



Standard Guide for Forensic Analysis of Fibers by Infrared Spectroscopy¹

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1. Scope

1.1 Infrared (IR) spectrophotometry 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 **Definitions**—For definitions of terms used in this guide, refer to Terminology **D123**.

3.2 Definitions of Terms Specific to This Standard:

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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.

3.2.1 **absorbance (A)**—the logarithm to the base 10 of the reciprocal of the transmittance, (T):

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

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

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

3.2.4 **absorptivity (a)**—absorbance divided by the product of the sample pathlength (b) and the concentration of the absorbing substance (c):

$$a = A/bc$$

3.2.5 **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.

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

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

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}).

3.2.9 **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 monochromator 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.

3.2.11 **generic class**—a group of fibers having similar (but not necessarily identical) chemical 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 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

3.2.12 *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}).

3.2.13 *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.

3.2.15 *manufactured (man-made) fiber*—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}).

3.2.17 *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}).

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

3.2.19 *subgeneric class*—a group of fibers within a generic class that share the same polymer composition; 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 :

$$T = I/I_o$$

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

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

4.1 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 spectrophotometry 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.

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).

4.2.4 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
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 spectrophotometry 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: Anidel, Fluorocarbon, Lastrile, Novoloid, Nytril, Polycarbonate, PBI, Sulfar, Vinal, and Vinyon.

5.4.1 Exemplar data, reference standards, or examiner experience, or combination thereof, may be inadequate for characterization of these fibers by optical microscopical and

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

microchemical techniques. For these fiber types, IR spectroscopic confirmation of polymer type is advisable.

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

5.6 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.

5.7 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
- 6.2.4 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 (13). Leaving the fiber unflattened, while allowing crystallinity-sensitive bands to be observed unaltered, results in distortion of peak heights due to variable pathlengths (14). In certain situations, a combination of both approaches is advisable.

6.4 Because flattening the fiber is destructive of morphology, the minimum length of fiber necessary for the analysis should be used. A typical IR microscope is optimized for a 100 μm -spot size, thus little beam energy passes through a point that is farther than 50 μm from the center of the field of view. Hence, analytical performance will not necessarily be improved with the use of fibers greater than 100 μm in length.

6.5 The flattened fiber may be mounted across an aperture, on an IR window, or between IR windows. Common IR window materials used for this purpose include, but are not limited to, KBr, caesium iodide (CsI), barium fluoride (BaF_2), zinc selenide (ZnSe), and diamond. The choice of window material should not reduce the effective spectral range of the detector being used. When the fiber is mounted between two IR windows, care must be taken to avoid light by-pass around the fiber; otherwise an interference pattern will be introduced in the spectrum of the sample. Where 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, which would

arise if the spectrum of an air “gap” between the two IR windows was acquired or if the fringes would distort the ratioed spectrum.

6.6 Where several fibers are mounted on or in a single mount, they should be well separated (microscopically) so that their positions can be unambiguously documented for later retrieval or reanalysis, or both, and to prevent spectral contamination from stray light that might pass through another fiber. It is important that the longitudinal plane (flattened surface) of the fiber be as nearly parallel to the IR window or other mount as possible.

7. Analysis

7.1 A mid-infrared spectrophotometer (FT-IR is the current standard, but dispersive IR is not excluded) and an infrared microscope that is compatible with the mid-range spectrophotometer is recommended (15). The lower frequency cutoff will vary with the microscope detector used (preferably no higher than 750 cm^{-1}).

7.1.1 Useful sample preparation accessories include, but are not limited to, sample supports, infrared windows, presses, dies, rollers, scalpels, and etched-tungsten probes.

7.2 All spectrophotometer and microscope components should be turned on and allowed to reach thermal stability prior to commencement of calibration and operational runs. This may take up to several hours. It should be noted that most FT-IR instruments are designed to work best when left on or in the standby mode 24 hours a day.

7.3 It is essential that instrument performance and calibration be evaluated routinely, at least once a month, in a comprehensive manner.

7.4 The preferred performance evaluation method is in accordance with Practice E1421, Sections 1–7, 9.5, and 9.5.1. In brief, this includes:

- 7.4.1 System throughput,
- 7.4.2 Single-beam spectrum,
- 7.4.3 100 % T line, and
- 7.4.4 Polystyrene reference spectrum.

7.5 The apertures that control the areas (fields) of sample illumination and detector measurement in an IR microscope may be of fixed or variable size, and may 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, flatness, and linearity will usually limit the size of the illumination and detector apertures used for analysis. In general, the illuminating and detector fields should lie within the boundaries of the fiber edges.

7.6 Not all systems provide for the control of both illumination and detector measurement fields; the following recommendations can be modified to suit the constraints of a particular system design.

7.7 The objective or condenser, or both, should be adjusted (if possible) for any IR window that lies between the optic and the sample in the beam path. This compensation reduces spherical aberration and permits more accurate focus.

7.8 Infrared spectrophotometers and microscopes exhibit a polarization bias. This fact, coupled with the pleochroism associated with most fibers, makes it essential that fiber alignment be consistent throughout an analysis and preferably for all fiber analyses performed on a given system. A vertical or “north-south” alignment is typically used.

7.9 Samples should be focused as close to the center of the sample volume as possible and centered on the optical axis of the system. The condenser should be focused and recentered if necessary (this is best accomplished using a circular field aperture).

7.10 The detector measurement aperture width should be adjusted to just slightly less than the width of the fiber, but preferably not less than 10 μm (16). The aperture length may vary with sample geometry, but should not be so great as to allow the detector to be saturated when acquiring a background spectrum. The illuminating field aperture should be adjusted so that the image of its edges coincide 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. Sample and background scans must be run under the same conditions. If necessary, parameters can be subsequently modified and new sample and background spectra acquired.

7.11 A background spectrum refers to a reference energy spectrum other than the sample of interest, that includes the source energy, detector response and all energy losses of the optics. The IR window or windows with KBr crystal are all considered part of the system. The system parameters for the background spectrum must be identical to the parameters used for the sample spectra. These parameters include but not limited to resolution, mirror velocity, aperture, number of scans, and spectral region. If necessary, parameters may be subsequently modified and a new background spectrum obtained for the new sample conditions.

7.12 Resolution should be set at 4 cm^{-1} (one data point every 2 cm^{-1}). Higher resolution may be used; however, the additional data points typically yield no further analytical information for polymer samples. Because the apertures are

adjusted to fit the sample, it is usually most convenient to acquire the sample spectrum prior to acquiring the background spectrum.

7.13 It is generally useful to save all data on computer-based media just after it is generated and prior to any modification. Consideration should be given to storing the original interferogram data prior to Fourier transformation. Data that is processed during subsequent presentations can then be restored from saved files of the original background and sample interferogram.

7.14 Successful identification of fiber polymers by their IR spectra depends on experience and familiarity with fiber reference spectra. Spectral identification is accomplished by comparison with spectra of known reference standards. A library of reference IR spectra is essential. A library of reference fiber IR spectra obtained using the same technique used for the unknown fiber is desirable. It is also desirable to have available authentic samples of the fibers to be identified.

7.15 Dispersive instruments should be checked according to manufacturers’ recommendations. Instrument performance records may be maintained on hard copy or computer disk, or both. Report documentation may vary by laboratory, but should include the date, the operator, the system parameters, and the original instrumental output data.

8. Report Documentation

8.1 For identification, the positions of the absorption bands according to wavelength or wavenumber and their relative intensities must be compared with those of a known reference spectrum. It is desirable to examine the fibers by other methods, such as polarized light microscopy.

8.2 The generic class of manufactured textile fibers can be unequivocally identified. The subgeneric class of synthetic manufactured fibers may be identified.

8.3 Similarity or dissimilarity in the IR spectra can be noted when making a fiber comparison.

8.4 A physical copy of the spectra obtained should be placed in the case file.

9. Keywords

9.1 fibers; forensic science; infrared spectrophotometry

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