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Forensic Fiber Examination Guidelines

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Scientific Working Group on Materials Analysis
(SWGMAT)
(formerly the Technical Working Group on
Materials Analysis [TWGMAT])
Fiber Subgroup

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Acknowledgments

Victoria Forensic Science Centre, Australia
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Chapter 1 of Forensic Fiber Examination Guidelines

Introduction to Forensic Fiber Examination

1.0. Scope

This document is an outline of fiber analysis methods intended for use by forensic fiber examiners. The particular methods employed by each examiner, laboratory, or both will depend upon sample size, sample suitability, laboratory equipment, and examiner training.

2.0. Reference Documents

SWGMAT Quality Assurance Guidelines
SWGMAT Trace Evidence Handling Guidelines

3.0. Terminology

Known (Sample): A subset of a larger population or sample originating from a verifiable source, collected as representative of that larger grouping; for example, a 2" x 2" section of carpet from a suspect's living room.

Questioned (Sample): Materials collected as or from items of evidence that have a known location but an unknown origin; for example, loose fibers collected from a victim's clothing.

Class Characteristics: Traits that define a group of items collectively.

Class: A group of items that share properties or characteristics.

Individual Characteristics: Traits that define and identify an item as unique and exclusive of all other items.

Individual: A unique item that is identified as itself to the exclusion of all other items.

4.0. Summary of Fiber Analysis Guidelines

The various analytical methods available for fiber analysis yield different kinds of information. It is highly desirable to select a combination of methods and apply them in an order that provides the most exclusionary information first. By doing this, the examiner optimizes accuracy, precision, and production while most effectively using the laboratory's resources.

5.0. Significance and Use

5.1. Microscopy
At a minimum, a fiber examiner must employ a stereomicroscope, a comparison microscope, and a compound light microscope equipped with polarized light capability. The examiner must view questioned and known fibers side by side at the same magnifications in visible light, and alternative lighting, such as polarized light or fluorescent lighting, although not necessary, is recommended if the equipment allows. For some analyses, for example, in testing for solubility, it may be necessary to place questioned and known fibers next to each other on the same slide or in adjacent wells of a spot plate. Extreme caution must be exercised with loose fibers in these circumstances so as not to confuse the source of each fiber.

5.2. Comparison
Typically, fiber examinations involve a comparison of samples from known and questioned sources to determine whether they are consistent with having originated from the same source (e.g., carpet from a suspect's car compared with foreign fibers removed from the victim's clothing). This comparison involves the recognition and evaluation of class characteristics, which associate materials to a group but never to a single source. Conversely, individual characteristics allow the association between two or more items with each other to the exclusion of all other items. For fiber examiners, this most often occurs when pieces of fabric or cordage are physically matched.

5.3. Source Determination
Textile fibers can be exchanged between individuals, between individuals and objects, and between objects. When fibers are associated with a specific source, such as fabric from the victim, suspect, or scene, a value is placed on that association. The probative weight of this value is dependent upon many factors. The following subsections describe those factors.

5.3.1. Fiber type or types found;
5.3.2. Fiber color or colors;
5.3.3. Number of fibers found;
5.3.4. Fiber location or locations;

5.3.5. Fabric type or types;
5.3.6. Multiple fiber associations;
5.3.7. Nature of contact; and
5.3.8. Fiber transfer and persistence.

Whether a fiber is transferred and detected is also dependent on the nature and duration of the contact between the suspect, the victim, or both and the persistence of the fibers after they have been transferred.

5.3.1. Fiber Type or Types. The rarity or commonness of the fiber types found at a crime scene or on a victim or suspect affects their probative value. Cotton fibers are by far the most commonly used plant fibers in textile production. The type of cotton, the fibers' length, and the degree of twist contribute to the diversity found in cotton fibers. Processing techniques, such as mercerization, and color applications also influence the value of cotton fiber identifications. The presence of other less common plant fibers at a crime scene or on the clothing of a victim or suspect increases its significance.

The most common animal fiber used in textile production is wool originating from sheep. The fineness or coarseness of woolen fibers often dictates the end use of wool. The finer woolen fibers are used in the production of clothing, whereas the coarser fibers are found in carpet. The diameter and the degree of scale protrusion of the fibers are other important characteristics. Woolen fibers from other animals may also be found, including camel, alpaca, cashmere, and mohair. The identification of less common animal hairs, fibers, or both at a crime scene or on the clothing of a suspect or victim would have increased significance.

Over half of all fibers used in the production of textile materials are manufactured. Some manufactured fibers originate from natural materials such as cotton or wood, whereas others originate from synthetic materials. All nonnaturally occurring fibers are manufactured, but not all manufactured fibers are synthetic (e.g., rayon). Certain types of manufactured fibers are more common than others. Polyester and nylon fibers are the most commonly encountered manufactured fibers, followed by rayons, acetates, and acrylics. There are also many other less commonly manufactured fibers. The amount of production, the end use, the cross-sectional shape, microscopic characteristics, and other traits of the fiber help to influence the degree of rarity of a particular fiber type.

5.3.2. Fiber Color or Colors. One of the greatest variations seen in textiles is color. Thus, color greatly influences the significance of a fiber comparison. Synthetic dyes and pigments belong to 29 different chemical categories with more than a dozen different application methods (7). Even simple dyes might require between eight and ten processes to convert the raw materials into a finished dye. Given that the total annual production of any particular dye might not amount to more than 10 tons and that small process batches are becoming the rule in the dyeing industry, color becomes a powerful discriminant. Color is particularly significant when the gamut of colors is spread out over the range of garments and carpeting produced in any one year and even more so when multiplied by the number of garments and carpets produced in previous years.

Individual fibers can be colored before being spun into yarn, yarns can be dyed after being spun, or the fabric can be dyed before or after its construction. Color can also be applied to the surface of a fabric by printing. The absorbance of the dye along the fiber length suggests the dyes and dyeing method used. Fading and discoloration may also add increased significance to a fiber association.

5.3.3. *Number of Fibers.* The number of fibers identified on the clothing of a victim associated to the clothing of a suspect is important in determining actual contact. The greater the number of fibers, the more likely that direct contact occurred between these individuals. The converse is not necessarily true, however, and even one fiber association can have probative and scientific value. Additionally, finding no fibers does not de facto mean that no contact occurred. Each case is different, and the examiner must weigh all of the relevant factors before determining the significance of the evidence.

5.3.4. *Fiber Location.* Where the fibers are found also affects the probative value of a particular fiber association. The location of fibers on different areas of the body or on specific items at the scene can influence the significance of the fiber association.

5.3.5. *Fabric Type.* Fabric construction affects the number and types of fibers that may be transferred. Tightly woven or knitted fabrics shed fewer fibers than loosely knit or woven fabrics. Fabrics composed of filament yarns shed less than fabric composed of spun yarns. Certain types of fibers also transfer more readily. The condition and wear of the fabric also affects the degree of fiber transfers: Newer fabrics may have an abundance of loosely adhering fibers on the surface of the fabric, whereas worn fabrics may have damaged areas that easily shed fibers. Damage to a fabric caused during physical contact greatly increases the likelihood of fiber transfer.

5.3.6. *Multiple Fiber Associations.* If many different fiber types are associated among the suspect, victim, and scene, then the likelihood that contact occurred between these items is greatly increased. Each associated fiber transfer is considered to be an independent event, and multiple associations undermine an argument that the fibers were all deposited by coincidence.

5.3.7. *Nature of Contact.* The type of physical contact between a suspect and a victim helps to determine the number of fibers transferred and the value placed on their discovery. Violent physical contact of an extended duration may result in many fiber transfers.

5.3.8. *Fiber Transfer and Persistence.* Textile fibers are transferred to the surface of a fabric either by direct (primary) transfer or indirect (secondary) transfer. The likelihood of transfer depends on the types of fabric involved in the contact and the nature and duration of the contact. Studies have shown that transferred fibers are lost at a geometric rate, depending on the types of fabrics involved and on the movement of the clothing after contact (see endnote 1). For example, the clothing of a homicide victim may retain transferred fibers for a longer time because the victim is not moving. Therefore, under these circumstances it is difficult to predict precisely how many fibers might remain on the clothing of a living individual after a given period, but it is important for investigators to retrieve clothing immediately.

Whenever a fiber is found in relation to a crime scene, victim, or suspect, it has potential significance. Matching dyed fibers, whether manufactured or natural, can be very meaningful, whereas the matching of common fibers such as white cotton or blue denim cotton can would be less significant. In some situations, however, the presence of white cotton or blue denim cotton possibly still has some meaning in resolving the truth of an issue. The discovery of cross transfers (suspect[s] to victim[s] and vice versa) dramatically increases the likelihood that two items came into contact and greatly reduces the likelihood of chance occurrence.

When a fiber examiner associates a questioned fiber to a known textile item, there are ultimately two possible explanations: (a) The questioned fiber originated from the known textile, or (b) the questioned fiber did not originate from the known textile.

To say that the questioned fiber originated from the known textile, it either had to be the only fabric of its type ever produced or now existing, or the transfer of fibers was directly observed. As neither of these situations is likely to occur, fiber examiners must conclude that because the questioned fibers exhibit the same results in all tested properties as the fibers from the known sample, the questioned fibers are consistent with originating from the source textile. Other textile sources that incorporate the same fibers can be ruled out only by context and availability. In order to say that a fiber did not originate from a particular textile is to know the history of the textile or have observed the fiber transfer from another textile.

5.4. Volume of Fiber Production

It could be argued that the large volume of fibers produced reduces the significance of a fiber association discovered in a criminal case. It can never be stated with certainty that a fiber originated from a particular textile because other textiles are produced using the same fiber types and color. The inability to positively associate a fiber to a particular textile to the exclusion of all others, however, does not mean that a fiber association is without value. Considering the volume of textiles produced worldwide each year, the number of textiles produced with any one fiber type and color is extremely small. The likelihood of two or more manufacturers exactly duplicating all of the aspects of the textile is extremely remote (see endnote 2). Beyond the comments made previously about color, shade tolerance differs between dyeing companies. Therefore, color may vary demonstrably from batch to batch. Also, the life span of a particular fabric must be considered. Only so much of a given fabric of a particular color and fiber type is produced, and it will eventually end up being destroyed or dumped in a landfill.

The world produced approximately 80 billion pounds of fabric in 1995, about half of which was cotton (5). The other approximately 44 billion pounds of fiber were manufactured or synthetic. Table 1 provides U.S. fiber production levels.

Table 1: U.S. Annual Production for Manufactured Fibers: 1995 (millions of pounds)

Fiber	Product
Polyester	3,887
Nylon	270
Olefin	521

Rayon/Acetate/Triacetate	498
Acrylic/Modacrylic	432

(Table 1 [6]). All these fibers were used in a variety of applications including but not limited to clothing, household textiles, carpeting, and industrial textiles.

5.4.1. Significance. As an example, given a yarn-dyed nylon fiber from a knit polo shirt of a specific color, the significance could be described in the following way:

5.4.1.1. Total fiber production;

5.4.1.2. Total nylon (of that type) production;

5.4.1.3. Total nylon production in staple form;

5.4.1.4. Total production of Item 3 in a particular denier, cross-section, optical characteristics, and luster;

5.4.1.5. Total amount of Item 4 used in production of garments;

5.4.1.6. Total garments constructed in the same fashion, including knit specifications, collar, and sleeve incorporating Item 5;

5.4.1.7. Total of Item 6 in a specific color;

5.4.1.8. Total of Item 7 from indistinguishable dye lots;

5.4.1.9. Total of Item 8 available for merchandising;

5.4.1.10. Total of Item 9 sold;

5.4.1.11. Total of Item 10 still in existence;

5.4.1.12. Total of Item 11 available to be connected with a particular criminal offense; and

5.4.1.13. Total of Item 12 actually connected with a particular criminal offense (i.e., found and submitted as evidence).

The fiber examiner is still limited to stating that the questioned fibers are consistent with originating from the evidence garment, with the understanding that all other garments listed under Item 11 (subsection 5.4.1.11) may or may not be distinguishable from the evidence garment by fiber analysis alone. This argument in no way intimates a positive match to the evidence garment to the exclusion of all other garments. Production numbers for textiles may be available for use in interpreting the significance of evidence in a crime, but the examiner must be careful to be conservative in all estimates in order to avoid false inclusions (8). Calculating exact probability statistics for this type of evidence is problematic at best, and professional statisticians must be consulted before any calculations are reported or testified.

5.5. Fiber Source

If questioned fibers are associated with known fibers, the questioned fibers either originated from the known textile or from another fabric source, which not only is composed of fibers of the exact type and color but also from a fabric that had to be available to contribute those fibers through direct or indirect contact. The chance is, therefore, remote to encounter fibers from the environment of a victim that are identical to fibers from the suspects' environment or environments in the absence of contact (9). Put another way, the chance of finding known fibers

from a randomly selected suspect source that match the questioned fibers is remote (see endnote 3).

6.0. Sample Handling

Although examiners may be consulted concerning proper sample size, collection, or packaging, this may not happen, and the examiner must optimize the evidence that is submitted. Garfield (1) and others (2) list the following methods of sampling:

6.1. Probability Sampling (So-Called "Random Sampling")
Every unit in the population has a known, nonzero probability of being included in the sample (e.g., collecting about 33 percent of the fibers from a pillbox or by taping and mounting them);

6.2. Nonprobability or Judgment Sampling
Every unit in the population either is or is not included in the sample on the basis of certain characteristics it has in common with other units of interest (e.g., mounting only red trilobal carpet-type fibers from the victims' evidence given the suspect has red carpet as a possible source); and

6.3. Bulk or Lot Sampling
A sampling unit is taken from a larger amount of material that does not consist of discrete and identifiable units. Special considerations are involved with bulk sampling, such as where the sample is taken, how much sample is taken, and if the sample is considered representative of the lot (e.g., cutting a swatch from a garment for fabric and fiber examination).

Samples are adequate for analysis when they are taken in a manner consistent with generally recognized and accepted sampling techniques and practices within the context of the proposed analyses. All of the previously mentioned sampling methods have their place, and one may be more feasible than another, given crime scene or laboratory constraints. The examiner must be able to explain how the samples were taken and why that procedure was used.

Examinations typically should be conducted in the order of increasing magnification, from gross inspection to microscopical analysis. If sample size is limited, nondestructive methods must be exhausted before subjecting the sample to any destructive tests (e.g., pyrolysis).

It is highly desirable that the methods be selected in an order that provides the greatest discrimination between samples. An exclusion precludes further analysis, thereby maximizing the examiner's time and resources.

7.0. Analysis

There are three basic activities involved in an analysis (1): (a) collection of a representative sample; (b) preparation of the sample for analysis; and (c) analysis using appropriate methods.

Although these activities are ostensibly independent of each other, any one can have a significant effect on another. Because error is possible at each step, the examiner must be able to identify

these errors and avoid them. Any method of analysis has certain attributes such as accuracy, precision, specificity, sensitivity, dependability, and practicality that must be considered when choosing the most appropriate method to adequately answer the question at hand. Ultimately, it is the examiner's responsibility to evaluate all of the available information and decide the level of uncertainty that is acceptable with a given method on a given set of samples.

7.1. Physical Matches

A physical match occurs when two or more pieces of fabric or cordage are reconstructed to prove they were previously one continuous piece of fabric or cordage. This examination is conducted by describing and documenting any cut, torn, or damaged edges on questioned items and their correlation to like areas on known items. Photography is the recommended method of documentation.

Depending upon sample size, suitability, and exhibited characteristics, it may not be possible to effect a positive physical match. For descriptions of physical construction refer to the fabric and cordage guidelines in Chapter 7 of this document.

7.2. Fiber Examinations

Fiber identifications consist of determining the generic class of fiber type, which generally follows the Federal Trade Commission Guidelines (3). This analysis requires a sufficient number of examinations to unequivocally place the fiber in question into one and only one generic class (see Table 2).

Table 2: Federal Trade Commission Rules and Regulations Under the Textile Products Identification Act, 1954

Pursuant to the provisions of Section 7 of the Act, the following generic names for manufactured fibers, together with their respective definitions, are hereby established:	
Fiber Name	**Definition**
Acetate	A manufactured fiber in which the fiber-forming substance is cellulose acetate. Where not less than 92% of the hydroxyl groups are acetylated the term triacetate may be used as a generic description of the fiber.
Acrylic	A manufactured fiber in which the fiber-forming substance is any long-chain synthetic polymer composed of at least 85% by weight of acrylonitrile units.
Anidex	A manufactured fiber in which the fiber-forming substance is any long-chain synthetic polymer composed of at least 50% by weight of one or more esters of a monohydric alcohol and acrylic acid.
Aramid	A manufactured fiber in which the fiber-forming substance is any long-chain synthetic polyamide in which at least 85% of the amide linkages are attached directly to two aromatic rings.
Glass	A manufactured fiber in which the fiber-forming substance is glass.
Lyocel	A manufactured fiber composed of precipitated cellulose and produced by a solvent extrusion process where no chemical intermediates are formed.
Nylon	A manufactured fiber in which the fiber-forming substance is any long-chain synthetic polyamide in which less than 85% of the amide linkages are attached

	directly to two aromatic rings.
Metallic	A manufactured fiber composed of metal, plastic-coated metal, metal-coated plastic, or a core completely covered by metal.
Modacrylic	A manufactured fiber in which the fiber-forming substance is any long-chain synthetic polymer composed of less than 85% but at least 35% by weight of acrylonitrile units.
Novoloid	A manufactured fiber containing at least 85% by weight of a cross-linked novolac.
Olefin	A manufactured fiber in which the fiber-forming substance is any long-chain synthetic polymer composed of at least 85% by weight of ethylene, propylene, or other olefin units.
Polyester	A manufactured fiber in which the fiber-forming substance is any long-chain synthetic polymer composed of at least 85% by weight of an ester of a substituted aromatic carboxylic acid, including but not restricted to substituted terephthalate units and parasubstituted hydroxybenzoate units.
Rayon	A manufactured fiber composed of regenerated cellulose, as well as manufactured fibers composed of regenerated cellulose in which substituents have replaced not more than 15% of the hydrogens of the hydroxyl groups.
Saran	A manufactured fiber in which the fiber-forming substance is any long-chain synthetic polymer composed of at least 80% by weight of vinylidene chloride units.
Spandex	A manufactured fiber in which the fiber-forming substance is any long-chain synthetic polymer composed of at least 85% of a segmented polyurethane.
Vinal	A manufactured fiber in which the fiber-forming substance is any long-chain synthetic polymer composed of at least 50% by weight of vinyl alcohol units and in which the total of the vinyl alcohol units and any one or more of the various acetal units is at least 85% by weight of the fiber.
Vinyon	A manufactured fiber in which the fiber-forming substance is any long-chain synthetic polymer composed of at least 85% by weight of vinyl chloride units.

Fiber comparisons consist of determining if a questioned fiber or fibers exhibits the same chemical, microscopic, and optical properties as fiber or fibers comprising part or all of a known sample. A comparison requires an examiner to complete at least two of the analytical techniques listed for each of the following categories: generic class, physical characteristics, and color (see Figure 1). The techniques selected should independently confirm the results obtained. It should be noted that some techniques allow greater discrimination than others between apparently similar samples.

Figure 1: Analysis of Fibrous Materials

The analyst should perform a combination of methods that extract the greatest potential for discrimination between samples. The methods are presented in no particular order.

In order to perform an identification, the number of analytical tests performed is left to the discretion of the analyst. In order to perform a comparison, a minimum of two of the analytical techniques must be performed for each category (gray boxes).

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                         │  Fibrous Material   │
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              ┌───────────────────┬───────────────────────┐
      ┌───────────────┐   ┌───────────────────────┐
      │ Physical Match│   │ Physical Characteristics│
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                         ┌─────────────────────┐
                         │       Fibers        │
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 ┌──────────────┐  ┌─────────────────────┐  ┌──────────────┐
 │ Generic Class│  │Physical Characteristics│ │    Color     │
 └──────────────┘  └─────────────────────┘  └──────────────┘
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 │Polarized Light│ │   Stereomicroscopy  │  │Light Microscopy│
 │  Microscopy  │  └─────────────────────┘  └──────────────┘
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 │    FT-IR     │  │   Light Microscopy  │  │Microspectrophotometry│
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 │Pyrolosis-Gas │  │  Scanning Electron  │  │  Thin-Layer  │
 │Chromatography│  │     Microscopy      │  │Chromatography│
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 │  Microscopic │                           │ Fluorescence │
 │  Properties  │                           │  Microscopy  │
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 │  Solubility  │
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 │Thermal Analysis│
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 │Chemical/Physical Tests│
 │ (vegetable fibers)│
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 │Dispersion Staining│
 │ (mineral fibers)*│
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 │ X-Ray Diffraction│
 │ (mineral fibers)*│
 └──────────────────┘
 ┌──────────────────┐
 │Elemental Composition│
 │ (mineral fibers)*│
 └──────────────────┘
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* Not covered in these guidelines.

8.0. Report Documentation

Laboratory results should be reported in a uniform and consistent manner. Format, units of measurement, and accepted calculations should all be documented in the laboratory's manuals. The contributor of the evidence must be able to "interpret the results and understand their significance" (1). The International Organization for Standardization (ISO) recommends that reports be clear, accurate, and unambiguous in the presentation of results (4). Refer to the appropriate sections of the SWGMAT Quality Assurance Guidelines for further information.

9.0. References

(1) Garfield, F. M. *Quality Assurance Principles.* Association of Official Analytical Chemists, Arlington, Virginia, 1991.

(2) Levy, P. S. and Lemeshow, S. *Sampling of Populations.* John Wiley and Sons, New York, 1991.

(3) *Federal Trade Commission Rules and Regulations Under the Textile Products Identification Act,* Title 15, U.S. Code Section 70, et seq. 16 CFR 303.7.

(4) *International Standards Organization, Guide 25.* American National Standards Institute, New York, 1982.

(5) Layman, P. Growth in man-made fibers slowed in 1995, *Chemical and Engineering News* (May 27, 1996), p. 13.

(6) *Fiber Organon,* January 1996.

(7) Aspland, J. R. What are dyes? What is dyeing? In: *AATCC Dyeing Primer.* American Association of Textile Chemists and Colorists, Research Triangle Park, North Carolina, 1981.

(8) Deadman, H. A. Fiber evidence and the Wayne Williams Trial, *FBI Law Enforcement Bulletin* (March and May 1984).

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11.0. Endnotes

1. See, for examples, C. N. Lowrie and G. Jackson, "Secondary Transfer of Fibers," in *Forensic Science International* (1994) 64:73-82, and J. Roberston, C. B. M. Kidd, and H. M. P. Parkinson, "The Persistence of Textile Fibers Transferred During Simulated Contacts," in *Journal of the Forensic Science Society* (1982) 22:353-360.

2. See, for example, W. Bruschweiler and M. C. Grieve, "A Study on the Random Distribution of a Red Acrylic Target Fibre," in *Science and Justice* (1997) 37:85-90.

3. See D. A. Stoney article in *Journal of Forensic Sciences* (1984) 24:473-482.

Chapter 2 of Forensic Fiber Examination Guidelines

Microscopy of Textile Fibers

> ### *Read about …*
>
> **1.0. Scope**
> **2.0. Reference Documents**
> **3.0. Terminology**
> **4.0. Summary of Guidelines**
> **5.0. Significance and Use**
> **6.0. Sample Handling**
> **7.0. Analysis**
> **8.0. Report Documentation**
> **9.0. References**

1.0. Scope

This section describes guidelines for microscopical examinations employed in forensic fiber characterization, identification, and comparison. Several types of light microscopes are used including stereobinocular, polarized light, comparison, fluorescence, and interference. In certain instances, the scanning electron microscope yields additional information. The nature and extent of the fiber evidence will dictate which tests or techniques are selected and performed.

2.0. Reference Documents

SWGMAT Quality Assurance Guidelines
SWGMAT Trace Evidence Handling Guidelines

3.0. Terminology

Anisotropic: An object that has properties that differ according to the direction of measurement when viewed in polarized light.

Barrier Filter: A filter used in fluorescence microscopy that suppresses unnecessary excitation light that has not been absorbed by the fiber and selectively transmits only the fluorescence.

Becke Line: The bright halo near the boundary of a fiber that moves with respect to that boundary as the microscope is focused through best focus.

Becke Line Method: A method for determining the refractive index of a fiber relative to its mountant by noting the direction in which the Becke line moves when the focus is changed. The Becke line will always move toward the higher refractive index medium (fiber or mountant) when focus is raised and will move toward the lower refractive index medium when focus is lowered.

Birefringence: The numerical difference in refractive indices for a fiber, given by the formula: n_{ll}

- n. Birefringence can be calculated by determining the retardation (r) and thickness (T) at a particular point in a fiber and by using the formula: $B = r$ (nm)$/1,000$ T (μm).

Comparison Microscope: A system of two microscopes positioned side by side and connected via an optical bridge in which specimens are examined simultaneously in either transmitted or reflected light.

Compensator: Any variety of optical devices that can be placed in the light path of a polarizing microscope to introduce fixed or variable retardation comparable with that exhibited by the fiber. The retardation and sign of elongation of the fiber is then determined. Compensators can employ a fixed mineral plate of constant or varying thickness or a mineral plate that is rotated to alter the thickness presented to the optical path (and retardation introduced) by a set amount.

Compensator, Full Wave (Red Plate): A compensator using a plate of gypsum, selenite, or quartz, which introduces a fixed retardation between 530-550 nm (approximately the retardation of the first order red color on the Michel-Lévy chart).

Compensator, Quarter Wave: A compensator, usually with a mica plate, which introduces a fixed retardation between 125-150 nm.

Compensator, Quartz Wedge: A wedge, cut from quartz, having continuously variable retardation extending over several orders of interference colors (usually 3-7).

Compensator, Sénarmont: A quarter-wave plate inserted above the specimen in the parallel 0" position with a rotating calibrated analyzer. Measures low retardation and requires the use of monochromatic light.

Compensator, Tilting (Berek): A compensator typically containing a plate of calcite or quartz, which can be rotated by means of a calibrated drum to introduce variable retardation up to about ten orders.

Cortex: The main structural component of hair consisting of elongated and fusiform (spindle-shaped) cells. The cortex contains pigment grains, air spaces called cortical fusi, and structures called ovoid bodies.

Crimp: The waviness of a fiber.

Cross-Over Marks: Oblique flattened areas along silk fibers caused by the overlapping of extruded silk fibers before they have dried completely.

Cuticle: The layer of scales composing the outer surface of a hair shaft. Cuticular scales are normally classified into three basic types: coronal (crown-like), spinous (petal-like), and imbricate (flattened).

Delustrant: A pigment, usually titanium dioxide, used to dull the luster of a manufactured fiber.

Dichroism: The property of exhibiting different colors, especially two different colors, when viewed in polarized light along different axes.

Dislocations: Concerning natural fibers (e.g., flax, ramie, jute, and hemp) where distinct features in the shape of X's, I's, and V's are present along the fiber cell wall. These features are often useful for identification.

Dispersion of Birefringence: The variation of birefringence with wavelength of light. When dispersion of birefringence is significant in a particular fiber, anomalous interference colors not appearing in the regular color sequence of the Michel-Lévy chart may result. Strong dispersion of birefringence also interferes with the accurate determination of retardation in highly birefringent fibers.

Dispersion Staining: A technique for refractive index determination that employs central or annular stops placed in the objective back focal plane of a microscope. Using an annular stop with the substage iris closed, a fiber mounted in a high-dispersion medium will show a colored boundary of a wavelength where the fiber and the medium match in refractive index. Using a central stop, the fiber will show colors complimentary to those seen with an annular stop.

Dyes: Soluble substances that add color to textiles. Dyes are classified into groups that have similar chemical characteristics (e.g., aniline, acid, and azo). They are incorporated into the fiber by chemical reaction, absorption, or dispersion.

Excitation Filter: A filter used in fluorescence microscopy that transmits specific bands or wavelengths of energy capable of inducing visible fluorescence in various substrates.

Inorganic Fibers: A class of fibers of natural mineral origin (e.g., chrysotile asbestos) and manmade mineral origin (e.g., fiberglass).

Interference Colors: Colors produced by the interference of two out-of-phase rays of white light when a birefringent material is observed at a nonextinction position between crossed polars. The retardation at a particular point in a birefringent fiber is determined by comparing the observed interference color to the Michel-Lévy chart.

Isotropic: An object that is identical in all directions and invariant with respect to direction.

Light Microscope: A microscope that employs light in the visible or near-visible portion of the electromagnetic spectrum.

Lignin: The majority noncarbohydrate portion of wood. It is an amorphous polymeric substance that cements cellulosic fibers together. The principal constituent of woody cell walls.

Lumen: The cavity or central canal present in many natural fibers (e.g., cotton, flax, ramie, jute, and hemp). Its presence and structure are often a useful aid in identification.

Luster: The gloss or shine possessed by a fiber, resulting from its reflection of light. The luster of manufactured fibers is often modified by use of a delustering pigment.

Manufactured Fiber: A class name for various families of fibers produced from fiber-forming substances, which are synthesized polymers, modified or transformed natural polymers, and glass.

Medulla: The central portion of a hair composed of a series of discrete cells or an amorphous spongy mass. It may be air-filled and, if so, will appear opaque or black using transmitted light or white using reflected light. In animal hair, several types have been defined: uniserial or multiserial ladder, cellular or vacuolated, and lattice.

Michel-Lévy Chart: A chart relating thickness, birefringence, and retardation so that any one of these variables can be determined for an anisotropic fiber when the other two are known.

Microscopical: Concerning a microscope or the use of a microscope.

Modification Ratio: A geometrical parameter used in the characterization of noncircular fiber cross sections. The modification ratio is the ratio in size between the outside diameter of the fiber and the diameter of the core. It is also called aspect ratio.

Natural Fibers: A class name of fibers of vegetable origin (e.g., cotton, flax, and ramie), animal origin (e.g., silk, wool, and specialty furs), or of mineral origin (e.g., asbestos).

Pigment: A finely divided insoluble material used to deluster or color fibers (e.g., titanium dioxide and iron oxide).

Plane Polarized Light: Light that is vibrating in one plane.

Pleochroism: The property of exhibiting different colors, especially three different colors, when viewed in polarized light along different axes.

Polarized Light: A bundle of light rays with a single propagation direction and a single vibration direction. The vibration direction is always perpendicular to the propagation direction. It is produced by use of a polarizing filter, from ordinary light by reflection, or double refraction in a suitable pleochroic substance.

Polarized Light Microscope: A microscope equipped with two polarizing filters, one below the stage (the polarizer) and one above the stage (the analyzer).

Privileged Direction (of a Polarizer): The direction of vibration to which light emerging from a polarizer has been restricted.

Refractive Index: For a particular transparent medium, the ratio of the speed of light in a vacuum to the speed of light in that medium.

Relative Refractive Index: The estimate of the refractive index of a fiber in relation to the index of its surrounding medium.

Retardation (r): The actual distance of one of the doubly refracted rays behind the other as they emerge from an anisotropic fiber. Dependent upon the difference in the two refractive indices, $n2 - n1$, and the thickness of the fiber.

Sign of Elongation: Referring to the elongation of a fiber in relation to refractive indices. If elongated in the direction of the high refractive index, the fiber is said to be positive, and if elongated in the direction of the low refractive index, it is said to be negative.

Spherulites: Spheres composed of needles or rods all oriented perpendicular to the outer surface or a plane section through such a sphere. A common form of polymer crystallization from melts or concentrated solutions.

Stereomicroscope: A microscope containing two separate optical systems, one for each eye, giving a stereoscopic view of a specimen.

Surface Dye: A colorant bound to the surface of a fiber.

Synthetic Fibers: A class of manufactured polymeric fibers that are synthesized from chemical compounds (e.g., nylon and polyester).

Technical Fiber: A bundle of natural fibers composed of individual elongated cells that can be physically or chemically separated and examined microscopically for identifying characteristics (e.g., hemp, jute, and sisal).

Thermoplastic Fiber: A synthetic fiber that will soften or melt at high temperatures and harden again when cooled.

Thickness (T): The optical path through the fiber used for the calculation of birefringence, typically measured in micrometers.

4.0. Summary of Microscopy Guidelines

Textile fibers are examined microscopically. They are mounted on glass microscope slides in a mounting medium under a cover slip. The fibers are then examined microscopically with a combination of various illumination sources, filters, and instrumentation attached to a microscope to determine their polymer type and record any microscopic characteristics. Known and questioned fibers are then compared to determine if they exhibit the same microscopic characteristics and optical properties.

5.0. Significance and Use

Microscopic examination provides the quickest, most accurate, and least destructive means of determining the microscopic characteristics and polymer type of textile fibers. Additionally, a

point-by-point and side-by-side microscopic comparison provides the most discriminating method of determining if two or more fibers are consistent with originating from the same source. These guidelines require specific pieces of instrumentation outlined herein.

6.0. Sample Handling

6.1. Preparing Samples
Items of evidence are visually inspected, and tweezers are used to remove fibers of interest. Simple magnifiers and stereomicroscopes, with a variety of illumination techniques, may also be employed. Other methods such as tape lifting or gentle scraping are usually conducted after a visual examination. Tape lifts should be placed on clear plastic sheets, glass microscope slides, or another uncontaminated substrate that eases the search and removal of selected fibers. Do not overload the tapes. The tape lifts or any material recovered from scraping should be examined with a stereomicroscope, and fibers of interest should be isolated for further analysis. Fibers on tape lifts are removed using tweezers, other microscopic tools, and solvents (1-6). Tape should not be attached to paper or cardboard.

6.2. Avoiding Contamination
Take care to ensure contamination does not occur. This must be accomplished by examining questioned and known items in separate areas, at different times, or both. The work area and tools must be thoroughly cleaned and inspected before examining items that are to be compared.

7.0. Analysis

7.1. Microscopy
Fibers should be first examined with a stereomicroscope. Physical features such as crimp, length, color, relative diameter, luster, apparent cross section, damage, and adhering debris should be noted. Fibers are then tentatively classified into broad groups such as synthetic, natural, or inorganic. If the sample contains yarns, threads, or sections of fabric, construction should be recorded (7-9).

7.1.1. Side-by-Side Comparisons. If all of the characteristics are the same under the stereoscope, the next step is to examine the fibers with a comparison microscope. This side-by-side and point-by-point examination is the best technique to discriminate between fibers, especially those that appear to be similar. The physical characteristics of the fibers (see subsection 7.3) must be compared visually with the comparison microscope to determine if they are the same in the known and questioned samples. Photography is recommended to capture the salient features for later demonstration.

7.1.2. Illumination and Magnification. Comparisons should be made under the same illumination conditions at the same magnifications. For comparison microscopes, this requires color balancing the light sources. This is best achieved with two fibers or fiber samples from the same source mounted on two microscope slides, which are then compared. The visual responses from the two samples must be approximately the same color, brightness, and clarity. A balanced neutral background color is optimal.

7.2. Fiber Mounts

Many suitable media are available as temporary and permanent fiber mounts. The choice of mountant depends on availability, the particular application, and examiner preference. However, the following certain criteria (5, 10-15) must be met:

7.2.1. Mounting Media. An examiner should be aware of the possible deleterious effects that a mounting medium (especially solvent-based media) has on textile fibers, particularly when mounted for a long time. It is preferable that the mounted fibers that were previously examined microscopically be used for chemical analysis. If fibers must be removed for further testing, the mounting medium should be removed with a solvent that will not affect the structure or composition of the fiber.

7.2.2. Consistency of Mountants. Fibers that are to be compared microscopically must be mounted in a mounting medium. The same mountant should be used for both questioned and known fibers.

7.2.3. Indexing Mountants. If a solvent-based mounting medium is used for refractive index (q.v.) determination, the index of the mountant should be checked periodically against solid refractive index standards and, if necessary, readjusted to its proper value by the addition of solvent (16). Additionally, the refractive index of the medium can be measured directly, and the value can be recorded by the examiner. If such a medium is used for permanent mounts, the examiner should be aware of the different refractive indices for the fluid medium and the resin after solvent evaporation.

7.2.4. Using Liquids. Liquids used for exact refractive index determinations should be known to within +0.0005 refractive index units at nD. To make appropriate temperature corrections, values for the temperature coefficient (dn/dt) for each liquid and a thermometer covering the range 20-30°C, calibrated in tenths of a degree, should be available. High dispersion liquids (V < 30) are desirable for dispersion staining and the Becke line method (17). Cargille refractive index liquids are suitable for this purpose and are recommended for refractive index measurements of fibers.

7.3. Physical Characteristics of Manufactured Fibers

7.3.1. Fiber Diameter. The diameter of circular fibers can be measured using a calibrated eyepiece graticule. Noncircular fibers require special considerations (18). If fiber diameters are not uniform within a sample, a determination of the range of diameters exhibited by the sample is recommended.

7.3.2. Fiber Color. Color can be uniform along the length of a fiber, or it can vary. Variation in color between fibers in a sample should be recorded. The examiner should be able to distinguish between dyed, surface-dyed, and pigmented fibers.

7.3.3. Delustrant Particles. The presence or absence of delustrant particles is a useful comparative feature. If present, the size, shape, distribution, relative abundance, and general appearance should be noted. Delustrant particles, although not indicative of any particular

generic fiber type, can be characteristic of end-use properties needed by a manufacturer. Also, delustrants serve to eliminate all but manufactured fibers.

7.3.4. Cross-Sectional Shape. When viewed longitudinally on glass slides in a suitable mountant, the apparent cross-sectional shape of fibers can often be determined by slowly focusing through the fiber (optical sectioning). Actual fiber cross sections provide the best information on cross-sectional shape. (See section 8.1)

7.3.5. Fiber Surface Characteristics. Record fiber surface characteristics such as manufacturing striations, damage, and surface debris (e.g., droplets, blood, or other foreign material). Surface striations are more apparent in a mounting medium of refractive index significantly different from those of the fiber (7).

7.4. Physical Characteristics of Natural Fibers

7.4.1. Physical Features. Color, diameter, and miscellaneous physical features described previously should be noted for natural fibers. The following characteristics should also be noted.

7.4.2. Morphological Features of Animal Hairs. The principal morphological features of animal hairs are the root, medulla, cortex, and cuticle. Shield size and subshield strictures are also useful traits for species identification. Medullary and cortical structures are best observed on hairs mounted on a slide with a suitable mounting medium. Cuticular scales are best observed on replicas cast in a transparent polymer (scale casts). Scale counts (scales per 100 micrometers) can help distinguish specialty fur fibers (19-22). Silk, a protein fiber produced by caterpillars, has morphological features that differ from animal hairs. Some features of silk include crossover marks and a wedge to triangular cross section with rounded corners. In textiles, silk is occasionally seen as paired fibers cemented together, but it is most often found as single fibers (23).

7.4.3. Plant Fibers. Plant fibers can be encountered as the technical fiber (cordage, sacks, and mats) or as individual cells (fabrics and paper). The examination of technical fibers should include a search for epidermal tissue and crystals and the preparation of a cross section. Additionally, a chemical test for lignin may be performed. Technical fibers should be macerated, fabrics teased apart, and paper repulped for the examination of individual cells. Relative thickness of cell walls and lumen, cell length, and the presence, type, and distribution of dislocations should be noted. The direction of twist of the cellulose in the cell wall can also be determined (24). Other characteristic cells should be noted and compared to authentic specimens (25-27).

7.5. Physical Characteristics of Inorganic Fibers

7.5.1 Asbestos Minerals. Mineral fibers are commonly called asbestos, which is a general term for many naturally occurring fibrous hydrated silicate minerals. The asbestos minerals include chrysotile, amosite, crocidolite, fibrous tremolite/actinolite, and fibrous anthophyllite. Chrysotile belongs to the serpentine group of minerals that are layer silicates. The other asbestos minerals are amphiboles and are classified as chain silicates. Asbestos fibers alone or mixed with other

components occur in building materials and insulation products. Chrysotile is the only asbestos mineral that would be encountered as a woven fabric, but any of the asbestos minerals are found in pressed sheets such as gaskets. Take care when analyzing asbestos fibers because they are considered a potential health hazard.

All asbestos minerals can be easily identified by their optical properties using polarized light microscopy. Although not considered essential, the dispersion staining technique is extremely helpful (28-29). Scanning electron microscopy with energy dispersive spectrometry can also be used to characterize the asbestos minerals. Nonmicroscopical techniques for asbestos identification include X-ray diffraction and infrared spectroscopy.

Glass fibers are often encountered in building materials and insulation products. Glass fibers are also called manmade vitreous fibers (30). On the basis of the starting materials used to produce glass fibers, they can be placed into three categories: fiberglass (continuous and noncontinuous), mineral wool (rock wool and slag wool), and refractory ceramic fibers (glass ceramic fibers). Single crystal and polycrystalline refractory fibers such as aluminum oxide, silicon carbide, zirconium oxide, and carbon are not included because they are not considered glass fibers.

Light microscopy is used, together with classical immersion methods, to determine the refractive index for the classification and comparison of glass fibers. The dispersion staining technique is used when determining the refractive index and variation of the refractive index within a sample. Determination and comparison of the refractive index of noncontinuous (fiberglass) wool, rock wool, and slag wool can also be accomplished by annealing the fibers and using the double variation method (31-33). Solubility tests using 10 percent HCl should be conducted and the results noted. A binder resin that fluoresces under UV light may also be present on some glass wool products.

Scanning electron microscopy with energy dispersive spectrometry is used to provide elemental composition. Elemental ratios are used for comparison purposes. It is necessary to eliminate any absorption effects when acquiring the energy dispersive spectrum. Otherwise, artificial variation in the elemental composition is introduced (34).

7.6. Optical Characteristics

Detailed discussions of optical characteristics are provided by McCrone (35-38), McCrone, McCrone, and Delly (18), Bloss (39), Chamot and Mason (40), Hartshorne and Stuart (41), and Stoiber and Morse (42).

7.6.1. Refractive Index. The refractive index, n, of a transparent material is n = (speed of light in a vacuum)/(speed of light in the material). All transparent fibers other than glass display two principle refractive indices, one for light polarized parallel to the long axis of the fiber (n_{ll}) and one for light polarized perpendicular to the long axis of the fiber ($n_\perp$). For fibers examined in unpolarized light, a third quantity, n_{iso} (defined as $1/3[2\,n_\perp + n_{ll}]$), may also be estimated. Because refractive index varies with wavelength and temperature, a standard refractive index (n), is defined for all transparent materials as the refractive index at a wavelength of 589 nm (the D line of sodium) at 25°C.

The refractive indices of a fiber are determined by several methods. Whatever the method used, determination of n_{ll} and $n_{\perp}$ should be made using plane polarized light with the fiber aligned parallel and perpendicular to the privileged direction of the polarizer, respectively. The vibration direction of the polarizer should coincide with the horizontal line of the eyepiece graticule.

Refractive index measurements are either relative or exact. A relative refractive index measurement involves (a) determining whether an immersed object is higher or lower in refractive index than the immersion medium and (b) estimating the approximate refractive index on the basis of amount of contrast between the fiber and the medium. The contrast shows the amount of difference between the fiber and the medium. Exact numerical values for n_{ll} and $n_{\perp}$ of a fiber (at 589 nm at 25°C) can be determined by the Becke line method or by dispersion staining. Measurements using these methods have a precision of ±0.001 (18).

For a fiber displaying two refractive indices, birefringence is defined as $|n_{ll} - n_{\perp}|$. Birefringence is determined measuring n_{ll} and $n_{\perp}$ and using the previous formula or by determining the retardation with the corresponding thickness of the fiber and calculated with the following formula:
Retardation (nm)/Birefringence = 1,000 × Thickness (μm)

The retardation can be estimated by observing the interference color displayed at the point where the thickness of the fiber is measured and by comparing it to the Michel-Lévy chart. Take care when interpreting results from deeply dyed fibers, as the dye can obscure the interference colors. A wedge slice through the fiber; the use of various compensators such as the Sénarmont, quartz wedge, and tilting (Berek); or both can be used to make a more accurate determination of retardation (43). When measuring retardation of a fiber using a tilting compensator or quartz wedge, one must assure no error has been introduced because of differences in dispersion of birefringence between the compensator and the fiber (44). This is of special concern with the examination of fibers with high birefringence. The birefringence of noncircular fibers is estimated by measuring both retardation and thickness at two points along the fiber that represent their highest and lowest values (45).

7.6.2. Birefringence. For a birefringent fiber, the sign of elongation is positive (+) if $n_{ll} > n_{\perp}$ and negative (-) if $n_{ll} < n_{\perp}$. It should be noted that all common manufactured fibers with a birefringence higher than 0.010 have a positive sign of elongation. Full- or quarter-wave compensators are commonly used to make this determination for fibers with low birefringence (5, 39).

7.6.3. Pleochroism. Pleochroism (or dichroism) is the differential absorption of light by an object when viewed at different orientations relative to the vibration direction of plane polarized light. Certain dyed fibers and some mineral fibers may exhibit pleochroism.

7.6.4. Fluorescence. Fluorescence is the emission of light of a certain wavelength by an object when excited by light of a shorter wavelength (higher energy). Fluorescence may arise from fibers themselves or from dyes and other additives. Fibers should be mounted in a low- to- nonfluorescent medium to observe fluorescence. Examination using various combinations of

excitation and barrier filters is desirable. At each excitation wavelength, the color and intensity or absence of fluorescence emission should be noted (5, 7, 46-50).

7.7. Miscellaneous Techniques

7.7.1. Preparing Cross Sections. Physical cross sections from fibers as short as 1 mm can be prepared. Manufactured and vegetable fibers may be sectioned anywhere along their length (54-59). Animal hairs may be sectioned to yield additional identifying characteristics (60-61). When observing manufactured fiber cross sections, the general shape and distribution of delustrant, pigment particles, or both; the presence and size of spherulites or voids; depth of dye penetration; and surface treatments should be recorded when present. The fiber dimensions measured from a cross section can be used for the calculation of birefringence and the determination of the modification ratio of multilobed fibers.

7.7.2. Solubility Testing. Solubility is a destructive method. Solubility testing can, however, provide supplemental information to nondestructive methods. Possible reactions of fibers to solvents include partial and complete solubility, swelling, shrinking, gelling, and color change. If solubility tests are used as part of an identification scheme, appropriate controls should be run following the laboratory's quality assurance and control guidelines for a lot or batch of reagents or solvents. It is desirable to view known and questioned fibers simultaneously when comparing their solubilities (5, 62-64).

7.7.3. Heat Effects. A polarized light microscope equipped with a hot stage is recommended for observations of the effect of heat on thermoplastic fibers. Using slightly uncrossed polars, one may observe droplet formation, contraction, softening, charring, and melting of fibers over a range of temperatures. These observations, including melting temperature or temperatures, should be recorded. Because manufactured fibers are composed of mixtures of chemical compounds rather than pure polymers and are a combination of crystalline and amorphous regions, changes are observed over a temperature range rather than at a single melting point (5, 7, 65-69). Fibers should be mounted in an inert, heat-resistant medium, such as a high-temperature-stable silicone oil, to ensure reproducible melting behavior (70-71). Accurate and reproducible results are best obtained using a heating rate of no greater than 1-2°C/minute when near the initial melting temperature. The hot stage should be calibrated using appropriate standards, following established guidelines. The recommended melting point apparatus should be adjustable for temperatures from ambient to at least 300°C, in increments of 0.1°C, and should allow a heating rate of as low as 1°C/minute (72-79).

7.7.4. Fiber Surface Morphology. Scanning electron microscopy with energy dispersive spectroscopy (SEM-EDS) is used as an imaging and microanalytical tool in the characterization of fibers. Fiber surface morphology can be examined with great depth of field at continually variable magnifications. Fibers, prepared cross sections, or both are mounted to a specimen stub and may be conductively coated to prevent possible electron beam charging. The use of a suitable calibration standard is recommended for the accurate measurement of fiber cross sections.

Applications of SEM-EDS to fiber analysis include the characterization of fiber cross sections, identification of pigments and delustrants by elemental analysis, fiber damage due to cuts and tears (51-53, 80-82), trace debris on fibers, surface feature modifications such as washer/dryer abrasion (83), and acid-washed treatment of denim garments (84). Authors have examined fiber bonding in nonwoven fabrics and shrink-proofing treatment of wool (85). Surface imaging using the SEM-EDS as an aid in the identification of animal hair-scale structure has been reported (86).

8.0. Report Documentation

The examiner's analytical notes should reflect the particular characteristics used in the microscopic comparison, especially any calculated values, descriptions, diagrams, or photographs. A positive association is when the questioned and known fibers exhibit the same microscopic characteristics and optical properties in all tested parameters and are therefore consistent with originating from the same source. A negative association is when the questioned and known fibers are different in some significant aspect and are therefore from separate sources. An inconclusive result indicates that no conclusion could be reached, and some explanation is required as to why a definitive conclusion was not possible.

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Chapter 3 of Forensic Fiber Examination Guidelines

Visible Spectroscopy of Textile Fibers

1.0. Scope

A quantitative and objective method of color analysis and comparison is an integral part of any fiber color comparison. Visible spectroscopy can be used for this purpose. When it is used only in the visible wavelength range, the additional use of thin-layer chromatography is recommended as a complementary technique for dye analysis. The calculation of complementary chromaticity coordinates (colorimetry) is not required for forensic fiber color comparisons.

2.0. Reference Documents

SWGMAT Quality Assurance Guidelines
SWGMAT Trace Evidence Handling Guidelines
ASTM E1492-92 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Laboratory
ASTM E175-83 Terminology of Microscopy

3.0. Terminology

Absorbance: The measure of concentration of material present, that is, the negative log (base 10) of transmittance [-log 1/T] of product of extinction coefficient, pathlength, and concentration, written as A = 0bc.

Calibration: Determining the response of some analytical method to known amounts of pure analyte.

Concentration: The amount of solute in a given volume of solution.

Frequency: The number of times per unit time that the magnitude of an electromagnetic wave goes from maximum to minimum then back to maximum amplitude.

Grating: A reflective surface covered with evenly spaced, microscopic grooves, whose purpose is to separate the individual wavelengths from white light. The distance between grooves and the angle of the faces are determined by the wavelengths to be separated. The grating (except for diode arrays) is rotated at a set speed, and the desired wavelength is emitted through an exit slit onto the sample or standard.

Noise: Any signal generated by the detector not directly responding to the light transmitted at the required wavelength.

Pathlength: The distance the light passes through the sample.

Scanning: The process where the wavelength range of the system is viewed in order, usually from lowest to highest wavelength.

Slit-Width: Size of the opening of slit through which light emerges. Size depends on wavelength range, separation ability of wavelength selector, and desired isolation of specific wavelength.

4.0. Summary of Guidelines

These guidelines are concerned with the application of quantitative and qualitative visible microscopical spectroscopy, within the range of 380 nm to 760 nm, to single questioned fibers and to sets of known fibers in forensic investigations.

The method described in these guidelines has some limitations including its unsuitability for use on opaque fibers that have not been reduced in cross section before analysis, fibers with a colorant level that is insufficient for detection, and cases where different fibers have been colored with different compounds of very similar chemical structure, such as some varieties of synthetic indigo dyes.

5.0. Significance and Use

These guidelines are intended to help and advise individuals and laboratories that conduct forensic fiber examinations and comparisons in their effective application of visible spectroscopy to the analysis of fiber evidence. It is intended to be applicable to a wide range of visible-range spectrometers.

6.0. Sample Handling

The general handling and tracking of samples shall meet or exceed the requirements of ASTM 1492-92 and the relevant portions of SWGMAT Quality Assurance Guidelines.

7.0. Analysis

Because software and hardware configurations vary between instrumentation and manufacturers, the operator or operators must be familiar with the manufacturer's operating manuals.

7.1. Mounting of Fibers or Sample Preparation

Critical microspectrophotometric analysis requires that the specimen mounting medium must have low to negligible visible or ultraviolet fluorescence. Mounting media meeting this criterion include XAM, glycerol, Phytohistol, Fluoro Mount, Permount, and Norland Optical Adhesive 65. Some of these media exhibit weak fluorescence but not at intensities that interfere with the subject analysis. This list is not meant to be totally inclusive or exclusive.

Occasionally, an aromatic solvent reduced mounting media, such as XAM, has an adverse effect on some fiber dyes and fluorescent brighteners, dissolving them and allowing them to diffuse from the fiber. This normally happens very quickly after mounting. If, on mounting the known sample, bleeding is apparent, use another mountant for the preparation of the known and questioned fibers.

It is important that a minimum amount of mountant be used consistent with a thin, flat, and void-free preparation. Ensure that the longitudinal axis of the fiber remains parallel (as far as possible) to the plane of the microscope slide surface.

7.2. Known Fiber Sample Selection

Known fiber sample selection should represent the complete range of fiber colors and dyeing depths represented in the known fabric, yarn, or other fiber source. Take care to ensure that the sample reflects the extent of wear; biological deterioration; thermal or mechanical change, or both; bleaching; and laundering artifacts exhibited by the item. Known fibers should be well separated (microscopically) and mounted the same as the questioned fibers, ensuring that the fibers are mounted in a single layer.

7.3. Spectrophotometers System Calibration

Before calibration and operational use, all visible spectroscopy components should have a warm-up period; the amount of time will depend on the instrumentation used. Absorption spectrophotometry is an inherently quantitative procedure and requires appropriate calibration of wavelength and photometric response. It is essential that a wavelength calibration be run at least monthly. Calibration prior to any casework (to a maximum of once per day) ensures proper system functioning and provides a simple paper trail to detect and correct any systematic errors that occur. This can be accomplished by using primary or secondary standard filters such as holmium or didymium oxide glass, which have well-documented absorption peaks. Besides providing a check on wavelength accuracy and spectrometer resolution, a record of the absorption values found for the calibration peaks during previous calibration runs can provide a day-to-day check on the precision of the photometer absorption values. This check should not replace periodic photometric calibration.

The instrument-operating parameters for the calibration run should be the same as those that will be used for normal casework scans. To provide comparable daily calibration data, the set-up in the optical path must be reproducible. This includes the setting of the objective in a consistent focal position; keeping the measuring, luminous field diameters, or both at similar relative size (q.v.); and placing the calibration standard at a constant point in the optical path.

Once every three to six months, or before casework where the intervening interval between analysis has exceeded this period, it is essential that the performance of the instrument be evaluated comprehensively. This will involve the use of the same wavelength calibration standards used in the daily calibrations but with instrument settings chosen to maximize system accuracy, precision, and resolution. The minimum slit widths (for increased resolution); increased scan optimization, the averaging of more photometer readings at each scan step (for improving signal-to-noise ratio and photometer precision), or both; and selecting the minimum scan or data-collection step available is recommended. There should be at least two scan steps or data-collection points per resolution unit.

Under optimized conditions, the system's wavelength accuracy should be within plus or minus one resolution unit. Larger values might suggest the need to apply a calibration offset value to the monochromator during system start-up or the need for system maintenance.

Instrumental parameters used during absorbance calibration should be the same as the parameters selected for fiber analysis. Such parameters include, but are not limited to, aperture sizes and alignments, resolution settings, scan rates, and scanning ranges. The use of the same settings will ensure that calibration noise levels, system dynamic range, and linearity represent casework results.

7.4. Instrumental Photometric Accuracy and Stability
Instrumental photometric accuracy and stability can be established using either primary or secondary neutral-density absorbance standards. These standards can either be gray, glass-absorbance filters or coated, wide-band interference filters. A typical assortment of absorbance standards might have values of 0.1A, 0.3A, 0.5A, 1.0A, 2.0A, and 3.0A. These will serve to establish system absorbance linearity and dynamic range.

The filters can be placed either at, or outside, the sample focal point or at a conjugate focal point, but the filters should be referenced against a piece of clear glass of similar thickness and refractive index to that of the filter. When they are not placed in the sample plane, a blank slide with appropriate mountant and cover glass should be in the sample plane to ensure that Köhler illumination is maintained.

7.5. Instrument Photometric Accuracy
Instrument photometric accuracy should be within ±5% transmittance (%T) or ±0.02 absorbance units (A) for true values above 0.1A (< 80%T). Instrument photometric stability or precision should be within half the allowed accuracy variation or ±0.005A for true values greater than 0.1A. Day-to-day photometric accuracy and precision can be checked as previously described. Photometric response should be linear between 0.1A and 2.0A, within ±5% transmittance (%T) or ±0.02 absorbance units (A) for true values greater than 0.1A (< 80%T).

7.6. Calibration Records
Calibration records must be maintained on hard copy or computer disc and should include the date, the system parameters, and the original instrument output data, including system background scans and unratioed object or sample scans.

Many other system parameters can be measured and recorded such as dark current, 100% line stability versus time, and scattered light interference. These measurements will be sufficient to maintain quality assurance on the instrumentation.

7.7. Microspectrophotometer Apertures

The apertures that control the areas (fields) of sample illumination or detector measurement in a microspectrophotometer can be of selectable fixed size, variable size, or both and can be either rectangular or circular. The relative position and size can greatly affect microspectrophotometer performance.

7.7.1. Circular Versus Rectangular Apertures. In systems with circular (pinhole) aperture systems, the relative size of circular illumination (field) and detector (measuring) apertures can vary in much the same manner as rectangular apertures, but their size (diameter) ratio seldom exceeds 1:2. These diaphragms are generally composed of a series of fixed diameters rather than being continuously variable. These systems are not as sensitive to sample orientation as are slit aperture systems, but their signal-to-noise ratios can be lower because of their reduction in sampling area size.

7.7.2. Luminous Field Versus Measuring Field. If the measuring field is set smaller than the luminous field, the luminous field is set as large as possible within the edges of the sample. The measuring aperture can then be selected with a diameter equal to or less than the luminous field. This setting yields a relatively high absorbance value for a given dye depth by eliminating stray light at the edges of the sample from entering the detector. The drawback to using this aperture combination is in its sensitivity to sample and luminous field aperture-focusing errors and in its difficulty to use reproducibly.

7.7.3. Larger Measuring Field. A measuring field diameter can be selected such that it is larger than the sample width. This aperture combination allows the collection of stray light from all illuminated regions of the sample, including diffracted and scattered light at the sample edges. This setting is less sensitive to focusing errors and more easily yields reproducible measurements from a single sample position (i.e., improves precision).

7.8. Focusing

Samples should be focused and centered on the optical axis of the system. The focus should be set as close to the center of the sample volume as sample geometry and cross-sectional shape permit. The system should be designed and set up for Köhler illumination with the sample preparation in focus on the microscope stage. The luminous field must be centered on the optical axis of the system.

After the luminous field aperture (rectangular or circular) is centered below the focused sample, the sample is moved aside and the condenser does not produce a magenta diffraction image on the aperture edges. The sample is then repositioned over the luminous field. This focusing can be accomplished without moving the sample but doing so increases the difficulty of focusing.

The system detector-measuring aperture should be selected (sized) and centered over the luminous field aperture. The size and relative position of the apertures must not vary between the sample (object) scans and background (system or blank) scans in a given set of comparisons.

7.9. A System Blank or Background

A system blank or background refers to a background reference absorption spectrum and includes the absorbance contributions of all system components except the sample of interest. The sample slide, mountant, and coverslip are all considered parts of the system, beginning with the lamp power supply and ending with the data output device.

The parameters for blank scans should be identical to the parameters that will be used for the sample (object) scans. These parameters include voltages, scan rates, monochromator resolution, detector gain, scan steps, and, if available, any other system modifiers such as scan averaging routines, optimization factors, (low-energy scan delays) parametric corrections, and monochromator filter-change positions.

7.10. Optimization Factors

Optimization factors reduce monochromator scan rates and increase detector measurement times at each scanning step as system energy falls below its peak value. In nonscanning systems, a similar effect might be produced by increasing data-point averaging. The application of these factors can be used to reduce noise and to increase measurement precision and accuracy in low-energy or high-absorbance regions of the scan.

7.11. Detector Sensitivity

Detector sensitivity (gain or voltage) should be set at the maximum blank energy-transmission wavelength of the system in the scan region of interest. Monochromator resolution should be set at 5 nm or better to ensure the detection of inflection points in absorbance curves. The monochromator driver should be set to advance at least two steps per resolution unit (approximately 2 nm steps in this example) Nonscanning systems should be set to acquire at least two data points per desired resolution unit.

7.12. Visible Spectrum

The visible spectrum is generally regarded as existing between 380 nm and 760 nm, but it can vary by ±20 nm with different systems and operator preferences. Narrow portions of this region are scanned as necessary to resolve portions of the absorption spectrum, but it is necessary to scan at least the region from 400 nm to 700 nm. The photometric value at each scan step can be derived from an average of 2 to 50 measurements to improve precision and reduce signal noise. A nominal value of 10 measurements per step is usually adequate unless the sample exhibits extreme absorbance values or small cross sections.

7.13. Sample Scans

Sample scans should be run under the same conditions as those used for system blank scans. If these conditions produce unsatisfactory data, parameters can be modified and a new system blank run before new sample scans.

7.14. Saving Data
It is generally useful to save all data on disc just after it is generated and prior to calculating means or normalizing or developing a statistical analysis of the data. Any data that becomes altered during subsequent analysis can then easily be restored from saved files. This as-generated data can also serve to address challenges to processed data and satisfy opposing counsel demands.

7.15. Heterogeneity of Fibers
Most materials are heterogenous at microscopic levels and may require absorbance spectral scanning at more than one location either on one or more fibers to yield representative mean values for the whole sample. Single fibers may not be dyed uniformly, and natural fibers generally exhibit nonuniform cross sections along their length. These conditions can produce both real and apparent variations in dyeing depth at different places along a fiber. Measuring sites should be chosen to avoid obvious inhomogeneities occurring within the area being measured.

Initial evaluations may show that a single scan is sufficient for comparison if the fiber is uniformly dyed. At least five and as many as ten locations along a single fiber or fibers may need to be scanned if the measurements are needed to produce a representative mean absorbance curve and standard deviation curves for an individual fiber.

Synthetic fibers may yield good results with fewer scan locations than natural fibers. Known item samples of fibers may exhibit dyeing variations among the single fibers in each item. These sets of fibers should be sampled to exhibit the widest visual range of dyeing depths in each of them.

Consideration should be given during the sampling of the known materials to the conditions that led to the production of the questioned fiber transfer. If those conditions could lead to selectivity or bias in fiber transfer, it should be reasonably replicated during known fibers selection.

At least five fibers from a manufactured fiber set or ten fibers from a natural fiber set should be analyzed to produce a useful mean value spectral absorbance and standard deviation curve for the set. Both of the extremes and midranges of apparent dyeing depths should be represented in the scans. Take care to sample a variety of fiber thicknesses and cross sections.

8.0. Report Documentation

It is not necessary to make colorimetric measurements for the meaningful forensic comparison of colored textile fibers. The goal of the forensic examiner is to measure samples reproducibly so that they can be compared. Spectral records, such as printed data or stored on disc, must bear a case number, exhibit number, date, and name of operator. If, as is normal in case work, color values are not being measured, spectra are recorded in transmittance or absorbance according to operator preference. It is recommended, however, to use absorbance when recording spectra from very dark fibers.

Questioned and known spectra can be compared by overlaying them on a light box or by plotting them sequentially on the same graph. Each questioned fiber spectrum must be compared to the known fiber spectra, to determine if a positive association is found. The position of the peak maxima (nm), peak width, and peak intensity must all be considered.

A positive association is noted when the questioned spectrum is consistent in all absorbance values to at least one of the known spectra. A negative association (exclusion) is when either the suspect spectrum is totally different to that of any known fiber, or it falls outside the range produced by the known spectra. An inconclusive result is when there are no significant points of comparison in either the questioned or the known spectra (e.g., spectra from microscopically black or from very pale fibers that are outside the dynamic range of the instrument).

All spectra should be stored on disc (if possible) and clearly labeled so they are easily retrievable. Hard copies of all the relevant spectra should be stored in the appropriate case file. Spectra collected for reference data on color or in research work should be stored on separate discs and kept separate from case work spectra. It is optional whether chromaticity values appear on spectral printouts. The phrases spectral match or no differences are to be avoided. The wavelength range over which the spectra have been recorded should be stated.

9.0. References

This list does not contain colorimetry references.

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Chapter 4 of Forensic Fiber Examination Guidelines

Thin-Layer Chromatography of Nonreactive Dyes in Textile Fibers

1.0. Scope

Metameric coloration of fibers can be detected using UV/visible spectroscopy. If spectroscopy is restricted to the visible spectral range, differences in dye components may remain undetected. One method of detecting additional components is to use thin-layer chromatography (TLC). TLC is an inexpensive, simple, well-documented technique that can be used, under certain conditions, to complement the use of visible spectroscopy in comparisons of fiber colorants. The principle of the method is that the dye components are separated by their differential migration caused by a mobile phase flowing through a porous, adsorptive medium.

2.0. Reference Documents

SWGMAT Quality Assurance Guidelines
SWGMAT Trace Evidence Handling Guidelines
ASTM E1492-92 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Laboratory

3.0. Terminology

Activation: The heating of the adsorbent layer on a plate to dry out the moisture and maximize its attraction and retention power.

Adsorbent: The stationary phase for adsorption TLC.

Adsorption: The attraction between the surface atoms of a solid and an external molecule by intermolecular forces.

Chamber: A glass chamber in which TLC development is carried out.

Thin-Layer Chromatogram: The series of spots visible on the adsorbent layer after development.

Chromatography: A method of analysis in which substances are separated by their differential migration in a mobile phase flowing through a porous, adsorptive medium.

Development: The movement of the mobile phase through the adsorbent layer to form a chromatogram.

Dye Extraction: The removal of the dye from a fiber by incubating it in an appropriate solvent.

Eluent: The solvent mixture that acts as the mobile phase in TLC.

Extractant:

Metameric Pair: Two colors that appear the same under one illumination but different under another illumination.

Mobile Phase: The moving liquid phase used for development.

Normal-Phase Chromatogram: Adsorption in which the stationary phase is polar in relation to the mobile phase.

Origin: The location of the applied sample or the starting point for the chromatographic development of the applied sample.

Resolution: The ability to visually separate two spots.

Retardation Factor (RF): The ratio of the distance traveled by the solute spot's center divided by the distance traveled by the solvent front, both measured from the origin.

Saturation: Chamber equilibration with mobile-phase solvent vapor prior to chromatography.

Solute: In TLC, a mixture of components to be separated.

Solvent Front: The final point reached by the mobile phase as it flows up or across the TLC plate during development of the chromatogram.

Spot: A round zone of sample application at the origin, or in a chromatogram, a round zone caused by migration of a component of the solute.

Spotting: Applying a solute sample at the origin of the TLC plate.

Stationary Phase: The solid adsorbent coating layer of a TLC plate.

Tailing: A spot distorted during development into an elongated streak.

Thin-Layer Chromatography (TLC): A separation technique in which the flow of solvent causes the components of a mixture to migrate differentially from a narrow initial zone in a thinly applied porous adsorptive medium.

4.0. Summary of Guidelines

These guidelines are intended to advise and assist individuals and laboratories that conduct forensic fiber examinations and comparisons in their effective application of TLC to the analysis of fiber evidence.

These guidelines are concerned with the extraction of dyes from single fibers and from bulk material, classification of the dye or colorant, application and development of the extractants on TLC plates using an optimal elution system, and evaluation and interpretation of the resulting chromatograms. The protocols and equipment mentioned in this document are not meant to be totally inclusive or exclusive.

Not all fiber type or dye class combinations are covered in these guidelines.

5.0. Significance And Use

5.1. Forensic Analysis of Fiber Colorants Using TLC
Forensic analysis of fiber colorants using TLC should be considered for single-fiber comparisons only when it is not possible to discriminate between the fibers of interest using other techniques, such as comparison microscopy (brightfield and fluorescence) and microspectrophotometry in the visible range.

5.2. Extraction Procedures Carried Out Prior to TLC Analysis
The extraction procedures carried out prior to TLC analysis can provide useful information about dye classification. TLC can provide useful qualitative information about dye components. Similar colors made up of different dye components can be differentiated using this technique. The application of TLC may serve to discriminate between fibers, or it may confirm their similarity.

5.3. Situations That Preclude TLC
TLC is not possible in some circumstances: Short lengths of fibers or pale colored fibers do not have an adequate concentration of colorant present to be examined, dye extraction from some fibers is impossible, or desire to preserve evidence for possible analysis by another examiner precludes removing the color for analysis.

5.4. Characterizing Dyes and Evaluating Eluent Systems
Dye from the known material should first be characterized and eluent systems evaluated to

achieve optimum separation of the extract. Dye is then extracted from single known and questioned fibers, using an equivalent amount of material.

5.5. Standard Dye Mixtures Compensate for TLC's Nonreproducibility
TLC is inherently nonreproducible because of variance in eluents and temperature conditions. Standard dye mixtures (16) should therefore be used to check eluent performance.

Examples for the preparation of standard dye mixtures are given in Appendix A.

6.0. Sample Handling

6.1. Using ASTM 1492-94 Standards
The general handling and tracking of the samples should meet or exceed the requirements of ASTM 1492-94.

6.2. Pretreatment and Sample Preparation
Pretreatment (e.g., mounting medium and washing solvent) and sample preparation must be identical for all known and questioned fibers being compared on one TLC plate. For removing single fibers from slide preparations, the following procedure is recommended:

6.2.1. Clean the Coverslip. Any traces of marker pen ink should be cleaned from the coverslip using an appropriate solvent (e.g., acetone);

6.2.2. Crack the Coverslip. The coverslip should be cracked all around the fiber, and an appropriate solvent, which will dissolve the mountant but not affect the fiber or the colorant, should be used; and

6.2.3. Remove and Clean the Fiber. The fiber should be removed and washed in the solvent.

7.0. Analysis

The ease of dye extraction and the particular extractant required will depend on the generic class of the fiber and the type of dye present. The generic class of the known and questioned fibers must be determined prior to TLC analysis.

Dye classes are classified into broad groups on the basis of their chemical properties or method of application. The determination of the dye class of the known fibers can be helpful in establishing the best extractant, as well as to assist in the subsequent selection of the most efficient eluent system.

Documented extraction schemes (see Appendix B) can be used to determine the dye class of fibers of known generic classes and, thus, the optimum extractant. Dye classification is performed on single fibers or tufts of fiber removed from the known item. A new fiber or tuft can be used for each classification stage.

7.1. Dye Extraction
Known and questioned fibers must be extracted at the same time under the same conditions. Single fibers can be extracted in a short length (about 25 mm) of fine capillary tube (internal diameter of about 1.5 mm) that is sealed at one end. A fine wire can be useful in pushing the fiber down the tube. The tube must be appropriately labeled.

About 10 µL of the appropriate extractant (as recommended in Appendix C and Appendix D) should be introduced into the tube to cover the fiber sample. A fine glass pipette or syringe can be used for this. The tube should be heat sealed to avoid evaporation and incubated for a constant time and temperature (as recommended in Appendix B), preferably in an oven. Periodic checks for dye extraction should be made every 15 minutes for up to 1 hour.

7.2. Dye Extraction for Bulk Material
Larger fiber tufts (e.g., known samples) can be extracted in a Durham tube or other suitable small stoppered glass tube, using about 100 µL of solvent in a sand bath or oven heated to 100°C. Periodic checks should be made every 15 minutes for up to 1 hour.

7.3. Nonextractable Dyes
If classification indicates that a nonextractable dye or pigment other than a reactive dye is present, then place one known and one questioned fiber in labeled capillary tubes. Add approximately 10 µL pyridine/water (4:3) and attempt to extract at about 100°C for one hour. If neither fiber extracts, a positive association is noted. If the questioned extracts and the known does not (or vice versa), it is negative. If both questioned and known bleed dye into solution, there can be sufficient dye for analysis.

7.4. Elution
Aluminum-backed silica gel 60F 254 plates measuring 5 cm × 7.5 cm are recommended for normal-phase TLC of fiber dyes (16). Plates should be stored in a desiccator. If this is not possible, they should be heat activated before use.

Both known and questioned dyes to be compared must be applied to the same plate. The extract should be spotted onto the plate about 1 cm from the lower edge. This can be done using a double-drawn capillary tube or other suitable device. Spots should not be too near the edge of the plate or to each other. Care should be taken to avoid scratching the adsorbent coating layer.

Spots should be dried using a hair dryer or hot plate, and repeated applications should be made until the spot is strongly colored. The spot size should be uniform and not exceed about 2 mm in size.

At least two (preferably more) known spots should be included on each plate, on both sides of the questioned sample or samples. It is advisable to include a standard dye spot. A note must be made of the sample order on the plate itself. Plates must be thoroughly dried before developing.

7.5. Development Chamber
Chromatograms can be developed vertically in a glass chamber, which can be as simple as a covered glass beaker. Commercial tanks are available (16). Twin trough tanks allow the solvent

to be transferred to the plate side without removing the cover, but extreme care must be taken when doing this.

The eluent should be added to the tank and allowed to stand in the closed container for a few minutes before development, which allows the chamber to be saturated with the solvent vapor. (This will not be complete if a beaker is used, but equilibration is not critical when sample size is very small and the elution time is short.)

The level of the eluent in a vertical tank should be at least 0.5 cm below the origin or application spots on the TLC plate. The plate should be eluted until good resolution is achieved (normally 2 cm from the origin) but not so far as to allow the spots to become diffuse, which makes visualization difficult. The plate should be removed, and the position of the solvent front marked. The plate should be dried in a hot air stream. The eluent should be discarded.

7.5.1. Selecting the Eluent. Five parameters must be considered when selecting the optimum eluent:
7.5.1.1. Separation of component dyes;
7.5.1.2. Sharpness of bands;
7.5.1.3. Movement from the origin;
7.5.1.4. Components traveling at or close to solvent front; and
7.5.1.5. Strength of dye extract from questioned fibers.

There are numerous published TLC solvent systems that can be applied to the development of particular fiber and dye class combinations (see Appendixes C and D).

Two or more systems should be assessed with the known fibers to determine the optimum eluent system that can be used for comparison with the questioned fibers.

Equivalent lengths of fiber should be used for pale fibers or short sample lengths. The extract from known material should be applied to the TLC plate and developed in the trial eluents as previously described.

If the eluents produce poor separation, other eluents appropriate to the dye class are evaluated. In exceptional circumstances, eluents appropriate to other dye classes can be used.

After a suitable eluent system has been found, comparison of known and questioned fibers can be carried out. Co-chromatography can be carried out for bulk samples.

After drying, plates should be examined immediately in visible and in longwave ultraviolet light. Band positions and colors should be noted.

The color and fluorescence of the spots and the distance from the center of each spot to the origin can be measured and recorded. The method of documentation is a matter of individual laboratory preference.

Plates and samples must be identifiable. Plates must be either documented by photography or retained and stored out of direct sunlight in a manner designed to minimize fading or both.

8.0. Report Documentation

Chromatograms of dyes from the same fibers run in different eluent systems or on different plate types are considered mutually exclusive. The spot colors, fluorescence, sequence, and position of the spots obtained from the dye of the questioned fibers are compared to those from the corresponding known fibers.

A positive association occurs when the band colors, fluorescence, sequence, and positions are consistent between questioned and known fibers. A negative (exclusion) association is noted when either the questioned or known patterns show no similarities, or where there are a number of coincident bands, but one or more bands are missing from the questioned or known. An inconclusive association is noted when there are no bands on the TLC plate because insufficient colorant is present in the extract. In cases where the amount of extract is very small, the distance traveled by the eluent is very small, and in some cases the spots may not be well-defined. In these circumstances, attempts to calculate the Retardation Factor (RF) values can easily be inaccurate and therefore meaningless.

The TLC methods applied to the forensic comparison of fiber colorants must have been published in a recognized forensic journal, forensically relevant textbook, or in an accredited forensic laboratory manual.

Plates must be identifiable with respect to case number, sample source, examiner, and date. Case documentation on TLC must include the source of the samples; method of dye classification; details of extractants or eluent systems tested, used, or both; and the results. The use of standard dye mixtures as system performance checks is strongly recommended.

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Appendix A: Suggested Standard Dye Mixtures

Standard dyes should be used to check eluent performance. The following list suggests some suitable mixtures but is not totally inclusive or exclusive. Any (simple) mixture of dyes separating in these eluents can be used.

Recommendations for Preparation of Standard Dye Mixtures:
Approximately 5 mg of each dye component is made up to a final volume of 25 mL with pyridine/water (4:3 v/v). Use until the supply is exhausted.

Solution A for eluents 1, 2, 3, 4, 9, 10, 12
Solway green G (CI Acid Green 25), Solway blue RNS (CI Acid Blue 47) and Naphthalene fast orange 2GS (CI Acid Orange 10).

Solution B for eluents 5, 7, 8
Supracet fast orange G (CI Disperse Orange 3), Supracet fast violet B (CI Disperse Violet 8) and Supracet scarlet 2G (CI Disperse Orange 1)

Solution C for eluent 6
Supracet fast orange G (CI Disperse Orange 3) and Supracet fast violet B (CI Disperse Violet 8).

Solution D for eluent 11
Solway green G (CI Acid Green 25), Supracet fast orange G (CI Disperse Orange 3) and Supracet fast violet B (CI Disperse violet 8).

Testing of Eluents and Extraction Solutions
Checks are carried out prior to use on eluent performance, and the extractants are tested to ensure that they have not been contaminated. Suggested TLC plates are Merck DC Alufolien Kieselgel

60F254 (7.5 × 5.0 cm). The standard dye and extraction solution are spotted side by side onto a TLC plate that rests on a hot plate set at approximately 70°C or is dried with a heat source such as a blow-dryer. They are spotted 1 cm from the base and eluted as described in section 7.0 (Analysis; click to view) of Chapter 4. The chromatogram is compared with previous stored tests to ensure that the separation is acceptable and that there are no visible bands obtained from the extraction solution. If unacceptable, fresh eluent/extractant is made and tested.

Appendix B: Classification of Dyes

WOOL Fibers (17)

Stage 1
Pyridine/Water (4:3), 100°C, 10 minutes
Good extraction □□ACID dye
Little or no extraction—Go to Stage 2

Stage 2
2% aqueous oxalic acid, 100°C, 20 minutes then pyridine/water (4:3), 100°C, 10 minutes
Improved extraction □□METALIZED Dye
Little or no extraction □□REACTIVE Dye

COTTON and VISCOSE Fibers (15)

Stage 1
Glacial acetic acid, 100°C, 20 minutes
Good extraction □□AZOIC Dye
Little or no extraction
Go to Stage 2

Stage 2
Pyridine/Water (4:3), 100°C, 20 minutes
Good extraction □□DIRECT Dye
Little or no extraction
Go to Stage 3

Stage 3
Dithionite/polyvinylpyrrolidone, (see endnote 1 of this appendix) 100°C, 20 minutes (see endnote 1)
Apply extract to TLC plate; check color of spot
Fiber color changed □□REACTIVE Dye
(No colored spot or spot not original fiber color)
Fiber color unchanged □□INGRAIN Dye
(No colored spot or spot not original fiber color)
Fiber color changed

Go to Stage 4
(original colored spot)

Stage 4
10-14% Sodium hydroxide, 100°C, 10 minutes (new fiber)
Fiber color changed □□SULFUR Dye
Fiber color unchanged □□VAT Dye

ACRYLIC Fibers (13)

Stage 1
Formic acid/water (1:1), 100°C, 20 minutes
Good extraction—Go to Stage 2

Stage 2
TLC procedure—methyl acetate eluent
Movement □□DISPERSE Dye
No movement—Go to Stage 3

Stage 3
TLC procedure—methanol eluent
Sharp line at solvent front □□ACID Dye
Little or no movement or smearing □□BASIC Dye

POLYESTER Fibers (7)

Stage 1
Chlorobenzene, 130°C, 10 minutes
Good extraction □□DISPERSE Dye
Little or no extraction—Go to Stage 2

Stage 2
Dimethylformamide/formic acid (1:1), 100°C, 20 minutes
Good extraction □□BASIC Dye.

POLYAMIDE Fibers (7)

Stage 1
Chlorobenzene, 150°C, 15 minutes
Little or no extraction—Go to Stage 2
Good extraction □□DISPERSE Dye

Stage 2
Pyridine/water (4:3), 100°C, 20 minutes
Little or no extraction □□REACTIVE or DIAZO Dye
Good extraction—Go to Stage 3

Stage 3
TLC procedure—methanol eluent
Sharp line at solvent front ACID Dye
Little or no movement or smearing BASIC Dye

POLYPROPYLENE Fibers (11)

Stage 1
Methyl acetate/water/acetic acid (5:5:1), 100°C, 20 minutes
Good extraction DISPERSE Dye
Little or no extraction—Go to Stage 2

Stage 2
Pyridine/ water (4:3), 100°C, 20 minutes
No extraction PIGMENT
Some extraction—Go to Stage 3

Stage 3
2% aqueous oxalic acid, 100°C, 20 minutes then Pyridine/water (4:3), 100°C, 20 minutes
Improved extraction METALLIC Dye
No improvement ACID Dye

ACETATE/TRIACETATE Fibers (9)

Stage 1
CA pyridine/water (4:3), room temperature, 15 minutes
CTA pyridine/water (4:3), 100°C, 20 minutes
Little or no extraction = DIAZO Dye
Good extraction = DISPERSE Dye

Endnote

1. Dithionite/polyvinlypyrrolidone—Use immediately and discard excess:
80 mg sodium dithionite
30 mg polyvinylpyrrolidone
450 mL 10% sodium hydroxide
9 mL water.

Appendix C: Choice of Extractants

Dye classification indicates the best extraction solution for a particular fiber. Useful published information is summarized in the following table.

Dye Class	Fiber Type/ Reference	Extraction Solution

Acid[a]	Polypropylene/11	Pyridine/Water (4:3)
Acid	Nylon/7	
Acid	Wool/17	
Acid	Cotton/14	Glacial Acetic Acid (1:1)
Basic	Polyester/7	Pyridine/Water (4:3)
Direct	Cotton/12	
Disperse	Polyester/7	
Disperse	Polypropylene/11	
Disperse	Acetate-Triacetate/9	
Metalized	Wool/17	2% Aqueous Oxalic Acid then Pyridine/Water (4:3)
Metalized	Polypropylene/11	
Basic	Acrylic/13	Formic Acid/Water (1:1)
Reactive, Sulfur, Vat, Diazo, Ingrain and pigmented dyes do not extract		

[a] Dyes rarely extract from polypropylene, but if they do, they are most likely to be acidic.

Appendix D: Composition of Eluents

Table 1: Eluent Systems
The following list summarizes eluent systems that have been recommended in the relevant forensic literature. This list is not meant to be totally inclusive or exclusive.

Eluent	Solvents	Proportions (v/v)	Reference
1	n-Butanol, acetone, water, ammonia	5:5:1:2	8, 16
2	Pyridine, amyl alcohol, 10% ammonia	4:3:3	8, 10, 16
3	n-Butanol, ethanol, ammonia, pyridine, water	8:3:4:4:3	8, 10, 16
4	Methanol, amyl alcohol, water	5:5:2	10, 16

5	Toluene, pyridine	4:1	8, 16
6	Chloroform, ethyl acetate, ethanol	7:2:1	10
7	n-Hexane, ethyl acetate, acetone	5:4:1	8, 16
8	Toluene, methanol, acetone	20:2:1	8, 16
9a	n-Butanol, acetic acid, water	2:1:5	8
10	n-Butanol, ethanol, ammonia, pyridine	4:1:3:2	8, 16
11	Chloroform, butanone, acetic acid, formic acid	8:6:1:1	8
12a	n-Butanol, acetic acid, water	4:1:5	8, 16

The ethanol used is 99%. The ammonia is 0.880 SG unless otherwise stated.

Table 2: Eluents Recommended for Certain Dye Classes
Certain fiber type or dye class combinations have been found to give better separation in certain eluents. These are shown in the following table and are recommended as a first choice.

Fiber Type	Dye Class	Eluent Number
Wool	Acid or Metalized	1.2
Cotton and Viscose	Direct	1, 4, 3
Acrylic	Basic	11, 12, 1
Polyester	Disperse	6, 7, 8, 5
Polyamide	Acid	9, 10
Polypropyleneb		

a Dyes rarely extract from polypropylene, but if they do, they are most likely to be acidic.
b Polypropylene rarely contains an extractable dye. If the dye can be extracted, an eluent appropriate to the dye class is used.

Chapter 5 of Forensic Fiber Examination Guidelines

Pyrolysis Gas Chromatography of Textile Fibers

1.0. Scope

These guidelines are intended to assist individuals and laboratories that conduct pyrolysis gas chromatography (PGC) in their selection, application, and evaluation of PGC as a method for forensic fiber case work. Some of the procedures referenced in these guidelines involve the use of hazardous chemicals, temperatures, or some combination of both. These guidelines do not address the possible safety hazards or precautions associated with their application. It is the responsibility of the user of this document to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use.

2.0. Reference Documents

SWGMAT Quality Assurance Guidelines
SWGMAT Trace Evidence Handling Guidelines
ASTM E 1610-94 Standard Guide for Forensic Paint Analysis and Comparison

3.0. Terminology

Chromatography: A method of analysis in which substances are separated by their differential migration in a mobile phase flowing through a porous and absorptive medium.

Gas Chromatogram: The visual display of the progress of a separation achieved by chromatography. A chromatogram shows the response of a chromatographic detector as a function of time.

Capillary Column: A columnar assembly of a thin film on the inner periphery and an unobstructed and open lumen running the entire length of the column, which acts as the stationary phase or plate of the chromatograph.

Mobile Phase: In gas chromatography, the mobile phase is the inert carrier gas that moves the volatile analytes through the length of the column.

Packed Column: A metal tube evenly filled with a solid support material that is coated with a liquid stationary phase of low vapor pressure.

Stationary Phase: In a packed column, the stationary phase is a low vapor pressure liquid that coats a solid support. Compounds are selectively retained on the basis of their solubility in this liquid. In a capillary (WCOT) column, the stationary phase is generally a modified or unmodified polysiloxane compound coating the walls of a fused silica column. Compounds are selectively retained on the basis of their interaction with the coating's functional groups.

4.0. Summary of Guidelines

These guidelines are intended to advise and assist individuals and laboratories that conduct forensic fiber examinations and comparisons in their effective application of pyrolysis and pyrolysis gas chromatography (PGC) to the analysis of fiber evidence. The guidelines are concerned with the pyrolysis of single fibers and fibers from bulk material, classification of the generic class of polymer, and interpretation of the resulting pyrograms. The protocols and equipment mentioned in this document are not meant to be totally inclusive or exclusive.

5.0. Significance and Use

Pyrolysis is a destructive analytical method; therefore, consideration must be given to the applicability of this procedure to each case depending on the sample size and the amount of sample consumption that can be tolerated (8). Pyrolysis of polymers is the breaking apart of larger polymer chains into smaller fragments by the application of heat in an inert atmosphere (8). When the heat energy applied to the polymer chains is greater than the energy of specific bonds in that polymer chain, these bonds will fragment in a predictable and reproducible way at a specific temperature. In PGC, the fragments generated by pyrolysis are introduced into a gas chromatograph (GC) for separation and characterization. PGC can be used to identify the generic type of an unknown fiber, and in some cases it can identify subclasses within a generic class (1).

Each laboratory should develop its own standard chromatograms of the different generic fibers if performing fiber identification analyses. These chromatograms demonstrate the analytical potential as well as the limitations of PGC performed on fibers by a particular system.

The potential of pyrolysis gas chromatography of fibers include:
5.1. Comparative analyses of two or more fibers;
5.2. Identification analyses of known or questioned fibers or both. With regard to some fiber types, such as acrylics, PGC can be used in conjunction with infrared spectroscopy to provide differentiation within generic classes (6); and

5.3. The technique is extremely sensitive and can be used to analyze a wide variety of materials (7).

As with any instrument, PGC has limitations to its application, the two most important of which are the number of parameters and the control of these parameters. First, PGC encompasses a great variety of parameters. Fluctuation in any one of these parameters will produce pyrolysis product changes. These parameters can be related to the sample and its preparation and can include sample homogeneity, sample size, sample shape, sample placement within and contact with the quartz tube, and sample weight. Other variables related to the pyrolysis instrument include the temperature of the pyrolysis, the rate of temperature rise, the time of the pyrolysis, and the pyrolysis chamber atmosphere. Second, the variables must be controlled to ensure reproducible results. For more complex samples, the reproducibility of replicate sample pyrograms becomes more involved. Therefore, users should establish their systems' capability to discriminate various copolymer ratios.

6.0. Sample Handling

Proper sample preparation and technique are prerequisites for obtaining reproducible results. Fibers being compared should be analyzed using the same parameters and approximately the same sample size and shape (2, 3).

Samples are prepared using low-power magnification, and clean tools must be used to handle samples and the quartz tube (4).

7.0. Analysis

The user is required to maintain authenticated and traceable reference standards of fibers for comparison, identification analyses, or both. These known standards can include fibers obtained from testing services, manufacturers, or both. Control samples should be routinely run as established by laboratory procedures.

7.1. Sample Temperature
A pyrolysis instrument must be able to heat a sample to a preset temperature at a known rate for a specific amount of time. These conditions must be accurately reproducible and predictably varied (5).

The gas chromatograph used in fiber pyrolysis should (a) have a reproducible temperature profile and stable carrier gas flow rate; (b) have a capillary column capable of distinguishing different fiber types; and (c) have the capability to reproducibly separate and identify pyrolysis products.

After establishing standard methods and protocols, standard pyrograms should be run to check the temperature setting and resulting pyrogram pattern. A polymer material such as low-density polyethylene or polypropylene can be used for routine performance checks. The frequency of routine instrument performance checks should be established by each laboratory.

The instrument performance sample should be an easily obtainable material that yields reproducible chromatograms having peaks over the entire range of the pyrogram with major peaks near the start, in the middle, and near the end.

The pyrolysis unit must be checked in conjunction with the gas chromatograph at routine intervals as established by each laboratory. The pyrolyzer should be checked after the gas chromatograph has been checked.

7.2. Pyrolyzer Calibration

A pyrolyzer can be calibrated by observing the melting points (mp) of two inorganic compounds within a 7-mm band approximately in the center of the quartz tube probe. Possible compounds include potassium chloride (KCl) mp (approximately 770°C); sodium tungstate (Na_2WO_4) mp (approximately 700°C); potassium iodide (KI) mp (approximately 686°C); or potassium iodate (KIO_3) mp (approximately 560°C). Any pyrolysis unit should be recalibrated by the manufacturer when shipped for necessary repairs.

An instrument performance sample should be introduced into the GC during routine performance checks as established by each laboratory. New instrument performance sample chromatograms must be compared with previous ones in order to establish relative sensitivity, resolving power, and baseline profiles. This ensures that the case samples and the reference library are still comparable. In some cases instrument performance changes sufficiently to require that new reference standards be generated. These instrument performance sample chromatograms should be kept in the instrument logbook for a predetermined length of time as established by laboratory protocol.

7.3. Analysis Procedures

The following series of procedures must be followed for an analysis:

7.3.1. Run controls and blanks as established by individual laboratory procedures. Allowable maximum peak heights in blank samples should be defined in laboratory procedures;

7.3.2. Run an instrument performance sample according to laboratory procedures and ensure the instrument is operating properly; and

7.3.3. Adjust column head pressure and split-flow rates in accordance with established procedures.

7.4. Sample Comparison: Known to Questioned

Run known and questioned samples under the same conditions and compare their chromatograms. Known samples should be run in duplicate to assess variations in the pyrograms and to ensure reproducibility.

7.5. Identification By Means of a Reference Library

Identification is accomplished by comparison of a known sample, questioned samples, or both to a reference library. To do so, the individual performing the analysis should run the known sample and questioned sample and compare their chromatograms to the reference library. The library chromatograms should originate from the same instrument and protocol used in the current analysis. All identifications must be confirmed by running an authenticated fiber

reference standard at the time of analysis. Known samples, and questioned samples if necessary, should be run in duplicate to assess variations in the pyrograms and to ensure reproducibility.

7.6. Procedures Established by Laboratory Protocol
The following procedures should be performed as established by laboratory protocol:
7.6.1. Check to ensure even spacing along the platinum coil on a pyroprobe coil unit. This coil should be visually inspected before each use;
7.6.2. The pyrolysis quartz tube must be heat cleaned after each use. If the quartz tube is reused, each laboratory should develop, document, and use a cleaning procedure that can be demonstrated to be noncontaminating to subsequent runs; and
7.6.3. Check gas cylinders and change when the pressure drops to a predetermined level. If gas-line moisture traps, oxygen scrubbers, and so forth are being used, these should be changed when tanks are changed or as necessary to maintain system performance.

7.7. Scheduled Routine Maintenance Procedures
Scheduled routine maintenance procedures must be performed per individual laboratory procedures. Record performed maintenance in an instrument logbook. This must include cleaning the detector, reassembling the detector and checking flows, changing GC septa and pyroprobe O-ring seals, cleaning injection port, checking glass liners during routine maintenance, performing other cleaning as needed, and performing any additional scheduled routine maintenance.

Instrument performance must be checked whenever a new column is installed or whenever repairs are done to the pyrolyzer.

8.0. Report Documentation

Documentation must include data obtained through the analytical process. Deviations from the written protocol (other than standard operating procedures) must be documented.

The following instrumental variables (parameters) must be recorded in the laboratory and be accessible for later reference or included in the case file:
8.1. Specific GC used;
8.2. Type of GC column, including
8.2.1. Length;
8.2.2. Diameter;
8.2.3. Coating;
8.2.4. Coating thickness;
8.2.5. Type of carrier gas and detector;
8.2.6. Flow rates;
8.2.7. Split flow (if applicable); and
8.2.8. Chart speed (if applicable);
8.3. Oven temperature program, including
8.3.1. Initial temperature;
8.3.2. Ramp rates;
8.3.3. Final temperature-to-temperature holding durations;

8.3.4. Injector and detector temperatures; and
8.3.5. Specific pyrolyzer unit used;
8.4. Pyrolysis temperature, including
8.4.1. Interface temperature;
8.4.2. Ramp rates;
8.4.3. Final temperature; and
8.4.4. Temperature holding durations (interval).

The data generated by PGC is dependent upon various factors such as sample size, condition, and handling. Likewise, interpretation of PGC data is dependent upon the training and experience of the examiner. Awareness of the strengths and limitations of the technique must be considered. In addition, the examiner must assess the variability of the instrument and variations within the pyrograms. Therefore, the examiner must complete a formalized training program conducted under the supervision of an experienced examiner prior to casework. This training program must include analyzing and comparing pyrograms of various polymers in order to discriminate between polymer types.

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Chapter 6 of Forensic Fiber Examination Guidelines

Infrared Analysis of Textile Fibers

1.0. Scope

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 (FT-IR) spectrometers has greatly simplified the IR analysis of single fibers, thus making the technique feasible for routine use in the forensic laboratory.

These guidelines are 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 these guidelines are 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.

2.0. Reference Documents

SWGMAT Quality Assurance Guidelines
SWGMAT Trace Evidence Handling Guidelines
ASTM E1492-92 Practice for Receiving, Documenting, Storing, and Retrieving Evidence in a Forensic Laboratory
ASTM E 131-93 Terminology Relating to Molecular Spectroscopy
ASTM 1421-94 Standard Practice for Describing and Measuring Performance of Fourier Transform Infrared (FT-IR) Spectrometers: Level Zero and Level One Tests

3.0. Terminology

Absorbance (A): the logarithm to the base 10 of the reciprocal of the transmittance, (*T*).

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

Absorption Band: A region of the absorption spectrum in which the absorbance passes through a maximum.

Absorptivity (a): Absorbance divided by the product of the sample pathlength (b) and the concentration of the absorbing substance (c). $a = A/Bc$

Absorption Spectrum: A plot, or other representation, of absorbance, or any function of absorbance, against wavelength, or any function of wavelength.

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.

Background: Apparent absorption caused by anything other than the substance for which the analysis is being made.

Cellulosic Fiber: Fiber composed of polymers formed from glucose.

Far-Infrared: Pertaining to the infrared region of the electromagnetic spectrum with wavelength range from approximately 25 to 1,000 μm (wavenumber range 400 to 10 cm^{-1}).

Fourier Transform (FT): A mathematical operation that converts a function of one independent variable to one of a different independent variable. In Fourier transform infrared (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.

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 transform to obtain an amplitude-wavenumber (or wavelength) spectrum.

Generic Class: A group of fibers having similar (but not identical) chemical composition. 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 (12).

Infrared: Pertaining to the region of the electromagnetic spectrum with wavelength range from approximately 0.78 to 1,000 μm (wavenumber range 20,000 to 4,000 cm^{-1}).

Infrared Spectroscopy: Pertaining to spectroscopy in the infrared region of the electromagnetic spectrum.

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.

Manufactured (Man-Made) Fiber: Any fiber derived by a process of manufacture from any substance that is not, at any point in the manufacturing process, a fiber.

Mid-Infrared: Pertaining to the infrared region of the electromagnetic spectrum with wavelength range from approximately 2.5 to 25 m (wavenumber range 4,000 to 400 cm^{-1}).

Spectrometer: Photometric device for the measurement of spectral transmittance, spectral reflectance, or relative spectral emittance.

Subgeneric Class: A group of fibers within a generic class that shares the same polymer composition. Subgeneric names include, for example, nylon 6, nylon 6,6, and poly(ethylene terephthalate).

Transmittance (T): The ratio of radiant power transmitted by the sample, I, to the radiant power incident on the sample, I_o . $T = I/I_o$

Wavelength: The distance, measured along the line of propagation, between two points that are in phase on adjacent waves.

Wavenumber: The number of waves per unit length, in a vacuum, usually given in reciprocal centimeters, cm^{-1}.

4.0. Summary of Guidelines

These guidelines cover identification of fiber polymer composition by interpretation of absorption spectra obtained by microscopical infrared spectroscopy. They are intended to be applicable to a wide range of infrared spectrometer and microscope configurations.

Spectra may also be obtained by a variety of alternative IR techniques. Other techniques (not covered in the scope of these guidelines) 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 surface of the prism (1):

4.1. Diamond Cell (Medium or High Pressure)
This technique is used with a beam condenser (2-4). This combination is frequently used with a spectrometer configured for mid- and far-IR and thus provides a wider spectral range;
4.2.2. Thin films: solvent (5, 6), melt (3), or mechanically prepared (7);
4.2.3. Lead foil technique (5); and
4.2.4. Micro-KBr (or other appropriate salt) pellets (8, 9).

This list is not meant to be totally inclusive or exclusive.

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

Polyester
Vinal[4]
Acrylic
Modacrylic
Rayon
Vinyon
Anidex (no longer produced in the United States [10])
Novoloid[4]
Saran
Aramid
Nylon
Spandex
Azlon[4]
Olefin
Sulfar
Fluorocarbon
Polybenzimidazole (PBI)
Triacetate
NytrilPolycarbonate
Rubber

Although natural fibers may also be analyzed by IR spectroscopy, they are excluded from these guidelines because no additional compositional information is provided over that yielded by light microscopy.

5.0. Significance and Use

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 spectrometer coupled with an IR microscope. Fiber polymer identification is made by comparison of the fiber spectrum with reference spectra.

Consideration should be given to the potential for additional compositional information that may be obtained by IR spectroscopy over PLM alone (see the microscopy guidelines in Chapter 2). The extent to which IR spectral comparison is indicated will vary with specific sample and case evaluations.

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

The following generic types of fiber are rarely encountered in routine forensic examinations:
Anidex Polycarbonate
Lastrile Vinyon
PBI Fluorocarbon

Vinal Novoloid
Azlon Sulfar
Nytril

Exemplar data, reference standards, examiner experience, or combinations may be inadequate for their characterization by microscopical and microchemical techniques. For these fiber types, IR spectroscopic confirmation of polymer type may be advisable.

Because of the large number of subgeneric classes, forensic examination of acrylic fibers is likely to benefit significantly from IR spectral analysis (13).

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.

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, IR spectroscopy, or pyrolysis gas chromatography. Infrared analysis offers the advantage of being the least destructive of these methods.

6.0. Sample Handling

The general handling and tracking of samples should meet or exceed the requirements of ASTM 1492-94 (14).

The quantity of fiber used and the number of fiber samples required will differ according to the following:
6.1. Specific technique and sample preparation;
6.2. Sample homogeneity;
6.3. Condition of the sample; and
6.4. Other case-dependent analytical conditions, concerns, or both.

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 (15). Leaving the fiber unflattened, while allowing crystallinity-sensitive bands to be observed unaltered, results in distortion of peak heights due to variable pathlengths (16). In certain situations, a combination of both approaches may be advisable.

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.

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, CsI, BaF2, ZnSe, and diamond. The choice of window material should not reduce the effective spectral range of the detector being used. 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 that would arise if the spectrum were acquired of an air gap between the two IR windows.

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, 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.

7.0. Analysis

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

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

7.1. Equipment Readiness

All spectrometer 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. Analysts should refer to the manufacturer's guidelines for the optimum performance of their instruments.

7.2. Instrument Performance and Calibration

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

The preferred performance evaluation method is in accordance with ASTM 1421-94, Sections 1-7, 9.5, and 9.5.1 (17). In brief, this includes evaluating the following:
7.2.1. System throughput;
7.2.2. Single-beam spectrum;
7.2.3. 100% T line; and
7.2.4. Polystyrene reference spectrum.

Dispersive instruments should be checked according to manufacturers' recommendations. Instrument performance records may be maintained on hard copy, 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.

7.3. Sample Illumination and Detector Measurement Apertures

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.

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.

7.4. Objective and Condenser Adjustment

The objective, 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.

7.5. Polarization Bias of the Infrared Spectrometers and Microscopes

Infrared spectrometers 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.

7.6. Focusing

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.

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 (18). 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.

7.7. Background Spectrum

A background spectrum refers to a reference absorption spectrum, which includes the absorbance contributions of all system components except the sample of interest. The IR window or windows with KBr crystal are all considered part of the system. The system parameters for background spectra should be identical to the parameters used for sample spectra (with the possible exception of gain and number of scans). These parameters include resolution, mirror velocity, and spectrometer aperture size.

7.8. Resolution

Resolution should be set at 4 cm^{-1} (one data point every 2 cm^{-1}). Higher resolution may be used. The additional data points, however, 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.

7.9. Sample and Background Scans

Sample and background scans should be run under the same conditions. If necessary, parameters can be subsequently modified and new sample and background spectra acquired.

7.10. Data Storage

It is generally useful to save all data on disk 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 damaged during subsequent processing can then be restored from saved files.

7.11. Identification of Fiber Polymers by IR Spectra

Successful identification of fiber polymers by 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.

8.0. Report Documentation

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 confirm the identification by other methods (i.e., previously performed analytical techniques such as PLM).

The generic class of manufactured textile fibers can be unequivocally identified with the exception of rayon versus lyocell (these generic classes may be differentiated by their optical properties). The subgeneric class of synthetic manufactured fibers may be identified.

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

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Chapter 7 of Forensic Fiber Examination Guidelines

Fabrics and Cordage

1.0. Scope

These guidelines are intended to assist individuals and laboratories that conduct examinations of fabrics and cordage. They are not intended as a detailed methods description or protocol for the analysis and comparison of fabric and cordage evidence but as guidelines to an acceptable procedure to strengthen the usefulness of the analytical method.

2.0. Reference Documents

SWGMAT Quality Assurance Guidelines
SWGMAT Trace Evidence Handling Guidelines

3.0. Terminology

Core: A fiber or fibers running lengthwise through the center of a cordage.

Course: The row of loops or stitches running across a knit fabric, corresponding to the filling in woven fabrics.

Crown: The raised portion of a strand in a twisted cordage.

Knit Fabric: A structure produced by interlooping one or more ends of yarn or comparable material.

Pitch: The number of crowns per inch of the same strand.

Ply: The number of single yarns twisted together to form a plied cord. An individual yarn in a plied yarn or cord.

Selvage: The narrow edge of woven fabric that runs parallel to the warp. It is made with stronger yarns in a tighter construction than the body of the fabric to prevent raveling.

Strand: A single fiber, filament, or monofilament.

Twist (Lay): The direction of twist in yarns is indicated by the capital letters S and Z. Yarn has an S-twist if when it is held vertically the spirals around its central axis slope in the same direction as the middle portion of the letter S, and it has a Z-twist if the spirals slope in the same direction as the middle portion of the letter Z.

Wale: A column of loops lying lengthwise in a knit fabric.

Warp: The set of yarn in all woven fabrics that runs lengthwise and parallel to the selvage. It is interwoven with the filling.

Weft (Filling): In a woven fabric, the yarn running from selvage to selvage at right angles to the warp.

Woven Fabric: Generally used to refer to fabric composed of two sets of yarns, warp and weft (filling), that is formed by weaving, which is the interlacing of these sets of yarns.

4.0. Summary of Guidelines

Because of their general availability, fabric and cordage are often encountered by forensic scientists, who must compare these types of evidence in order to determine if the two pieces could have originated from the same source. Structural details such as design, construction, and composition can provide information that may assist the examiner in reaching a conclusion.

5.0. Significance and Use

The construction, composition, and color of textiles involved in crimes are useful comparison characteristics for forensic examinations. Textiles appear in a variety of weaves, knits, and nonwoven constructions, and a combination of fabric types can occur in any one textile. The range of colors in which textiles are offered in the marketplace is enormous. Therefore, the construction, composition, and color of a textile can aid the examiner in including or excluding a textile for consideration in a forensic examination.

A complete characterization of the fabrics including their construction and other materials used in the completion of a textile (e.g., sewing thread) is a critical component of a comprehensive forensic fabric or cordage examination.

6.0. Sample Handling

Photograph the item prior to conducting any analyses in order to provide documentation of original condition. Document and remove other evidence (e.g., hair, blood, and paint) that may require additional analysis. Document and record descriptions of any physical damage (e.g., worn, cut, broken, and frayed). The following general macroscopic characteristics should be observed and documented:

6.1. Severed ends for possible physical matches;

6.2. Knots, ligatures, or both;

6.3. Dimensions: size, length, diameter, etc.;

6.4. Components: number, type, and twist;

6.5. Color;

6.6. Dyed; and

6.7. Natural.

Do not bring a questioned specimen (e.g., a piece of fabric, yarn, and tuft of fibers) in contact with the known fabric from which it is suspected to have originated until you have performed a preliminary examination of the questioned specimen.

Do not alter the condition of a questioned specimen (e.g., shape, position, layers, or relation of one yarn to another) before a preliminary examination and before receiving a known sample for comparison.

Do not cut a sample to be used for composition testing from ends of yarn or edges of fabric if there is a possibility of physically matching a questioned specimen to a known specimen. Take the known sample away from the existing edge or edges and mark the location as known taken.

Fabric and cordage may be a source of other types of physical evidence (hairs, fibers, blood, etc.). In addition, cuts, tears, knots, and severed ends may be of forensic value. Therefore, fabric and cordage evidence should be examined in a manner that preserves these types of evidence.

All pertinent data collected on questioned and standard samples should be placed into or referenced within the specific case file.

Reference samples should be maintained. These reference samples should be supplied by the primary manufacturer. If not purchased from a primary source (manufacturer), structural components must be verified by a secondary source.

7.0. Analysis

Preliminary examination of fibers composing a fabric or cordage, with any adhering matter, should include its general appearance under a low-power microscope before a sample is mounted on a slide. Any adhesives or other material used in bonding fabrics, carpet backings, and so forth should also be noted.

Physical matches should always be considered if two pieces of fabric or cordage need to be compared. If the ends have been cut or torn, a physical match may be possible. A physical match must be documented by photography. Additionally, describing the condition of corresponding

threads and their relative positions in the damaged area on the questioned and known pieces (so-called longs and shorts) provides a detailed corroborative description.

If a physical match is not possible, comparison of the parameters determined in the checklist will assist the examiner in determining if the two pieces could have originated from the same source.

7.1. Fabric
Fabric examinations are primarily a process of deconstructing the fabric by dissecting its constituent elements. Each of these elements can have a number of sub-elements, all of which must be characterized to complete the examination. These elements include the following:
7.1.1. Construction (woven, knit, nonwoven);
7.1.2. Threads per inch in warp and weft direction;
7.1.3. Staple or continuous fibers in yarns;
7.1.4. Yarn twist;
7.1.5. Number of plies;
7.1.6. Direction of twist of plies;
7.1.7. Number of filaments in each ply;
7.1.8. Composition of yarn;
7.1.9. All fiber types composing the fabric;
7.1.10. Colors and design;
7.1.11. Blend of two or more types of fibers within each ply; and
7.1.12. Sewing threads, buttons, decorations, and so forth as detailed previously.

The information contained on tags in textiles should also be recorded, especially the registered number (RN) and the woolen products label (WPL) number. These refer to the manufacturer of the textile and can assist the examiner with tracking a particular textile or garment (4, 5).
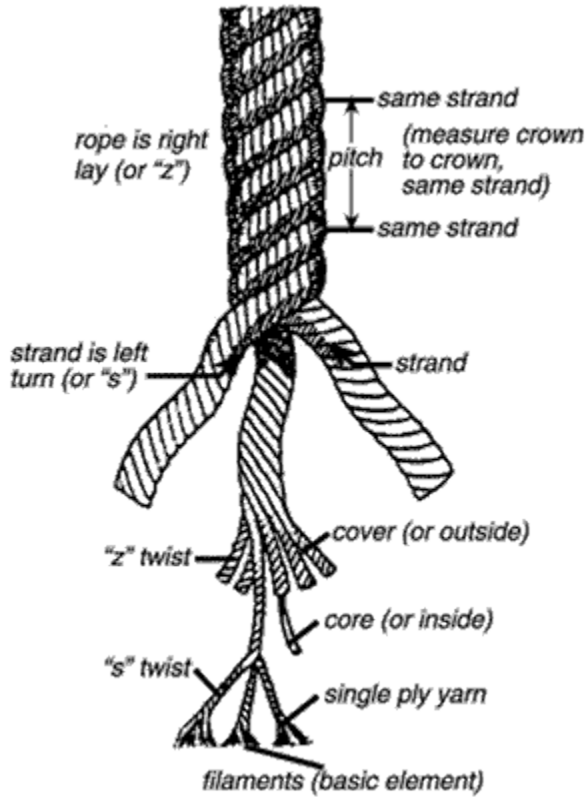
7.2. Cordage
The initial step in the identification of rope and cordage is to determine its construction and assembly. Wiggins (2) recommends that a laboratory develop a checklist for this purpose. The checklist should include, but is not limited to, the following characteristics (see Figure 2):
7.2.1. Diameter;
7.2.2. Staple or filament fibers;
7.2.3. Twisted, braided, or nontwisted;
7.2.4. Twist;
7.2.5. Crowns or turns per inch;
7.2.6. Number of plies or braids;
7.2.7. Twist of each ply or braid;
7.2.8. Crowns or turns per inch; and
7.2.9. Filaments in each ply or braid, which are evaluated for the following characteristics:
7.2.9.1 Core, if any;
7.2.9.2. Twist;
7.2.9.3. Crowns or turns per inch;
7.2.9.4. Number of filaments;
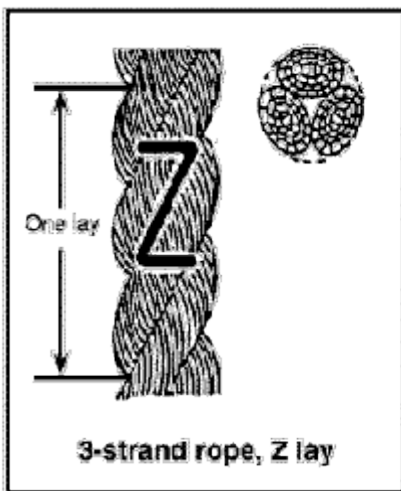7.2.9.5. Color or colors;
7.2.9.6. Coatings, if any;

7.2.9.7. Tracers, if any; and
7.2.9.8. Coatings.

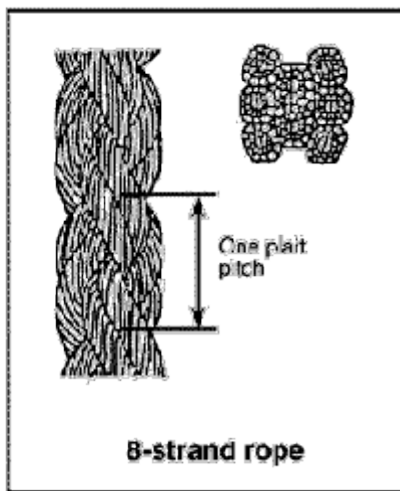## Figure 2: Fiber Rope Components and Constructions

Three-strand rope components

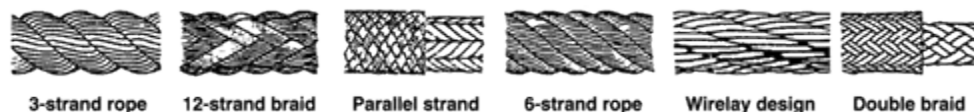3-strand rope, Z lay

8-strand rope

3-strand rope   12-strand braid   Parallel strand   6-strand rope   Wirelay design   Double braid

Note: From *The Cordage Directory*, published by The Cordage Institute, Hingham, Massachusetts, 1998.

After the construction has been established, then the constituent fibers should be analyzed with the appropriate microscopic and instrumental techniques (q.v.). Additional characteristics may be used if necessary to adequately describe the cordage (3).

8.0. Report Documentation

Physical matches should be reported so they indicate if the two or more pieces of material were at one time a continuous piece of fabric or cordage. If no physical match is possible, a complete fiber comparison, including construction, must be performed. If the items are the same in all tested characteristics, then the examiner would report that the two objects exhibit the same color, construction, and composition and are consistent with originating from the same source.

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