

Technical Procedure for the Examination of Fibers

- 1.0 Purpose** – This technical procedure shall be followed for the examination of individual fibers.
- 2.0 Scope** – This procedure applies to the analysis of single fiber samples within the Trace Evidence Section.
- 3.0 Definitions** – N/A
- 4.0 Equipment, Materials, and Reagents**

4.1 Equipment

- Stereomicroscope
- Polarized light microscope (PLM)
- Comparison microscope
- FT-IR with microscope attachment
- Microspectrophotometer
- HPLC
- Pyrolysis-GC-MS
- Muffle furnace
- Microtome
- UV light
- Hotplate

4.2 Materials

- Forceps
- Probes
- Scalpel
- Razor blades
- Glass slides and cover slips
- Quartz slides and cover slips
- Joliff Cards
- Wire loop
- Filler fibers
- Capsule mold
- Polyethylene sheets
- Micropipette and tips
- Roller
- KBr discs and holder
- Small crucible with lid

4.3 Reagents

- Nail polish, evaporated to approximately 50 % concentration
- Xylene Substitute
- Glycerol (low fluorescing)
- Permout

- Epoxy
- Norland Optical Adhesive
- Acetonitrile
- Chloroform

5.0 Procedure

5.1 Analytical Approach

5.1.1 General guidelines:

5.1.1.1 The Forensic Scientist shall approach a fiber comparison by attempting to show that the samples are different. The failure to detect any significant differences, after exhausting the methodology available to the Forensic Scientist, results in the conclusion that the known and questioned fibers could have had a common origin.

5.1.1.2 The questioned and known fibers are analyzed using the same techniques and are compared at every step throughout the process. If a difference is found, the analysis may be concluded at any step.

5.1.1.2.1 The order of the examination is based on the quantity, quality, type of the evidence and the Forensic Scientist's training and experience.

5.1.1.2.2 Some of the tests available to fiber-trained Forensic Scientists are destructive. When sample size is limited, destructive testing, if necessary, shall only be performed after all non-destructive testing is complete.

5.1.1.3 All results shall be based on the Forensic Scientist's knowledge and experience and the case being examined. Results shall be in agreement with the technical reviewer.

5.1.2 Review the request for analysis.

5.1.3 Open evidence container and describe the evidence present.

5.1.4 Collect standards of the known. If examining the item for fiber transfer, process the item to remove any adhering fiber evidence following the Trace Evidence Section [Technical Procedure for the Collection and Preservation of Evidence](#).

5.1.5 Perform a preliminary evaluation of the known standard.

5.1.5.1 Record the overall characteristics of the known fibers using the stereomicroscope.

5.1.5.2 Mount the fibers as described in 5.2.

- ### 5.3 Natural Fiber Analysis

5.3.1 Vegetable-Based Fibers

- 5.3.1.1** Vegetable-based fibers are generally found in bundles. For microscopic analysis, the fibers must be broken down to the ultimates. Some may be pulled apart manually, while others may require chemical maceration.
- 5.3.1.2** Examine fibers both longitudinally and in cross-section.
- 5.3.1.3** Examine the internal and external characteristics using the PLM. These characteristics include, but are not limited to, the following:
- Surface texture: smoothness, pitting, scales, dislocations, cross-hatching, etc.
 - Plant interstructure: lumen, holes, voids, crystals, spiral elements, etc.
- 5.3.1.4** Using the fluorescence capabilities of the comparison microscope, observe the color and intensity of a fiber's fluorescence.
- 5.3.1.4.1** The two different fluorescence cubes used are A (wide UV, 340-380 nm) and H3 (wide band blue, 420-490 nm).
- 5.3.1.4.2** Non-fluorescing mounting media shall be used.
- 5.3.1.5** Perform either the Dry Twist Test or the Herzog test on the fiber ultimates. These tests are not required for cotton.
- 5.3.1.6** If the sample size permits, ash the fibers in the muffle furnace. Heat at approximately 600 °C for 3-4 hours or until completely ashed.

5.3.2 Animal-Based Fibers

- 5.3.2.1** Animal-based fibers are essentially animal hair. However, in the course of a fiber examination, only the most common animal hair types used in textile production shall be examined. If the hair in question is not one of the common textile fibers, analysis shall follow the Trace Evidence Section [Technical Procedure for Hair Analysis](#).
- 5.3.2.2** Hair-based features shall be examined under the light microscope. This includes, but is not limited to, the following:
- Surface features (scales)
 - Cortex features (pigment, ovoid bodies, etc.)
 - Medullary features (size and pattern)
 - Root structure

- 5.3.3** Based on the microscopic characteristics and physical tests, an identification of the fiber type may be made.

5.4 Man-Made Fiber Analysis

- 5.4.1 Using a light microscope, examine the general fiber properties to include size, optical cross-section, surface markings and striations, inclusions (including delustrant) and dyes or colorants.
- 5.4.2 Using a PLM, examine the optical properties of the fibers. These include, but are not limited to, sign of elongation, extinction points, birefringence, and pleochroism.
- 5.4.3 If the fibers are consistent in the longitudinal orientation, they shall be cross-sectioned.
- 5.4.4 Using the fluorescence capabilities of the comparison microscope, observe the color and intensity of a fiber's fluorescence.
 - 5.4.4.1 The two different fluorescence cubes used are A (wide UV, 340-380 nm) and H3 (wide band blue, 420-490 nm).
- 5.4.5 Based on the microscopic characteristics, a tentative identification of the fiber type may be made. However, instrumental analysis shall be used to confirm all man-made fiber identifications.

5.5 Solubility Testing

- 5.5.1 Acetonitrile at room temperature (approximately 75 °F)
 - Acetate – Soluble
 - Triacetate – Insoluble
- 5.5.2 Butyrolactone at room temperature (approximately 75 °F)
 - Acrylic – Insoluble
 - Modacrylic – Soluble
- 5.5.3 Additional solubility testing may be conducted as necessary. See Attachment “A General Table of Solubility for the Qualitative Analysis of Textile Fibers” for suggested reagents.

5.6 Thin Layer Chromatography (TLC)

- 5.6.1 If sufficient sample is present, TLC may be used for dye separation.
- 5.6.2 A minimum of one solvent system is required.
- 5.6.3 The known and questioned samples shall be run on the same plate at the same time.
- 5.6.4 To document the results, photocopy, scan or photograph the TLC plate and calculate the R_f values of the spots.
- 5.6.5 Suggested extraction and eluent systems can be found in the attachment “Suggested Extraction and Eluent Systems for Thin Layer Chromatography.”

5.7 Guidelines for Fiber Analysis Result Statements

5.7.1 The reports shall read as follows. The wording of the results shall accurately describe the evidence at hand.

5.7.2 Positive

5.7.2.1 This statement shall be used when the questioned and known samples are consistent in color and composition.

5.7.2.1.1 Example: Example: Item A was found to be consistent with Item B. Therefore, Item A could have originated from [the same source as] Item B.

5.7.2.2 No comparison performed, only identification of an item.

5.7.2.2.1 Example: Item A was identified as _____.

5.7.2.3 Qualifying statements shall be added to the report as needed, especially regarding the commonality of certain types of fibers. This includes, but is not limited to, the following:

- White, undyed cotton fibers
- Blue and white variegated cotton fibers (denim)
- White, undyed wool fibers

5.7.3 Inconclusive

5.7.3.1 These statements shall be used when, based on the acquired data, no conclusion could be reached.

5.7.3.1.1 Example: Item A was found to be consistent in __ to Item B; however, slight differences were noted in __. Therefore no conclusion could be reached as to whether or not Item A could have originated from [the same source as] Item B.

5.7.3.1.2 Example: Due to the size/condition of Item A, no conclusion could be reached as to whether or not Item A could have originated from [the same source as] Item B.

5.7.4 Negative

5.7.4.1 This statement shall be used when one or more of the characteristics associated with the questioned and known fibers are different.

5.7.4.1.1 Example: Item A is not consistent with Item B. Therefore, Item A could not have originated from [the same source as] Item B.

5.7.4.2 No fiber associations between items.

5.7.4.2.1 Example: No fiber associations were noted between Item A and Item B.

5.7.5 No Analysis

5.7.5.1 No analysis is performed.

5.7.5.1.1 Example: The above listed evidence is being returned unanalyzed. If you have any questions, please contact the Forensic Scientist who issued this report.

5.7.5.2 No analysis is performed due to the results of the DNA analysis.

5.7.5.2.1 Example: Based on the results of DNA analysis, the above listed evidence is being returned unworked. If you have any questions, please contact the Forensic Scientist who issued this report.

5.7.5.3 No analysis is performed due to the size/condition of the sample.

5.7.5.3.1 Due to the size or condition of the fiber sample, no analysis could be conducted.

5.8 Standards and Controls – N/A

5.9 Calibrations – This procedure does not require any calibrations or performance checks. However, it does utilize instruments that require performance checks. See the individual technical procedures for the operations of those instruments.

5.10 Maintenance – No maintenance is required in this procedure. However, the procedure does utilize instruments that require maintenance. See the individual technical procedures for the operations of those instruments.

5.11 Sampling and Sample Selection

5.11.1 No sampling is performed. When sample selection occurs, it shall be based on the Forensic Scientist's training and experience.

5.11.2 If at any point during the course of examination the items are found to be inconsistent with one another, analysis may be halted and a lab report shall be issued stating a negative finding.

5.11.3 If no fiber standards are submitted, the evidence may be returned to the agency unworked.

5.11.4 If DNA analysis is being performed on the evidence in the case, based on the results of the DNA analysis, the fiber evidence may be returned unworked.

5.12 Calculations – N/A

5.13 Uncertainty of Measurement – N/A

6.0 Limitations - Fibers are a manufactured material. It shall not be possible to identify a fiber as having come from a particular source to the exclusion of all others.

7.0 Safety - Crucibles removed from the muffle furnace are very hot and may cause burns. Care shall be exercised when using these items.

8.0 References

8.1 ASTM / SWG Guidelines

ASTM Standard D276, 2000a, "Standard Test Methods for Identification of Fibers in Textiles." ASTM International, West Conshohocken, PA, 2000.

ASTM Standard E175, 1982 (2005e1), "Standard Terminology of Microscopy." ASTM International, West Conshohocken, PA, 2005.

ASTM Standard E227, 2002, "Standard Guide for Forensic Examination of Non-Reactive Dyes in Textile Fibers by Thin-Layer Chromatography." ASTM International, West Conshohocken, PA, 2002.

ASTM Standard E2224, 2002, "Standard Guide for Forensic Analysis of Fibers by Infrared Spectroscopy." ASTM International, West Conshohocken, PA, 2002.

ASTM Standard E2228, 2002, "Standard Guide for Microscopic Examination of Textile Fibers." ASTM International, West Conshohocken, PA, 2002.

SWGMA. Forensic Fiber Examination Guidelines. For. Sci. Comm. 1999: 1(1).

8.2 Books

Billmeyer, F.W. and M. Saltsman. *Principles of Color Technology*. 2nd Ed. New York: John Wiley and Sons, 1981.

Caitling, D. and J. Grayson. *Identification of Vegetable Fibres*. New York: Chapman and Hall, 1982.

Hall, D.M. *Practical Fiber Identification*. 2nd Ed. Auburn, AL: Auburn University, 1982.

Hatch, K.L. *Textile Science*. New York: West Publishing Company, 1993.

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Saferstein, R. *Forensic Science Handbook Volume II*. Englewood Cliffs, NJ: Prentice Hall, 1988.

Saferstein, R. *Forensic Science Handbook Volume III*. Englewood Cliffs, NJ: Prentice Hall, 1993.

The Textile Institute. *Identification of Textile Materials*. 7th Ed. Portsmouth: Eyre & Spottiswoode Limited, 1975.

8.3 Journal Articles

Deedrick, Douglas W. and Sandra L. Koch. "Microscopy of Hair Part II: A Practical Guide and Manual for Animal Hairs", *Forensic Science Communications* 6.3 (July 2004).

Grieve, M.C. and L.R. Cabiness. "The Recognition and Identification of Modified Acrylic Fibers." *Forensic Science International* 29 (1985): 129-146.

Palenik, S. and Fitzsimons. "Forensic Microscopy, Fiber Cross-Sections: Part II." *Microscope* 38 (1990): 313-320.

Valaskovic, G.A. "Polarized light in multiple birefringent domains: a study of the Herzog effect." *The Microscope* 39 (1991): 269-286.

8.4 Training Materials

Introduction to Hairs and Fibers, Training Materials, FBI.

Microscopy of Wood and Vegetable Fibers (Training Materials), McCrone Research Institute, John D. Shane, Instructor, March 2000.

9.0 Records – N/A

10.0 Attachments

- A General Table of Solubility for the Qualitative Analysis of Textile Fibers
- Suggested Extraction and Eluent Systems for Thin Layer Chromatography

Revision History		
Effective Date	Version Number	Reason
09/17/2012	1	Original ISO Document

A General Table of Solubility for the Qualitative Analysis of Textile Fibers

Table of Solubilities

Chemical Agent	Conc. (% by weight)	Density (at 75°F)	Temp (°F)	Secondary (Acete)													
				Acetate	Acrylic	Modacrylic	Nylon	Nylon	Nylon	Qlefin	Polyester	Rayon	Saran	Spandex	Cotton & Flax	Silk	Wool
Acetic Acid, glacial			75	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Acetic Acid, glacial			200	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Acetic Acid, glacial			244*	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Acetone			75	S	P	P	S	S	S	S	S	S	S	S	S	S	S
Acetone			133*	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Acetone	65		75	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Acetonitrile			180*	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Acetonitrile			200	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Ammonium Thiocyanate	70		266*	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Ammonium Thiocyanate			200	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Benzyl Alcohol			75	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Butyrolactone			200	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Carbon tetrachloride			170*	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Chloroform			75	S	S	S	S	S	S	S	S	S	S	S	S	S	S
m-Cresol			75	S	S	S	S	S	S	S	S	S	S	S	S	S	S
m-Cresol			200	S	S	S	S	S	S	S	S	S	S	S	S	S	S
m-Cresol			396*	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Cyclohexanone			75	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Cyclohexanone			200	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Cyclohexanone			313*	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Dimethyl Acetamide			75	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Dimethyl Formamide			75	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Dimethyl Formamide			200	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Dimethyl Formamide			307*	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Dimethyl Sulfoxide			75	S	S	S	S	S	S	S	S	S	S	S	S	S	S
1,4-dioxane			75	S	S	S	S	S	S	S	S	S	S	S	S	S	S
1,4-dioxane			200	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Ethylene Carbonate			85	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Formic Acid			37-38	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Hydrochloric Acid			20	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Hydrochloric Acid			75	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Hydrochloric Acid			15.5	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Monochlorobenzene			269*	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Nitric Acid			70	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Nitric Acid			75	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Nitric Acid			1.407	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Nitric Acid			1.362	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Pyridine			200	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Sodium Hydroxide			40	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Sodium Hydroxide			at boil	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Sodium Hypochlorite- solution (pH11)			(3.6 or 5.0% avail. Chlorine)	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Sulfuric Acid			75	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Sulfuric Acid			75	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Sulfuric Acid			69	S	S	S	S	S	S	S	S	S	S	S	S	S	S
Sulfuric Acid			1.3	S	S	S	S	S	S	S	S	S	S	S	S	S	S
m-Xylene			200	S	S	S	S	S	S	S	S	S	S	S	S	S	S
m-Xylene			282*	S	S	S	S	S	S	S	S	S	S	S	S	S	S

* ~ = water used as diluent if less than 100%
* at the boil

^ Zefran insoluble
Courrelle and Dralon, partially soluble

I = Fiber insoluble
S = Fiber soluble or completely disintegrated
P = Fiber partly soluble or partly disintegrated

Adapted from:
Hall, D. Practical Fiber Identification
Auburn: Auburn University, 1962, p. 36.

Suggested Extraction and Eluent Systems for Thin Layer Chromatography

Suggested Extraction Systems:

Dye classification indicates the best extraction solution for a particular fiber. Useful published information is summarized below:

Dye Class	Fiber Type	Extraction Solution
Acid ¹	Polypropylene	Pyridine / Water (4:3)
Acid	Nylon	Pyridine / Water (4:3)
Acid	Wool	Pyridine / Water (4:3)
Acid	Cotton	Glacial Acetic Acid
Basic	Polyester	Pyridine / Water (4:3)
Direct	Cotton	Pyridine / Water (4:3)
Disperse	Polyester	Pyridine / Water (4:3)
Disperse	Polypropylene	Pyridine / Water (4:3)
Disperse	Acetate / Triacetate	Pyridine / Water (4:3)
Metalized	Wool	2% Aqueous Oxalic Acid then pyridine / water (4:3)
Metalized	Polypropylene	Pyridine / Water (4:3)
Basic	Acrylic	Formic Acid / Water (1:1)
Reactive, Sulfur, Vat, Diazo, Ingrain and pigmented dyes do not extract		

¹ Dyes rarely extract from Polypropylene, but if they do, they are most likely to be Acidic.

Choice of Eluent System:

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 ²	Proportions (v/v)
1	n-Butanol, acetone, water, ammonia	5:5:1:2
2	Pyridine, amyl alcohol, 10 % ammonia	4:3:3
3	n-Butanol, ethanol, ammonia, pyridine, water	8:3:4:4:3
4	Methanol, amyl alcohol, water	5:5:2
5	Toluene, pyridine	4:1
6	Chloroform, ethyl acetate, ethanol	7:2:1
7	n-Hexane, ethyl acetate, acetone	5:4:1
8	Toluene, methanol, acetone	20:2:1
9 ³	n-Butanol, acetic acid, water	2:1:5
10	n-Butanol, ethanol, ammonia, pyridine	4:1:3:2
11	Chloroform, butanone, acetic acid, formic acid	8:6:1:1
12 ³	n-Butanol, acetic acid, water	4:1:5

² The ethanol used is 99 %; the ammonia 0.880 SG unless otherwise stated.

³ These eluents form an upper and lower phase. Use the upper phase as the eluent.

Eluents Recommended for Certain Dye Classes:

Certain fiber type/dye class combinations have been found to give better separation in certain eluents. These are shown in the table below and are recommended as a first choice.

Fiber Type	Dye Class	Eluent Number
Wool	Acid or Metallized	1,2
Cotton and Viscose	Direct	1,4,3
Acrylic	Basic	11,12,1
Polyester	Disperse	6,7,8,5
Polyamide	Acid	9,10
Polypropylene	Polypropylene rarely contains an extractable dye. If the dye can be extracted an eluent appropriate to the dye class is used.	

Adapted from:

ASTM Standard E227, 2002, "Standard Guide for Forensic Examination of Non-Reactive Dyes in Textile Fibers by Thin-Layer Chromatography, ASTM International, West Conshohocken, PA, 2002.