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Analytical Methods for Developing Fibers as Forensic Science Proof: A Review with Comments

REFERENCE: Fong, W., **"Analytical Methods for Developing Fibers as Forensic Science Proof: A Review with Comments,"** *Journal of Forensic Sciences*, JFSCA, Vol. 34, No. 2, March 1989, pp. 295-311.

ABSTRACT: A review and commentary of the principal methods for developing textile fibers as evidence are given. The methods reviewed proceed from the preliminary phase of finding and handling to the subsequent phases of identification, characterization, and comparison. The latter phase is arranged to progress from methods that require only microscopic amounts of fibers and are nondestructive to those that require progressively larger amounts of fibers and are destructive. The relevant literature in the time period 1950 to 1987 is included.

KEYWORDS: criminalistics, fibers, physical evidence

This paper is a review with commentary of the approaches which have been applied toward fiber examinations as they are described in the forensic science and relevant scientific literature. Articles found in the principal forensic science journals printed in English up to January of 1987 form the basis of the review.

The topics selected for review are arranged according to the following format:

Processing:
- recovery
- searching and segregating
- mounting

Identification, characterization, and comparison:
- microscopy
- microspectrophotometry and solution spectrophotometry
- refractive index and birefringence
- density
- melting point and solubilities
- cross sections
- reactivity to test reagents
- thin-layer chromatography
- infrared spectrophotometry
- pyrolysis gas chromatography

Within the scope of each topic discussed, an attempt has been made to present the material

Received for publication 14 Sept. 1987; revised manuscripts received 15 Jan. and 16 May 1988; accepted for publication 17 May 1988.

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in a chronological sequence, although this was not always possible. It was also inevitable that there would be some overlapping of topics.

Processing

Recovery

Methods for searching evidence items for fibers were given in 1953 by Kirk [*1*]. These included instructions for collection by the passage of the application nozzle of a vacuum collecting device over the surface of the item of interest. The resultant vacuumings presented the laboratory analyst with a formidable task for efficient and rapid search since they consisted of dust-laden fluff, quite often in large amounts.

The simple alternative of press application of the adhesive side of cellophane tape to lift loose fibers was referred to in 1965 by Frei-Sulzer [*2*]. However, his resorting to tape lifts was occasioned more by the inability to derive the zones of contact when a vacuum filter was used and less by the difficulties inherent in searching through large amounts of fluff. Later, in the same year, Martin [*3*] discussed the considerations relating to the usage of adhesive tape lifts.

The disadvantage of strip lengths of single-faced adhesive tapes lies in their lack of systematic efficiency and rapidity when they are applied to large surface areas such as those found on clothing, bedding, and fabric-covered furniture. This problem was addressed in 1984 by Fong [*4*], who described a simple device fashioned by spiralling double-faced adhesive tape onto a paint roller of small size, over which there had been drawn a tight fitting polyethylene bag. In his discussion, Fong advises that readily apparent fibers be recovered by strips of single-faced adhesive tape and that then the roller device be passed over the surfaces of the article of interest. Overloading of the adhesive surface in contact is readily detected by the feeling of loss of adhesion, and, if required, a replacement adhesive sleeve can be quickly prepared for additional passes.

Fong is relatively unconcerned about localizing the site of recoveries to permit deriving the source of fibers. This is evident in his answer to commentary on this subject offered by Grieve [*5*], who cites in support of his comment the work of Frei-Sulzer. Fong's answer is rooted in the logic that the location of a fiber is not necessarily the location of original deposition. Thus its usage as a foundation for the reconstruction of the events surrounding the offense is problematic and should be avoided. That there is the distinct probability of redistribution of fiber location is confirmed by results of a study performed by Robertson and Lloyd [*6*]. However, the particular circumstances of each case can modify the procedures to be applied. Thus, the matter of whether to localize or not to localize is a matter best left to individual judgment based upon circumstances.

Results of a study of the effectiveness of adhesive tape recoveries when compared with other methods were reported in 1975 by Pounds [*7*]. Referring to the results of his previous work, the author points out that searches for fibers were unsuccessful in 60% of cases and that success was dependent upon the time since deposition. Using a fluorescent tag technique, Pounds studied the relative effectiveness of vacuuming, shaking, lifting with adhesive tape, and brushing with a nylon fabric brush. He reported poor results with both the vacuuming and shaking, but high success for larger fibers with both the nylon brush and a "low-adhesive" lifting tape (similar to that used in lifting developed latent fingerprints). A "high-adhesive" tape was also very effective, but its disadvantages were in the recovery of large numbers of background fibers and the difficulties experienced in the removal of the fine fibers from the adhesive without breakage.

Pounds gives recommendations on procedures that include the application of a judgment by the worker as to how many fibers are likely to have been transferred; if the number estimated is few, the "high-adhesive" tape is advocated, but if the number estimated is large,

the "low" should be used. Also, usage of the nylon brush followed by application of adhesive tape pressings to the brush was found to be effective.

No loss attributable to packaging of evidence articles in paper or plastic bags was encountered. Such a loss, attributable to electrostatic attraction, was suggested by Frei-Sulzer.

A noteworthy method for collecting fibers from a person's head hair was described in 1975 by McKenna and Sherwin [*8*]. It consists of the passing of a comb loaded with cotton fibers through the subject's hair.

Searching and Segregating

The task of searching tape lifts has received scant notice in the literature. Grieve and Garger [*9*] described a grid template used in the search of the tapes under a stereoscopic microscope. As part of a comprehensive scheme for examining fiber evidence, Fong [*4*] performs the task by overlapping low-power microscopic fields. Also, when fibers resist removal, the tips of the jeweler's forceps used are wetted with a small amount of the fluid used to mount the fibers. The fluid loosens the fiber from the adhesive. Additional steps involves transferring the fibers to a slide prewetted with the mounting fluid, segregating them to separate locations on the slide based upon obvious differences, applying a coverslip, sealing the coverslip to the slide by use of a molten edge sealant, applying numerical markings to the top of the coverslip with a permanent marker to aid in the location of the segregated fibers, and labeling the slide appropriately.

Mounting

In 1982, Cook and Norton [*10*] reported the results of a study to find a mounting media for fibers which would have the most of a series of desirable attributes. Twelve mountants were studied for their effect on fibers dyed with dyes known to be labile. The mountants selected were outside the range of 1.5 to 1.6 in refractive index, the range into which most fibers fall to achieve desired contrast. Slide mounts of fibers, half of which were stored in the dark and the other half in light, were studied at periodic intervals up to one year. Mountants causing leaching and dye fading were found. The mountant which best met the criteria was XAM neutral medium improved white. This mountant is readily available in the United Kingdom, but much less so in the United States. It is unfortunate that some of the media which have found a place other than in the United Kingdom (for example, Aroclor 5221, as recommended by McCrone [*11*]) were not included in this study.

Identification, Characterization, and Comparison

Some textbooks addressed principally to fibers and their identification are by Krauss [*12*], Heyn [*13*], and Stoves [*14*]. They cover the traditional microscopic methods, stains, solubilities, and cross sections. A more recent textbook is that of the Textile Institute [*15*]. Identification procedures have also been given in forensic science textbooks such as Kirk [*1*] and Frei-Sulzer [*2*]. The latter is limited to natural fibers. A manual issued in 1978 by the Metropolitan Police Forensic Science Laboratory [*16*] covers a wide range of instructions on the subject.

Microscopy

Morphological characteristics observed through normal optical microscopy can identify most textile fibers of natural origin. Animal fibers have scales, wood fibers are pitted, cotton has ribbon-like twists, silk fibers have crossover marks, and bast fibers have nodes. Further differentiation within these classes is more difficult; an example is the differentiation of sin-

gle fibers of flax and hemp. Man-made fibers, on the other hand, are not so easily characterized, having, with few exceptions (for example, recent texturized fibers), a uniform cross-sectional shape and diameter throughout their length. Their rapid and reliable identification has occupied the attention of many contributors to technical journals.

In 1952, Heyn [*17*] gave details for determining the birefringence of synthetic fibers as a method for their identification. This was followed in 1958 by an article by Longhetti and Roche [*18*] which adapted the methods of Heyn to the special needs of criminalistics. Their work included supplementary information on solubility tests for very short fibers. Culliford [*19*] followed with additional contribution in 1963.

Polarized light microscopy offers several advantages which meet needs peculiar to the forensic science worker: nondestructive, rapid, applicable to small samples, and based upon well-accepted scientific principles. Its disadvantage is in the requirement that the refractive index of the liquids used must be changed to effect the measurement of $n_{\parallel}$ and $n_{\perp}$. This can be inconvenient and time-consuming, especially in a slide preparation consisting of a mixture of several synthetic fibers.

In 1982, Fong [*20*] described a method to overcome this problem through use of a dispersion-staining objective as an adjunct to the polarizing microscope. Through its use all of the principal synthetic textile fibers can be identified by generic class in a single-liquid mount. The method involves mounting the fibers in a high-dispersion liquid having a refractive index approximately central to the known refractive indices of synthetic fibers, both $n_{\parallel}$ and $n_{\perp}$, and observing dispersion-staining color effects to estimate the two refractive indices. In subsequent contributions, Fong [*4,21*] gives additional characterizations for fibers not included in his original work and observations on the lack of permanency of the Cargille refractive index liquid used. He recommends the removal of matching fibers to resin mounts such as XAM neutral improved medium white.

Fong's single-liquid mount method was included as part of a comprehensive scheme for processing fiber evidence described in a paper published in 1984 [*4*]. The scheme has especial value in complex cases involving many possible sources of fibers.

Publication of the scheme aroused critical commentary by Grieve [*22*]. One of the criticisms involved the comparison of fibers without the convenience of a comparison microscope, which Grieve characterized as a "dangerous practice." Fong responded that if one's mental image retention ability is poor, there is no recourse except a comparison microscope [*23*]. The flames of this small-scale controversy were reignited shortly afterwards by Rowe [*24*].

Rowe's commentary was based on the work of Newhall et al. [*25*] and was written in support of Grieve's position. Newhall's work was done to determine the ability of subjects to match their memories of colors to a set of Munsell color standards. Newhall found "systematic errors in color matches obtained." In response, Fong pointed out that the reference cited by Rowe was inappropriately applied to fibers comparisons such as those experienced in criminal investigations. He conceded that difficult problem situations can arise, for which he offered a solution arrived at independently by some practitioners of comparative microscopy: mount the fibers on a single slide so that they can be seen in the same microscopic field.

Interference microscopy as an approach to forensic fiber identification was described in 1982 by Heuse and Adolf [*26*]. The approach, "profitably comprises polarization microscopy and the Becke line method and in particular enables the worker to obtain detailed information specific to the material under examination."

The method requires separate immersions in Cargille refractive liquids to determine the refractive indices $n_{\parallel}$ and $n_{\perp}$. One immersion can suffice when the birefringence is low and when accuracy is relatively unimportant. An advantage over conventional methods, which rely upon observation of the Becke line for determining refractive index, is that "relative phase difference" is used to arrive at a refractive index value through calculation. This phase

difference is readily found through measurement of the degree of displacement of the interference fringe when the fiber and immersion liquid are near to each other in refractive index.

Heuse and Adolf enter values for $n_{\parallel}$ and $n_{\perp}$ into a coordinate system in which $n_{\perp}$ is shown as the abscissa and $n_{\parallel}$ as the ordinate. These values identify the optical "standort" (locus) of a fiber. Entry of values for the commercially important fibers into the coordinate system gives rise to a "standort diagram" which is extremely useful for graphically showing the relative relationship of the different fibers based upon their optical properties. When plotted in detail, a standort diagram will show that certain fibers form "districts." This is a reflection that fibers can vary depending on deliberate structural modifications introduced at the time of manufacture. An outstanding example of a generic class of fibers having several districts, some occupying relatively large areas, is the polyester fibers.

Additional attributes of interference microscopy relate to the ability to make a qualitative estimation of the cross section of a fiber through interpretation of fringe pattern shapes and the detection of structural inhomogeneities through observation of departures from normal interference fringes which could constitute a batch-specific characteristic.

Double-beam interference microscopes, such as the Mach-Zehnder manufactured by the Leitz Company as used by Heuse and Adolf, are rarities in the United States. They are extremely expensive, which obviates the likelihood of their becoming common for forensic fibers identification, at least in the near future.

In a comparison and evaluation of methods for identifying synthetic fibers, Rouen and Reeve [*23*] considered polarizing microscopy, infrared spectroscopy, and pyrolysis-gas chromatography. Their work led to the conclusion that microscopic methods were the "most specific," while infrared spectroscopy and pyrolysis-gas chromatography methods had "less specificity," but were faster. Birefringence determinations were done by systematic changing of the refractive index of the immersion liquid; this is the apparent reason for the reported slowness of the microscopic method.

Infrared analysis was found to be unsatisfactory for the differentiation of natural origin synthetic fibers, particularly viscose products; however, this method was reported to be specific for synthetics.

Pyrolysis analysis was found to be only partially satisfactory for natural origin synthetic fibers and for polyacrylonitrile and polyester synthetic fibers.

To show the application of birefringence towards differentiating synthetic fiber falling within the same generic class, Johri and Jatar [*27*] described their studies of seven terene (polyester) fibers produced by four different manufacturers in India. They performed their birefringence determinations using a Ehringhaus compensator. Birefringence averages were found to differ significantly from sample to sample, but from the same sample were "quite constant."

An innovative polarizing microscopic approach towards textile fibers identification was proposed by Petraco et al. [*28*] in 1980. These authors, citing the work of Grabar and Haessly [*29*] as a basis, monitored the loss of order in a fiber as reflected by changes in its birefringence during heating. Birefringence was calculated based upon retardation, estimated from a Michael Levy interference color chart, and measured fiber thickness. Controlled heating of the fiber sample was accomplished with a Mettler hot stage, Model FP52, supported by a Mettler FP5 control unit. The method lends itself well to direct comparison of questioned and reference fibers by mounting them parallel to each other for simultaneous viewing on the same microscope slide. Also, graphical representations for comparative purposes—especially useful for showing differences in fibers from the same generic class—can be prepared from the data by using four-cycle semi-logarithmic graph paper. Their results show the potential for differentiating fibers belonging to the same generic class, for example, the polyesters and polyacrylonitriles. Graphical representations of the polyamides show the inability to differentiate Nylon 6 and Nylon 6.6, but these are differentiated from Antron®.

Aramid, Kevlar®, and Nomex® have very high melting points, and with the heating range of 25 to 300°C, no birefringence changes were observed. Also, the method is unsuitable for the examination of rayon, which fiber chars at temperatures approaching 300°C. As a method for differentiations within generic class, this method has much to recommend it. It has the disadvantage of being destructive, but this is a relatively small disadvantage since the sample length of fiber required is microscopic, 0.5 mm or less.

Microspectrophotometry and Solution Spectrophotometry

Comparison of colors through the characterization of dyestuffs by solution spectrophotometry and thin-layer chromatography of extracted dyes has found recent favor. However, these methods are destructive and therefore undesirable to those who are reluctant to sacrifice the required portions.

Microscopic comparisons of two similarly colored fibers consisting of colors produced by combinations of different dyes may fail to show a difference. Microspectrophotometry as a method for performing spectrophotometric determinations to detect the presence of a dye mixture yielding a metameric color can show this difference and has gained attention as a method for fiber comparison. It is nondestructive and well suited to the examination of single fibers of lengths 0.5 mm or less.

In 1979, Macrae et al. [*30*] compared the discriminating power of comparison microscopy (both white light and ultraviolet [UV] fluorescence), microspectrophotometry (MSP), solution spectrophotometry (SSP), and thin-layer chromatography (TLC) through the examination of a collection of visually similar dyed wool samples.

They show that each of the analytical techniques offers a definite improvement in ability to differentiate similarly colored wool fibers when compared with comparison microscopy. Since the place of comparison microscopy is well established, the authors directed their discussions towards the relative merits of the competing analytical techniques.

MSP was characterized as the natural partner to comparison microscopy. Both are nondestructive and applicable to single fibers, and MSP can be applied after microscopy without additional manipulation. Problems were experienced with MSP when the fiber sample was deeply dyed or heavily delustered or both. Results of MSP and SSP are not redundant, as expected: MSP was found to discriminate pairs of fibers not differentiated by TLC; the reverse was also the case.

If sufficient fiber is available and if the dyes can be efficiently extracted, SSP and TLC both offer excellent discrimination potential. However, as pointed out by the authors, "it is inevitable that in some case material applications insufficient sample will be available and in others it may not be possible to extract the dyes in sufficient quantity. The fiber examiner may be reluctant in some circumstances to destroy the limited specimens" [*30*].

However, this reviewer is troubled by the lack of definition of the term "duplicate samples" as used by Macrae et al. If by this term it is meant that only two fibers from the same source were measured by MSP or SSP then the range of variation, that is, "scatter," is not well established and thus the threshold for discrimination can be in doubt. In the reviewer's experience, a case yielding several fiber matches through comparison microscopy was additionally examined through MSP. Two of these matches involved relatively infrequently found Verel fibers—one red, the other blue—both with the same distribution of "splotchy" delustrant particles. MSP did not differentiate the pair of red Verel fibers; a 5-nm difference was shown between the two blue Verel fibers. These fibers resulted from secondary transfers since they were not the fibers which composed their fabric sources, and thus they were sample limited for TLC. A possible explanation of the MSP 5-nm difference is that the parent source consisted of a "bad batch" of dyed blue Verel fibers. The parent source was unknown, thus the range of variability was not determinable and the result was necessarily reported as "inconclusive" with respect to the likelihood of common origin.

The burden of showing "scatter" becomes greater with greater discriminatory power. It would be self-defeating to use a method which is so sensitive to slight differences that like fibers known to be from the same source are "eliminated" from consideration as having had a like source. Knowing "the threshold for discrimination" in the practice of criminalistics is akin to and as important as knowing "the point of diminishing returns" in the practice of economics.

Beyond their usefulness for comparison purposes, the results from MSP and SSP can be used to gather quantitative information which can be used to answer the question: How common is this color? Paterson and Cook [*31*] point out that while the human eye is an effective device for the measurement of color, the response of different individuals shows great variations, making the recognition and recording of color very subjective.

These workers attempt to define color objectively through the entry of absorbance measurement values from SSP into complementary chromaticity coordinates (CCC). A beam condenser which they designed for use with a UV/visible spectrophotometer [*32*] was used to measure absorbance of dyes extracted from wool fiber samples. The extracting solvent was pyridine and water (3 : 1). Coordinates for 48 wool samples were given which showed a great range of values. All samples could be easily distinguished despite the fact that a number of them were visually similar. The application of the system to forensic samples remains a problem because of inaccuracies at low dilutions despite the use of the beam condenser.

Accessories for use with a spectrophotometer to accomplish the same purpose have been described by Smalldon [*33*]. They consist of two capillary micro-cells: vertical and horizontal. They are made from rectangular brass blocks by drilling holes and boring windows and fit into the usual cuvette holders of a Unicam SP 800B UV/visible spectrophotometer. Smalldon reports an increase in sensitivity of $\times 100$ over conventional arrangements.

Refractive Index and Birefringence

Listings of the refractive indices of fibers can be found in textbooks by Krauss [*12*], by The Textile Institute [*15*], in an article by Longhetti and Roche [*18*], and in test methods of ASTM Methods for Identification of Fibers in Textiles (D-276). Forlini and McCrone [*34*] gave refractive indices as dispersion staining data for samples of 66 man-made fibers and 92 natural fibers.

Forlini and McCrone's data were arrived at through mounting the fibers in Cargille refractive index liquids and use of a dispersion staining objective and plane polarized light. Their refractive index listings are given under F, D, and C (486, 589, and 656 nm, respectively) for both $n_{\parallel}$ and $n_{\perp}$. Fibers exhibiting low birefringence were also examined by removal of the polarizer, and a refractive index representing the average of the $n_{\parallel}$ and $n_{\perp}$ readings was obtained and listed.

The authors report unexpected precision for some natural fibers; for example, three specimens of jute fibers differed by ± 0.002 for $n_{\perp}$ and ± 0.0015 for $n_{\parallel}$. Also, wool fibers differed by ± 0.003 for $n_{\perp}$ and ± 0.004 for $n_{\parallel}$.

Their data reveal other interesting insights. The possibility of differentiating at least some of the acrylics is shown, as are the clusters of plant fibers around $n_D^{25} = 1.530$, and of animal fibers (protein based) around $n_D^{25} = 1.550$. In addition, Heuse and Adolf [*26*] describe as an example of the power of the double-beam interference microscope the differentiation of hemp fibers from flax fibers, a differentiation problem they describe as resistant to virtually all other methods in common usage. Examination of Forlini and McCrone's data shows that using a Cargille refractive index liquid intermediate between $n_{\parallel}$ for hemp and $n_{\parallel}$ for flax will differentiate the two in virtually all instances through central-stop dispersion-staining color effects imparted to their edges, for example, blue for hemp and red orange for flax, equivalent to matching wavelengths of approximately 625 and 560 nm, respectively.

One of the principal approaches to the identification of man-made fibers used by the Met-

ropolitan Police Forensic Science Laboratory as described in their manual [*16*] is the determination of birefringence through use of an interference wedge, and, as required, the insertion of a Red I plate for determination of the sign of elongation of fibers exhibiting low interference colors between crossed polars. Determination of refractive index is said to be necessary only in rare instances. Examination of the data of Forlini and McCrone and other listings will show that measurements of interference, in the absence of information as to the actual refractive indices, can lead to difficulties, for example, the distinction of acrylics from modacrylics. Mounting the fibers in a liquid approximately intermediate in refractive index of n_D^{25} for acrylics and n_D^{25} for modacrylics will distinguish the two through their distinct differences in edge colors as revealed by a dispersion-staining objective with the central stop in place.

Dispersion staining is a powerful tool for the microscopist. The theory which underlies it has been given by McCrone et al. [*35*].

Dispersion-staining colors reflecting the refractive indices of all man-made fibers excepting glass will vary depending upon their lengthwise orientation with respect to the polarizer. The two extreme orientations during the rotation of a fiber can show different effects. If, as in Fong's single-liquid scheme, a high-dispersion Cargille liquid of nD25 = 1.525 is used, the following examples of approximate central opaque stop color effects would be noted: pale blue for $n_\parallel$ and blue for $n_\perp$ with acrylics, white for $n_\parallel$ and blue for $n_\perp$ with polyamides, yellow or yellow-orange for $n_\parallel$ and blue or magenta blue for $n_\perp$ with rayons, blue/blue magenta for $n_\parallel$ and white/"rainbow" for $n_\perp$ with olefins (polypropylene), and no dispersion-staining colors with polyesters, acetates, and triacetates. Conventional determinations involving Becke line, birefringence, and signs of elongation differentiate the latter three.

The approximate intermediacy of the liquid nD25 = 1.525 for the principal refractive indices of fibers becomes clear through study of Forlini and McCrone's data. Fong's method was intended for the identification of synthetic fibers, and, for this reason, matching wavelength information for the natural fibers was not given. This omission is usually of no consequence since the natural fibers are recognized by their morphological appearance through the microscope.

However, some difficulties can arise with wool fibers. Such fibers are readily recognized if the scales on their outer surfaces are prominent and a liquid having a refractive index far removed from the refractive index of the fibers—nD25 = 1.550—is used for the mountant to achieve contrast. On occasion, the scales are not prominent, and this, together with the usage of a liquid refractive index of nD25 = 1.525, not far removed from nD25 = 1.550, can pose a problem if Fong's scheme is used since scales are not readily apparent.

Forlini and McCrone's data list dispersion-staining data for wool. Under the conditions of the single-liquid mount scheme, wool exhibits central-stop dispersion-staining colors in the approximate range of yellow orange to yellow for $n_\parallel$ and red orange to orange for $n_\perp$, equal to approximate matching wavelengths of 510 and 540 nm, respectively. In addition, normal information from a polarizing microscope can be obtained, for example, presence of weak birefringence and positive sign of elongation.

Density

A nonmicroscopic and nondestructive method suitable for the examination of single fibers was described by Bresee [*36*]. He pointed out that if density was determined to four or five significant figures, it could be used to determine the percentage of crystallinity of fibers, to measure the amount of a fiber additive, and to monitor polymer degradation. He hypothesized that "density gradient analysis offers a means to discriminate between fibers on the basis of a myriad of commercial practices encompassing extrusion, dyeing, and finishing performed under various conditions along with an endless variety of consumer practices including laundering and outdoor exposure."

Experimental procedures to test this hypothesis were performed by studying 14 commercial polyester fibers of the group known as poly(ethyleneterephthalate), commonly referred to as PET. A density gradient column having a sensitivity of 0.0002 $g/cm^3 \cdot mm^{-1}$ was prepared by reverse stepwise addition using carbon tetrachloride/*n*-heptane in a 500-mL graduated cylinder maintained at 23°C in a constant-temperature bath. Columns were calibrated with glass floats whose densities were known to 0.0002 g/cm^3. Fiber samples dried and cut to lengths of 5 mm, agitated in *n*-heptane to remove adherent particulates and deaerated in a light vacuum to remove entrapped air, were placed with tweezers under the surface of the liquid column. After a 24-h settling period, the locations of the fibers were read through crossed polars and recorded. Locations of the standard floats were used to construct a calibration curve.

Data for untreated and treated fibers were given. Three of the fourteen samples were subjected to low- and high-temperature washes. Samples of these three were also subjected to outdoor weathering continuously for two months. The treatments described were made according to methods prescribed by the American Association of Textile Chemists.

For untreated fibers, the density gradient technique was shown to "possess the ability to discriminate among fibers having different trade names as well as among fibers having identical trade names but different type" [*36*]. This statement is based upon readings recorded to four or five significant figures. On the other hand, it was stated that "the method possesses limited ability in identifying a particular trade name or type of a fiber if unknown" [*36*]. This was especially true for delustered fibers.

For treated fibers, the author noted "that laundering and outdoor exposure produce changes of such magnitude in the density of the new fibers that any attempt to identify the fiber type of an unknown fiber by density analysis would be futile if the fiber has been subjected to consumer use" [*36*]. It was further noted "that these changes provide a sensitive means for discrimination among fibers having identical commercial origins but used by different consumers who have different laundering habits or spend different amounts of time outdoors" [*36*].

Bresee did not give information regarding the degree of density scatter for fibers from the same source. Such information is critical for estimation of the threshold level at which fibers from two different sources are said to be differentiated. This observation notwithstanding, his method deserves additional experimental assessment to establish its value.

Melting Points and Solubilities

Readily applied tests to confirm identifications made by polarized light microscopy can be useful to the forensic fiber analyst. Such tests are available—for example, melting point and solubility—but they are destructive. This is not a great disadvantage when the amount of sample required is such that tests can be performed on a microscopic scale, that is, 0.5 mm long and 20 to 30 μm in diameter, since a sample of this length can be cut from a fiber which has been initially characterized through polarized light microscopy and birefringence.

Listings of melting points and solubilities for man-made fibers have been given by Longhetti and Roche [*18*] and in a publication of the DuPont Textile Fibers Department [*37*]. Another source for solubility properties is published as a standard method by the ASTM (D 276-85). The latter is less useful for forensic fibers identification since it presupposes large sample sizes.

Determinations of melting points of synthetic fibers on the hot stage of a microscope present problems because those that melt do so nonreproducibly over an appreciable temperature range and many decompose completely before a melting point is reached. Grabar and Haessly [*29*] use a Kofler hot stage on a polarizing microscope to obtain useful melting points by observing the fiber behavior as it is being heated. Decomposition caused by exposure to air can be offset by immersing the fiber in inert DC 710 silicone oil.

Eutectic melting points, obtained by the addition of a second component to form a mixture with the fiber before heating, can be used to offset decomposition. The second component for use as a reference compound is p-nitrophenol (mp 113 to 114°C). Approximately 2 to 3 mg of the compound is mixed with the fiber, and microscopic observations are made with a coverslip in place. Grabar and Haessly list eutectic melting points which are substantially lower than melting points obtained using silicone oil.

Examination of the available melting point listings reveals that Nylon 6 can be differentiated from Nylon 6.6. This is an analytical problem which resists solution by most other methods with perhaps the exception of infrared spectroscopy and pyrolysis gas chromatography. A similar observation can be made regarding the discrimination of some of the polyesters.

Cross-sectioning

The microscopic appearance of a fiber in cross section can assist in its identification or characterize it beyond its generic identification. But fiber cross-sectioning is laborious for routine use. It is rarely employed for the identification of natural fibers, although references are given by Krauss [*12*] and Stoves [*14*] as an aid to the identification of animal hairs and for the differentiation of flax and hemp. Cross-sectioning of synthetic fibers has become increasingly important as manufacturers of synthetic fibers introduce more versions of texturized fibers into their product lines to impart desired properties for specific customer end uses.

Microscopic examination of the fiber in its longitudinal view can be used as a basis for deducing the cross-sectional appearance by the experienced fibers analyst. This approach can serve for most purposes, but in circumstances judged to be exceptional it is best to do a cross section.

Deadman [*38*] describes the critical role of cross-sectional studies of fibers recovered from bodies of murder victims in the Atlanta area over a 22-month period beginning in July 1979. Before the development of the suspect, Wayne Bertram Williams, analysts of the Georgia State Crime Laboratory had located a number of yellowish green nylon fibers and some violet acetate rayon fibers on the bodies. After the development of Williams as a suspect, direct comparison of fibers from a green carpet in Williams' home against yellowish green fibers from head hair of the nude body of the most recent victim established an association.

Cross-sectional studies of the yellowish green nylon from the two sources revealed an unusual trilobate cross section. This observation prompted extensive background investigations into their source by manufacturer. The results showed that the fibers from the green carpet had been produced by the Wellman Corporation during the period 1967 through 1974 and that it was highly unlikely that fibers having similar trilobate cross sections were made by other manufacturers.

Cross-sectioning can be done by either hand techniques or mechanical microtome methods. Hand techniques suffice when the amount of fibers is abundant. Such techniques include a cork method as described by Krauss [*12*] and the metal plate method of Schwarz [*39*] as described by Stoves [*14*] and the cross-sectioning devices by Hardy [*40*]. Frei-Sulzer [*2*] recommends that mechanical microtome methods be used when only single fibers are available.

In the manual available from the Metropolitan Police Forensic Science Laboratory [*16*], a technique for preparing fibers for cross-sectioning by mechanical microtoming is described. The principal thrust of the description is the casting of an embedding block using a methacrylate resin in a cell prepared from pieces of glass microscope slides. Before the embedding medium is poured into the glass cell, the fibers are aligned and stretched taut between two pieces of double-faced adhesive tape fixed to the bottom of the cell.

Grieve and Kotowski [*41*] describe a modification of this technique using a two-component epoxy resin marketed in the United Kingdom under the name "Strongbond" as the embedding medium. By so doing, they reduce the long polymerization time for methacrylate

from in excess of 5 h to 15 to 20 min. Earlier, Rosen and Kerley [*42*] had described the usage of epoxy resin as an embedding medium for the mechanical microtoming of hair.

Catalogued illustrations of fiber cross-sectional appearances at high magnification are rare. One source is a technical bulletin issued by the Du Pont Textile Fibers Department [*39*]. This bulletin shows photomicrographs in longitudinal and cross-sectional views, at ×250 M and ×500 M, respectively, of 35 man-made and 7 natural textile fibers. A more recent and exceptionally comprehensive cataloguing is that of the Textile Institute [*15*].

Reactivity to Reagents

Reactions to chemical test reagents offer a method for confirming the results of color comparisons effected through microscopic comparison. This approach effectively alters the fibers, and when the reaction is severe, the fibers are destroyed. As indicated previously, if the test can be applied on a microscopic scale, this disadvantage can be held to a minimum by using cuttings of very short length from the fibers previously examined through microscopy. The significance of these test reactions increases with increasing number of independent tests so that several cuttings from a limited sample length may be required.

Frei-Sulzer [*2*] uses short-length single fibers, and, as required, cuttings as short as 50 μm can be prepared. He places the fiber in a very small drop of water on a microscope slide, and, while wet, and without a coverslip, he makes observations at ×200 M. Reagents are added with a capillary tube and color effects are noted.

Two fibers can be examined in the same drop; one is the questioned and the other—made to be the longer of the two—is the reference fiber. This procedure offers the advantage of simultaneous viewing. Alternatively, two separate drops can be used.

Frei-Sulzer lists a series of test reagents: weak acids, strong acids, weak alkali solutions, strong alkali solutions, oxidizing agents, reducing agents. Also, he describes alternating test reactions and special test reactions to detect acidophil and basophil dyes.

Thin-Layer Chromatography

Efficiency of extraction of dyes from the fiber is critical for successful discriminatory analysis by either solution spectrophotometry or thin layer chromatography. In 1979, Macrae and Smalldon [*43*] described a simple classification and extraction scheme for dyed wool fibers. They pointed out that 20 to 50 ng of each dye component must be extracted in order to effect successful differentiation and that, in most fibers recovered in casework, the fibers are 2 to 10 mm in length and contain between 2 to 200 ng dye, depending on the depth of dyeing.

Nine solvents were selected for the study of their extraction efficiency of wool fibers dyed with acid, chrome, metal complex, and reactive dyes. Extraction was performed by placing an approximate 1-cm-length fiber into a capillary tube along with 5 μL solvent and then sealing the tube. The same was done with an additional fiber, excepting that water was used as a solvent. The latter tube was used as a reference color. After the extraction period, the tubes were compared under a low power microscope at ×16 and the efficiency of dye extraction assessed on a scale of 0 (no extraction) to 5 (complete extraction).

A three-step procedure was developed which allows the worker to deduce the most likely dye class present. Also, the information resulting was said to be useful in discriminating fiber pairs. It was found that 90% of the dyed wool fibers encountered were readily extracted with aqueous pyridine, presumably because they contain acid dyes.

This paper contains useful and practical information for the forensic fibers analyst. In addition to the discussions relating to forensic fibers examinations, the authors give a treatment of the technical considerations which underlie and a citation of references not normally encountered by most forensic scientists.

Solvent extraction schemes for other fiber types have been given by Home and Dudley [*44*] for cellulosic fibers and by Beattie, Roberts, and Dudley for cellulose acetate fibers [*45*], and for polyester, nylon, and polyacrylonitrile fibers [*46*].

Choosing the best TLC developing solvent system presupposes that knowledge of the usage class of the dye is known. Macrae and Smalldon's work with wool fibers determined the probable class of dye present by noting its reaction when aqueous pyridine was used for solvent extraction. In 1980, Resua [*47*] gave extended information which included 22 dyed fibers, 4 of which were wool and 18 synthetic (nylon, Orlon®, Dacron®, Acrilan®, Arnel®, Vycron, Verel, and acetate). An additional 36 solid-dye samples were included in his study. Classes of dyes studied were acid, basic, disperse, reactive, direct, and vat.

Resua's sample sizes, 5 mm long and 1 mm in diameter, are unrealistically large for normal case work. His extracting techniques are essentially the same as those described by Macrae and Smalldon. Extracts spotted onto TLC plates (silica gel 60 F-254, Merck) were developed systematically in a series of solvent systems. Two systems were used for acid/basic dyes and an additional two systems for disperse dyes. The TLC behavior of the dye in a solvent designated as the screening solvent system (SSS)—chloroform/methanol/acetic acid (70:20:10 v/v)—indicated the class of the dye and determined the subsequent TLC solvent system to be used. Reactive, direct, and vat dyes were listed as immobile to the SSS, and none of the remaining solvent systems were indicated as useful for their separation.

Resua states, "The extraction process is non-destructive leaving the fibers intact for further analyses or court presentation" [*47*]. This is somewhat contentious since many criminalists, especially those who act as consultants for the defense, would regard alterations of the magnitude involved in dye extraction of a fiber as destructive. This minor observation notwithstanding, the work as described by Resua is a valuable approach, especially when at least one of the fiber sources is ample.

Thin-layer chromatographic comparison of the extracted dye content of two fiber samples under separate chromatographic conditions, such as different solvent systems, can be done when sample amounts are adequate. Theoretically, such a procedure should give increased discriminatory power. Practically, however, the worker can become the potential victim of an embarrassing self-delusion if solvent pairs which give little, if any, increase in discriminatory power are used, and, if there is an increase, it is not known to what degree. Resua, DeForest, and Harris [*48*] give criteria for determining the relative discriminating power of solvent pairs. The authors calculate correlation coefficients of solvent pairs from R_f values determined by separation of each generically different solid-dye sample in each of five different solvent systems. Low correlation coefficients for solvent pairs and the showing of relatively random distribution of R_f values through the usage of "scatter" diagrams are desired attributes leading to high discriminatory power. Equally important is resolving power, which was evaluated through the individual solvent system's ability to distinguish the major and minor colored components which can be present but are difficult to distinguish or are not shown at all by some solvent systems.

The authors use samples which would be considered unrealistically high in casework. These sample sizes were 5 to 10 μL of an extract of a fiber sample 2.0 cm long and 0.2 mm in diameter depending on depth of dyeing; the same sample sizes were used with dye samples made up to 0.1% in pyridine/water extraction solvent. For the purpose of their study, such sample concentrations and volumes were justified. However, one is inclined to wonder whether or not the conclusions would be different if the sample sizes were more like those found in practice. The criterion for resolution was the ability to detect and separate both major and minor components from dark fibers known to be composed of mixtures of three different dyes. It is reasonable to expect that the minor components, and perhaps some of the major components, will not chromatograph when samples are single fibers 1.0 cm long and 0.02 mm in diameter, such as occur in casework. If so, there is a possibility, intuitively small, that different conclusions as to the most desirable solvent pairs would result.

There is included in the methods, principles of approach, and mathematical considerations much information which the forensic fibers analyst will find to be of practical value. The article deserves close scrutiny for the benefit to be gained from its thought provoking content.

Infrared Spectrophotometry

The advantages of infrared spectrophotometry for the identification of complex organic materials are well recognized. In 1968, Fox and Schuetzman [*49*] sought to apply these advantages to the identification of man-made fibers in sample sizes found in forensic science practice. They described methods for sample preparation and the effects of dyestuffs and finishes on the resultant spectra.

Sample preparation involved cutting a fiber into four to eight segments, each 1 to 1.5 mm in length. A micro-pellet press, under vacuum, was used to press out a KBr pellet, into which the segments had been "sandwiched." The resulting pellet was 1.5 mm in diameter and 1 mm thick.

A beam condenser was used to introduce the die containing the KBr pellet into the sample beam path of an infrared spectrophotometer. Spectra produced were compared with available reference spectra in the literature and found to "have equal or better resolution than those provided by other methods requiring larger samples and more rigorous preparation of the material to be tested" [*49*]. Dyestuffs and finishes did not interfere.

Grieve and Kearns [*50*] describe a method for sample preparation for recording spectra "using amounts of fiber comparable to those used by Fox and Schuetzman with superior results." They employ a "universal" solvent (phenol/chloroform, 10% w/v) and powdered KBr to simultaneously dissolve and grind fibers in the well of a spot test plate. Gentle warming of the spot plate on a hot plate is employed as required. The mat formed at the bottom of the well is removed, and dried in an oven for 1 h at 100°C and then used to prepare 1.5-mm disks. The principal advantages claimed were that preliminary work to find a suitable solvent is avoided and that a better distribution of the fiber throughout the disk results.

Sample preparation for infrared analysis employing the techniques of solvent cast film and lead foil were described in 1978 by Cook and Paterson [*51*]. For the solvent cast technique they found that the sample sizes required were 3 μg (1 cm length, 20 μm in diameter). Choice of a solvent was based upon a "cursory identification" using microscopy. They comment upon the "universal" solvent method of Grieve and Kearns, saying that while it "may alleviate the problem of solvent choice, the phenol-chloroform mixture will not dissolve all fiber types" [*51*]. Some fiber classes form "mats," which involve loss of spectral quality when dealing with small quantities of material. Viscose rayon fibers are not amenable to the solvent cast film technique, and the technique of aligning cut fibers in the central apertures of discs (as used by Fox and Schuetzman) or in the central aperture of a lead foil as described by these authors must be used. This latter method requires approximately 1 μg of fiber (2 mm length and 20 μm diameter). In 1983, Garger [*52*] described a solvent cast technique for obtaining infrared spectra of acrylic fibers from samples he estimated as being from 1 to 1.5 μg in weight. This estimate was based upon considerations given by Cook and Paterson, that is, 2 mm length and 20 μm diameter = 1 μg. This was estimated roughly as one third the size of those required by previous methods. The fiber is dissolved with dimethylformamide in a sealed melting point tube heated at 100°C in an oven for 10 to 30 min and then transferred as a micro drop to a heated anvil from a micro-die. Repeated transfers are required. The film is peeled off and boiled in water to remove residual solvent, dried, and then fused to a micro-pellet of KBr. The method is both detailed and involved. It requires specialized equipment and instrumentation together with skill based upon practice. It has the appeal of being applicable to very small samples, and, as applied to the problem of differentiations within genera of synthetic fibers (for example, acrylics), practical application.

In 1973, Smalldon [*53*] described a scheme for within genera characterization of acrylics which employed considerations of physical characteristics and polymer composition as given by infrared analysis. This was the first application of infrared analysis to within genera differentiation for forensic science purposes to be described.

The physical characteristics used were surface appearance, as observed at ×500 M with the fiber under transparent adhesive tape for maximum contrast, and cross-sectional appearance. The surface appearances could be long striations, short striations, irregular, and relatively smooth. The cross-sectional shapes could be round, bean, peanut, lobed, acorn, and mixed peanut and acorn.

Polymer compositions were determined through infrared spectroscopy for six acrylic polymer compositions. For further differentiation of similar spectra, the absorbance ratios of $C = O$ (stretching):$C \equiv N$ (stretching) were used. This was pointed out as having doubtful usefulness in casework unless the mean and standard error for this property could be determined from control samples.

Smalldon's infrared data were gathered by using a tuft of fiber weighing a few milligrams and cutting short lengths which were allowed to fall into the potassium chloride powder for sample preparation. He found that satisfactory spectra were obtained for amounts typical of casework by using the method of Fox and Schuetzman, that is, single fibers weighing 1 to 20 μg.

Further employment of infrared analysis to within genera differentiations was described in 1977 by Grieve and Kotowski [*54*]. These authors used infrared analysis and melting point determinations. Lengths of fibers used for infrared analysis were 15 to 20 mm and were prepared by the "universal solvent" method of Grieve and Kearns. Data were given for 49 samples of polyester fibers representing at least 9 different types provided by manufacturers in various parts of the world. Infrared spectroscopy was described as allowing the differentiation of various modified types, but the authors concluded: "Analysts should be aware of possible confusion arising over the identity of fibers which cannot be conclusively differentiated by this method. The need for further study, possibly by nuclear magnetic resonance, is indicated" [*54*].

Pyrolysis Gas Chromatography

In 1971, Bortniak et al. [*55*] described their attempts to differentiate microgram quantities of acrylic and modacrylic fibers using pyrolysis gas chromatography (PGC). A total of 27 acrylics and 14 modacrylics were examined and compared. Significant points of differentiation exhibited by the pyrograms were given in tabular form and typical chromatographic patterns of the members of major groups were illustrated.

The authors said: "An overriding concern from the forensic viewpoint is sample size. The method described here requires only 40 micrograms" [*55*]. Using Cook and Paterson's method for estimating weight (2 mm length and 20 μm diameter = 1 μg), this requirement amounts to eight fibers, each 1 cm long and 20 μm in diameter. The selection of 40 μg was due not to lack of sensitivity but rather to the difficulty of handling fiber samples weighing less. This was so, as the authors state, because the differentiations were based upon peak height ratios and it was absolutely essential that the weights be constant. These authors say that a Nylon 6 monofilament fiber approximately 150 μm in diameter and 1.4 mm in length weighs approximately 40 μg and that this weight produced pyrograms which gave acceptable minor peaks without major peaks going off scale.

Following the work of Bortniak et al., no contribution to the forensic science literature on the subject of fiber analysis through PGC was noted for a period of several years. Wheals [*56*] commented on this lack and offered the explanation that the pyrolysis products of many fibers were too polar to pass through the packed columns in use during this time. He suggested pyrolysis-capillary gas chromatography (PCGC)—as a technique to improve results.

Challinor [*57*] took note of Wheals's suggestion and applied PCGC to a wide range of polymeric physical evidence materials including synthetic fibers. His paper described the use of a standard Pye Curie-point pyrolyzer interfaced to the injection port of the capillary column with a hypodermic needle. This arrangement permitted rapid attachment and removal of a normal packed column.

Pyrograms of Nylon 6 and Nylon 6.6 are shown by Challinor, and it can be seen that these fibers are easily differentiated.

Additional evidence of the differentiating power of PCGC was reported by Wampler and Levy [*58*]. They gave readily differentiated pyrograms of Nylon 6, Nylon 11, and Nylon 12.

Concluding Observations

When the situation permits and when required, there is an impressive array of available scientific hardware beyond microscopy that can be brought to bear on the task of fiber characterization. The situation permits when all of the needed hardware is at hand and the fibers are in abundance. However, the occurrence of these two requirements in combination is rare.

If the necessary hardware is available but the fibers are sample limited, the resourceful criminalist can attempt to augment the evidential value using a planned approach which gains the maximum amount of useful information for the least amount of required sample. The basis for so doing will be found in the articles cited and discussed in this critical review. If the problem is that the necessary hardware is not available or is limited, resort to contracted services is a viable solution.

Whether or not characterization beyond polarized light and comparison microscopy is required is a matter of judgment in individual cases. It is not forensic science dogma that a method must be used because it has been described in the literature. Many criminalists are reluctant to sacrifice the required sample or risk the possibility of loss inherent in the application of other methods, such as thin-layer chromatography, infrared spectroscopy, and gas chromatography, which require manipulations on a micro scale.

When exhaustive search yields only one match[2] through microscopic comparison and it involves a common fiber by type and color, the requirement for additional examinations becomes a necessity. On the other hand, when several different matches are available, say three or more, and each is relatively infrequent by type and color, the requirement for additional examinations is either nonexistent or of low urgency. This follows from consideration of the extreme improbability of chance occurrence of multiple fiber matches between sources known to be unassociated. This consideration is implicated with interpretative probabilities, which are beyond the scope of this review.

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