discussion*—Generic names for manufactured fibers include, for example, rayon, nylon, and polyester. ~~(Generic~~Generic names to be used in the United States for manufactured fibers were established as part of the Textile Fiber Products Identification Act enacted by Congress in 1954 **(1)**.[3]

~~3.12~~

3.2.12 *infrared*—pertaining to the region of the electromagnetic spectrum with wavelength range from approximately 0.78 to 1000 µm (wavenumber range 12 800 to 10 cm^{-1}).

~~3.13~~

3.2.13 *infrared spectroscopy*—pertaining to spectroscopy in the infrared region of the electromagnetic spectrum.

3.2.14 *internal reflection spectroscopy (IRS)*—the technique of recording optical spectra by placing a sample material in contact with a transparent medium of greater refractive index and measuring the reflectance (single or multiple) from the interface, generally at angles of incidence greater than the critical angle.

~~3.15~~

3.2.15 *manufactured (man-made) fiber*~~—any fiber derived by a process of manufacture from any substance that, at any point in the manufacturing process, is not a fiber.~~

~~3.16—*a class name for various genera of filament, tow, or staple produced from fiber forming substance which may be (1)*~~ polymers synthesized from chemical compound, (*2*) modified or transformed natural polymers, or (*3*) glass.

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

~~3.17~~

3.2.17 *near-infrared*—pertaining to the infrared region of the electromagnetic spectrum with wavelength range from approximately 0.78 to 2.5 µm (wavenumber range 12 820 to 4000 cm^{-1}).

~~3.18~~

3.2.18 *spectrometer*—photometric device for the measurement of spectral transmittance, spectral reflectance, or relative spectral emittance.

~~3.19~~

3.2.19 *subgeneric class*—a group of fibers within a generic class that share the same polymer composition~~. S~~; subgeneric names include, for example, nylon 6, nylon 6,6, and poly(ethylene terephthalate).

3.2.20 *transmittance (T)*—the ratio of radiant power transmitted by the sample, *I*, to the radiant power incident on the sample, I_o; ~~*T = I/I* $_o$~~

~~3.21:~~

$$T = I/I_o$$

3.2.21 *wavelength*—the distance, measured along the line of propagation, between two points that are in phase on adjacent waves.

~~3.22~~

3.2.22 *wavenumber*—the number of waves per unit length, in a vacuum, usually given in reciprocal centimeters, cm^{-1}.

4. Summary of Guide

~~4.1This guideline covers identification of fiber polymer composition by interpretation of absorption spectra obtained by infrared microspectroscopy. It is intended to be applicable to a wide range of infrared spectrophotometery and microscope configurations.~~

4.1 This guideline covers identification of fiber polymer composition by interpretation of absorption spectra obtained by infrared microspectroscopy. It is intended to be applicable to a wide range of infrared spectrophotometery and microscope configurations. Additional information on infrared and microscopical analyses can be found in the sources listed in the Bibliography at the end of this guide.

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

4.2 Spectra may also be obtained by a variety of alternative IR techniques. Other techniques (not covered in the scope of this guideline) include: micro internal reflection spectroscopy (MIR), which differs from attenuated total reflectance (ATR) in that the infrared radiation is dependent upon the amount of sample in contact with the surface of the prism **(2)**:

4.2.1 Diamond cell (medium or high pressure) used with a beam condenser **(3-5)** (This combination is frequently used with a spectrophotometer configured for mid- and far-IR).

4.2.2 Thin films: solvent **(6, 7)**, melt **(4)**, or mechanically prepared **(8)**.

4.2.3 Lead foil technique **(6)**.

~~4.2.4Micro-KBr (or other appropriate salt) pellets~~

4.2.4 Micro potassium bromide (micro-KBr) (or other appropriate salt) pellets **(9, 10)**. This list is not meant to be totally inclusive or exclusive.

4.3 This analytical method covers manufactured textile fibers (with the exception of inorganic fibers), including, but not limited to:

Acetate	Modacrylic	Polyester	Vinal **(5)**
Acrylic	Novoloid **(5)**	Rayon	Vinyon
~~Anidex~~	~~Nylon~~	~~Saran~~	
Anidel	Nylon	Saran	
Aramid	Nytril	Spandex	
Azlon **(5)**	Olefin	Sulfar	
Fluorocarbon	Polybenzimidazole (PBI)	Triacetate	
Lastrile	Polycarbonate	Rubber	

Although natural fibers may also be analyzed by IR spectroscopy, they are excluded from this guideline because no additional discriminating compositional information of the fiber is provided over that yielded by light microscopy. However, infrared spectrophotometery may provide significantly useful information if there are dyes present in the natural fiber and can serve to distinguish among similarly colored fibers.

5. Significance and Use

5.1 Fiber samples may be prepared and mounted for microscopical infrared analysis by a variety of techniques. Infrared spectra of fibers are obtained using an IR spectrophotometer coupled with an IR microscope. Fiber polymer identification is made by comparison of the fiber spectrum with reference spectra.

5.2 Consideration should be given to the potential for additional compositional information that may be obtained by IR spectroscopy over polarized light microscopy alone (see Microscopy Guidelines). The extent to which IR spectral comparison is indicated will vary with specific sample and case evaluations.

5.3 The recommended point for IR analysis in a forensic fiber examination is following visible and ultraviolet (UV) comparison microscopy (fluoresence microscopy), polarized light microscopy, and UV/visible spectroscopy, but before dye extraction for thin-layer chromatography. This list of analytical techniques is not meant to be totally inclusive or exclusive.

5.4 The following generic types of fiber are occasionally encountered in routine forensic examinations: ~~Anidex,~~Anidel, Fluorocarbon, Lastrile, Novoloid, Nytril, Polycarbonate, PBI, Sulfar, Vinal, and Vinyon.

5.~~5~~4.1 Exemplar data, reference standards, ~~and/or~~or examiner experience, or combination thereof, may be inadequate for characterization of these fibers by optical microscopical and microchemical techniques. For these fiber types, IR spectroscopic confirmation of polymer type is advisable.

5.~~6~~5 Because of the large number of subgeneric classes, forensic examination of acrylic fibers is likely to benefit significantly from IR spectral analysis **(11)**.

5.~~7~~6 Colorless manufactured fibers are lacking in the characteristics for color comparison available in dyed or pigmented fibers. The forensic examination of these fibers may, therefore, benefit from the additional comparative aspect of IR spectral analysis.

~~5.8If~~5.7 If polymer identification is not readily apparent from optical data alone, an additional method of analysis should be used such as microchemical tests, melting point, pyrolysis infrared spectrophotometry, or pyrolysis gas chromatography. Infrared analysis offers the advantage of being the least destructive of these methods **(12)**.

6. Sample Handling

6.1 The general handling and tracking of samples should meet or exceed the requirements of Practice E1492 and Guide E1459.

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

6.2.1 Specific technique and sample preparation,

6.2.2 Sample homogeneity,

6.2.3 Condition of the sample, and

~~6.2.4Other case dependent analytical conditions and/or concerns.~~

6.2.4 Other case dependent analytical conditions or concerns, or both.

6.3 Sample preparation should be similar for all fibers being compared. Fibers should be flattened prior to analysis in order to obtain the best quality absorption spectra. Flattening the fibers can alter the crystalline/amorphous structure of the fiber and result in minor differences in peak frequencies and intensities. This must be taken into consideration when making spectral comparisons **(~~12~~13)**. Leaving the fiber unflattened, while allowing crystallinity-sensitive bands to be observed unaltered, results in distortion of

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