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

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1. Scope

1.1 Infrared (IR) ~~spectrophotometry~~spectroscopy 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~~(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 ~~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.~~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

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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. Terminology

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

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*):~~

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

3.2.1 ~~*absorption*~~*aperture, ~~band~~—n*—~~a region of the absorption spectrum in which the absorbance passes through a maximum.~~an opening in an optical system that controls the amount of light passing through a system.

~~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*):~~

~~$$a = A/bc$$~~

3.2.2 *attenuated total reflection ~~(ATR)~~—(ATR), n*—~~reflection that occurs when an absorbing coupling mechanism acts in the process of total internal reflection to make the reflectance less than unity.~~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*~~—*background, n*—apparent absorption caused by anything other than the substance for which the analysis is being made. **E131**

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

3.2.5 ~~*far-infrared*~~—*delustrant, n*—~~pertaining to the infrared region of the electromagnetic spectrum with wavelength range from approximately 25 to 300 µm (wavenumber range 400 to 30 cm~~a pigment, usually titanium dioxide, used to dull the luster of a manufactured fiber.~~$^{-1}$).~~ **E2228**

3.2.6 ~~*Fourier*~~*diffraction, ~~transform~~—n*—~~a mathematical operation that converts a function of one independent variable to one of a different independent variable.~~ 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]

~~3.2.9.1 *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.~~

3.2.7 *generic ~~class~~—class, n*—~~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.~~ 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 ~~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~~**2**).

3.2.8 ~~*infrared*~~—*interference fringes, n*—~~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~~the pattern that results from constructive and destructive interference of light waves.~~$^{-1}$).~~

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

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

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

3.2.10 *manufactured ~~(man-made)~~fiber, ~~fiber—~~n—*a class name for various genera of filament, tow, or staple produced from ~~fiber forming substance which may~~fiber-forming substances which can be (*1*) polymers synthesized from chemical ~~compound,~~ 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—*~~*mid-infrared, n—*pertaining to the ~~infrared~~IR region of the electromagnetic spectrum with wavelength range from approximately 2.5 to 25 µm (wavenumber range approximately 4000 to 400 cm^{-1}).

~~3.2.17 *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.~~

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

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

~~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.~~

4.2 ~~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~~	

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, ~~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.~~light microscopy is the primary method for the identification of natural 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.~~

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

5.2 ~~The recommended point for IR analysis in a forensic fiber examination is following visible and ultraviolet (UV) comparison microscopy (fluorescence microscopy),~~ IR analysis should follow visible and fluorescence comparison 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.~~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.

~~5.4 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 microchemical techniques. For these fiber types, IR spectroscopic confirmation of polymer type is advisable.~~

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 ~~subgeneric~~sub-generic classes, forensic examination of acrylic and modacrylic fibers is likely to benefit significantly from IR spectral analysis (~~11~~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 ~~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.~~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 ~~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~~IR spectroscopy can be used to obtain spectra for elucidation of the chemical composition of the fiber ~~(~~and~~12).~~ 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:~~to the following:

6.3.1 Specific technique and sample preparation,

6.3.2 Sample ~~homogeneity,~~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 or~~ 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 ~~Sample preparation should be similar for all fibers being compared. Fibers~~ 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 ~~absorption~~ 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 ~~must~~should be taken into consideration when making spectral comparisons (~~13~~5). Leaving the fiber ~~unflattened,~~ un-flattened, while allowing crystallinity-sensitive bands to be observed unaltered, results in distortion of peak heights due to variable pathlengths (~~14~~6). 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.

~~6.4 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.~~

6.7 The flattened fiber ~~may~~can 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~~Common IR window materials used for this purpose include, but are not limited to, potassium bromide (KBr), cesium iodide (CsI), barium fluoride (BaF_2 ~~pattern will be introduced in the spectrum of the sample. Where the fiber is mounted between two IR windows,~~), 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, which~~fringes that would arise if the spectrum ~~of an air "gap" were~~ acquired through the air gap between the two IR ~~windows was acquired or if the fringes would distort the ratioed spectrum.~~windows.

6.8 ~~Where~~When several fibers are mounted on or in a single mount, they should be ~~well~~physically separated ~~(microscopically)~~ so that their positions can be unambiguously documented for later ~~retrieval or~~ retrieval, 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}).~~*Equipment:*

7.1.1 ~~Useful sample preparation accessories include, but are not~~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 ~~**limited**~~(7). ~~to, sample supports, infrared windows, presses, dies, rollers, scalpels, and etched-tungsten probes.~~The lower frequency cutoff varies with the microscope detector used, and should be no higher than 750 cm^{-1}.

~~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.~~

~~7.2 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.~~*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 may be dictated 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 (**8**). The aperture length may 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.

~~7.6 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.~~

~~7.3 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.~~