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Differentiation of Beige Carpet Fibers Utilizing an Analytical Scheme Incorporating Microspectrophotometry

presented by

Kristi Lynn Davis

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DIFFERENTIATION OF BEIGE CARPET FIBERS UTILIZING AN ANALYTICAL SCHEME INCORPORATING MICROSPECTROPHOTOMETRY

By

Kristi Lynn Davis

A THESIS

Submitted to Michigan State University in partial fulfillment of the requirements for the degree of

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ABSTRACT

DIFFERENTIATION OF BEIGE CARPET FIBERS UTILIZING AN ANALYTICAL SCHEME INCORPORATING MICROSPECTROPHOTOMETRY

By

Kristi Lynn Davis

In this study, an analytical scheme was utilized that incorporated the technique of microspectrophotometry in order to differentiate beige carpet fiber samples. Seventy-two carpet fiber samples were analyzed and compared utilizing a scheme which employed the following techniques – polarized light microscopy (including refractive index measurements), notation of level of delusterance, diameter measurements, ultraviolet fluorescence, cross-sectional analysis, and microspectrophotometry. The utility of microspectrophotometry as a fiber analysis technique was demonstrated in this study, as fourteen of the fiber samples were differentiated solely by their microspectrophotometry data. In addition, it was also observed that there is variation among fibers obtained from a single source. Thus, it would be desirable for a forensic fiber analyst to obtain several known and suspect fibers to compare in a criminal investigation utilizing this scheme.

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INTRODUCTION

Fiber comparisons can provide a wealth of information to a forensic scientist analyzing physical evidence from a crime scene. Fiber associations made between a suspect and a victim or between a suspect and a crime scene, can lend useful information to a criminal investigation. Due to the omnipresence of fibers and their transferability, they often are recovered in a criminal investigation for forensic comparison purposes and can provide a significant quantity of physical evidence. In some instances, fiber evidence may stand alone as the principal physical evidence in a criminal case. Because of these factors, forensic scientists are continually attempting to develop improved analytical schemes for their analysis and comparison.

A variety of analytical techniques have been employed by the forensic science community to analyze fibers, including cross-sectional analysis, polarized light microscopy, ultraviolet fluorescence microscopy, pyrolysis gas chromatography, and infrared spectrophotometry. This study is an attempt to determine the utility of microspectrophotometry as an analytical tool in a beige carpet fiber comparison scheme. The other analytical tools employed in an attempt to differentiate 72 fiber samples included level of delusterance, diameter, optical characteristics, ultraviolet fluorescence microscopy, and cross sectional profile.

The fibers used in this study were obtained from carpet squares originating from several manufacturers, including different brands from each individual manufacturer. Residential carpet fibers were chosen for this study due to their prominence and transferability. In addition, all of the fibers used were considered to be "beige" in

appearance. Although this is a subjective color term, it is used in the text of this research for simplicity to describe the fiber samples used which were any of several shades of light tan or brown, commonly referred to as beige.

Fibers as Physical Evidence

Fibers occur as both natural and man-made materials. A multitude of fiber types exist for the production of a variety of textile materials, but three fiber types predominate in the manufacture of carpeting materials – nylon, olefin, and polyester. These are all man-made fibers consisting of long-chain synthetic polymers of varying composition [5].

Fibers occur as physical evidence in a variety of criminal cases, and come from a range of sources. For example, carpet fibers can be transferred to the clothing or footwear, of a suspect or a victim in a criminal case. Previous studies have been conducted dealing with the transfer and persistence of fibers, which is clearly central to their utility in a criminal investigation. In 1975, Pounds and Smalldon conducted a series of fiber studies. These studies focused on fiber transfer [1], the persistence of transferred fibers [2], the recovery of transferred fibers [3], and the physical mechanisms of transfer [4]. One of the most significant facts to emerge was that approximately 80% of transferred fibers are lost during the first 4 hours after transfer. If a positive result is obtained from clothing taken within this time frame, the chances of identical fibers having originated from a different source other than the suspect one are very small, unless contact was also made with an alternative source during the same time frame. The fact that commonly encountered and transferable fibers from a range of sources were

compared using an analytical scheme employing microspectrophotometry, makes the current study useful to the forensic science community.

Beige carpet fibers present additional complications to the forensic scientist, however. This is due to the ambiguity of their perceived color under a microscope, and the relative uniformity of their cross-sectional shape, regardless of manufacturer or brand. Both of these properties serve to limit the differentiating power of conventional analytical techniques. Thus, additional testing is often desirable, and indeed necessary, in order to distinguish these types of fibers from one another.

Microspectrophotometry

Molecules have the capacity to absorb electromagnetic radiation. Those molecules that can absorb wavelengths in the visible region of the spectrum are known as pigments or dyes. When a beam of white light is incident upon a pigment molecule, certain wavelengths of the light are absorbed. The remaining wavelengths, which are reflected or transmitted, cause the pigment to appear colored. Microspectrophotometry capitalizes upon this property of pigments and dyes, and provides valuable information for the comparison of colored fibers. It is particularly useful in the analysis of metameric pairs. A metameric pair exists when two colors appear visually consistent with one another in one illuminant, but have different spectral curves [5]. In other words, it is possible for two fibers to appear indistinguishable in color using only comparison microscopy, and yet have different visible absorption spectra.

In addition to the extremely useful discriminating power of the microspectrophotometer, this technique allows non-destructive analysis of very small amounts of fiber evidence, and establishes a permanent record of the fiber color in only a

few minutes. These spectra can then be stored in a library, or in database format, for future comparisons if desired. Although microspectrophotometry is relatively expensive in comparison to other methods of fiber analysis, it can be considered to be complementary to comparison microscopy—the basic technique for fiber examinations in the forensic laboratory. This is due to the fact that the same mounted fibers used in microscopy can be examined without any further manipulative effort—a property that is very useful when either known or questioned fibers are of limited availability [6]. In addition, there are no problems when dealing with spun dyed fibers, or those where the dye is too pale to produce a satisfactory result from thin layer chromatography [7], which has caused difficulties in dealing with fibers in the past.

In this study, a S.E.E. 1100 (Version 2.0, June 2001) microspectrophotometer and Grams 32 computer software program were used. Transmittance was the method of sampling employed, utilizing a halogen lamp. In addition, the instrument was routinely calibrated using a variety of filters. The accuracy of the wavelength measurements was calibrated using holmium oxide and didymium filters, because the spectra of these filters display sharp peaks at well-documented wavelengths. The holmium oxide filter has peaks in the ultra violet and visible wavelength regions of the electromagnetic spectrum, and is used to calibrate the wavelength scale in the region from 280 nm to 640 nm. The didymium filter has peaks in the visible and near infrared wavelength regions, and is used to calibrate wavelength from 440 nm to 880 nm. The photometric accuracy is calibrated using a series of neutral density filters (OD 0.1, 0.5, and 1.0). These filters are characterized by a flat optical response in the wavelength region from 250 nm to 1,000 nm. All of the filters used for calibration are part of a NIST traceable filter set provided

by S.E.E. Incorporated, and they adhere to the ASTM standard practices for describing and measuring performance of UV, visible, and near infrared spectrophotometers.

Microspectrophotometry provided an essential element to this study as a final discriminating technique for several of the fiber samples. Fourteen fiber samples were indistinguishable from one another after optical property comparison using polarizing microscopy, delusterance evaluation, diameter determination, ultra-violet fluorescence microscopy, and cross-sectional analysis. However, after microspectrophotometry was conducted, it was possible to differentiate these fourteen fiber samples.

REVIEW OF THE LITERATURE

A variety of analytical techniques have been researched and utilized in an attempt to differentiate fibers. Past research endeavors have used methods such as polarized light microscopy, cross-sectional analysis, diameter measurement, melting point determination, infrared spectrophotometry, pyrolysis capillary gas chromatography, energy dispersive x-ray fluorescence, dye extraction, thin layer chromatography, and microspectrophotometry. Although several of these studies utilized more than one of these techniques, none of them used the combination employed in the present research and none of them were performed solely on beige carpet fibers, as was conducted in this study.

Pyrolysis capillary gas chromatography has been used to sub-classify fibers on the basis of slight variations in the pyrograms [8]. This is a useful technique, though destructive, if it is unclear as to the sub-type of fiber involved in a particular case. For example, it is possible to distinguish a nylon 6 fiber from a nylon 6,6 fiber using pyrolysis gas chromatography if prior testing was conducted to identify the two fibers as being nylon fibers. In the present study, a sampling of beige carpet fibers was obtained, and many of these were of the same class. It is outside the scope of this study, however, to conduct pyrolysis gas chromatography, for this study is ultimately concerned with the ability of microspectrophotometry to differentiate carpet fibers after other techniques commonly employed by a forensic laboratory have been exhausted.

A study conducted by Koons involving energy dispersive X-ray fluorescence allowed elemental characterization of individual fibers of very limited quantity [9]. This

method is fast and non-destructive, and can be carried out on lengths of fiber of only a few millimeters in length. The purpose of the Koons study was to determine if it was possible to class automotive and residential carpet fibers separately based on the differing combinations of elements present in the x-ray fluorescence spectra. Many automobile carpet fibers, for example, contain metallized dyes and other metal containing additives – most notably, cobalt, chromium, and zinc – to prevent degradation of the fiber under harsh conditions. Such additives were less frequently present in the residential carpet fibers examined in the study, allowing for at least a partial source classification of the fibers. The most useful application of x-ray fluorescence measurements according to this study may be in identifying the manufacturer of a particular carpet or fiber based on detection of manufacturer-specific elemental additives. Because the fibers used in the current research are all residential carpet fibers, x-ray fluorescence would impart little differentiating capability.

Hartshorne and Laing were able to achieve discrimination of the two types of olefin fibers possessing substantial commercial applications—polyethylene and polypropylene. These fiber types are being increasingly used in ropes, clothing, blankets, carpet backings, and carpet pile [10]. Infrared spectrophotometry alone provided a means of discriminating between polypropylene and polyethylene by the presence of strong absorption bands of approximately equal intensity at 1455 and 1370 cm⁻¹ and the absence of a band at 730 cm⁻¹ in the spectra of polypropylenes. However, it was difficult to discriminate between the infrared spectra of polyethylenes, and polyethylene spectra could also be confused with polypropylene/polyethylene mixtures. It was discovered in this study that melting point determination was an extremely useful analytical tool for

discriminating between these types of fibers. Infrared spectroscopy coupled with melting point determination thus provide an analytical scheme that is able to identify polyethylene types, confirm the presence of polyethylene/polypropylene mixtures, and discriminate polypropylene from all other olefin types and mixtures.

Prior to the Hartshorne and Laing study, research was conducted by Grieve and Kotowski involving infrared spectroscopy and melting point determination on polyester fibers [11]. This study revealed that distinction between the different chemically modified polyesters is not always possible using the infrared spectroscopy/melting point determination analysis scheme. The authors proposed the utility of nuclear magnetic resonance spectroscopy as a possible method of distinction, but also noted that samples are often limited in forensic science cases. It is clear that microspectrophotometry would be a useful tool to employ in such instances.

Dye identification research was one of the first avenues explored in an attempt to classify and differentiate fibers. Dye identification can be used to differentiate between fibers that are otherwise similar, and can be an essential factor in trace evidence analysis. Feeman revealed the utility of combining chromatography—both paper chromatography and thin layer chromatography—with infrared spectrophotometry in order to extract and "fingerprint" particular dyes [12].

Dye extraction and analysis was also conducted by Macrae, Dudley, and Smalldon in conjunction with microspectrophotometry [6]. The samples selected for this study were wool fibers from three distinct color groups—red, blue, and black. This study evaluated the discriminating power of microspectrophotometry as compared with that of microscopy, solution spectrophotometry, and thin-layer chromatography. It was found

that although microspectrophotometry was not useful for the deeply dyed black fibers, it was highly discriminatory for the red and blue fibers, and could be employed when sample size is limited.

Microspectrophotometry was first developed by Caspersson for the location and identification of chemical constituents within biological cells [13]. Microspectrophotometry has since been used in fiber research to compare the differing colors of dyes. Grieve, Dunlop, and Haddock were able to show that if a combination of comparison microscopy, microspectrophotometry, and fluorescence examinations were conducted on a sample of fibers, it is possible to discriminate between fiber dyes, despite considerable color overlap [14]. A follow-up study to this research was conducted in 1990 in order to determine if any additional discriminating capability could be achieved when thin-layer chromatography is performed on extracted dyes [15]. It was discovered that thin-layer chromatography was only effective in separating five out of twenty-one pairs of dyes that were consistent spectrally, and that this technique is severely limited due to the difficulty in extracting certain types of dyes from single fibers.

MATERIALS AND METHODS

Seventy-two individual carpet fiber samples were obtained from retail beige carpet squares intended for residential use. In order to determine if fibers differ within a single carpet square, two fiber samples from different locations on each carpet square were obtained, numbered as consecutive samples, and analyzed independently. For example, samples 5 and 6 originated from different regions of one carpet square, and samples 7 and 8 originated from different regions of another carpet square. The only exceptions to this were in samples 1-4 and 13-16, in which four samples were taken from different regions of two separate carpet squares. Data relating to the sources of the samples is provided in Table 1.

All of the tests performed on the fiber samples in this study prior to microspectrophotometry were conducted in the Northville Laboratory trace evidence unit of the Michigan State Police. Individual fiber samples were analyzed and categorized in the following sequence: general fiber type (determined by optical properties under the polarizing light microscope), level of delusterance, diameter, ultraviolet fluorescence properties, cross-sectional profile, and microspectrophotometry visible absorption spectra. Each test was conducted on each fiber sample, and the results recorded (see Appendix A and Tables 2-11). The fiber samples were compared with one another in the order of the tests as listed above, and once final differentiation of a fiber sample could be established, no data for that fiber sample was further compared.

Sample Numbers	Carpet Square Number	Manufacturer
1,2,3,4	1	Argonne Industries
5,6	2	Aladdin
7,8	3	Beaulieu of America
9,10	4	Beaulieu of America
11,12	5	Mohawk
13,14,15,16	6	Aladdin
17,18	7	Aladdin
19,20	8	Creston Carpet Mills
21,22	9	Queen Carpets
23,24	10	Beaulieu of America
25,26	11	Aladdin
27,28	12	Aladdin
29,30	13	Mohawk
31,32	14	Stevens Carpets
33,34	15	Beaulieu of America
35,36	16	Globaltex
37,38	17	Mohawk
39,40	18	Globaltex
41,42	19	Queen Carpets
43,44	20	Stanwich
45,46	21	Lacieba
47,48	22	Mohawk
49,50	23	Mohawk
51,52	24	Mohawk
53,54	25	Globaltex
55,56	26	Beaulieu of America
57,58	27	Beaulieu of America
59,60	28	Globaltex
61,62	29	Beaulieu of America
63,64	30	Queen Carpets
65,66	31	Aladdin
67,68	32	Beaulieu of America
69,70	33	Stevens Carpets
71,72	34	Globaltex

Table 1 – Fiber Sample Source and Manufacturer Data

Various morphological and optical characteristics were observed for each fiber sample using a Nikon Optiphot-Pol polarizing microscope equipped with a calibrated eyepiece micrometer, which is located in the Michigan State Police laboratory. Several fibers from each fiber sample were mounted on a glass microscope slide in 1.530 +/-0.0002 Cargille refractive index fluid, and a cover slip was applied. Several properties of the fibers were noted – specifically, color, diameter, retardation colors, sign of elongation, level of delusterance, and refractive index (both parallel and perpendicular to the plane of polarized light – n_{ll} and n_{\perp} respectively) relative to 1.530.

The sign of elongation of all of the samples in the study was determined by examining the refractive index measurements. The sign of elongation of a substance is considered to be positive if the sample has a higher refractive index for light vibrating along its length (parallel to the sample) than that for light vibrating across its width. Retardation colors of any sample result due to interference of light rays, both constructively and destructively to produce a color. Due to differing degrees of phase lag between the two beams of light transmitted by a sample, the interference of the rays ranges between complete constructive and complete destructive interference. The colors that result from this interference are distinct for certain multiples of wavelengths, and can be used for comparison purposes between samples.

Ultraviolet fluorescence properties were subsequently noted using a Nikon S-ke ultraviolet microscope, with Nikon Episcopic Fluorescence attachment (EF-D). The mounted fibers from each fiber sample that were examined for the ultraviolet fluorescence microscopy analyses were the same mounted fibers as were used in the

polarized light microscopy analyses. The color of the fluorescence, if present, and the relative brightness of the fluorescence were noted for each fiber sample.

General fiber type was determined from the optical properties of each fiber sample, specifically their refractive indices, both parallel and perpendicular to the plane of polarized light, and the retardation colors of each fiber sample were evaluated. This scheme was followed regardless of the fiber type noted on the manufacturer's tag. The level of delusterance of each fiber sample was noted, also using the polarizing light microscope (not utilizing the polarizer at this point) and the same fiber mounts as for the optical characteristic determination. The level of delusterance was further analyzed and categorized after digital photographs of the fibers could be taken and compared. Because the polarizing light microscope was equipped with a calibrated eyepiece micrometer, it was also possible to make diameter measurements on the fiber samples at this point in the research. Several diameter measurements were taken over various regions of several fibers in a sample (the same mounted fibers for each sample that had been utilized for all of the testing thus far). For the purposes of this study, the largest diameter measurement obtained for each fiber sample was used for comparison purposes.

In addition, cross-sections were cut for several fibers of each sample using the method described by Palenik and Fitzsimons [16]. The fibers utilized to obtain cross-sections were different fibers from each sample than were utilized for the other analytical tests. The fibers to be cut were placed on a piece of low-density polyethylene film, and then covered with another piece of film. The film containing the fibers was then "sandwiched" between two glass microscope slides, and heated on a hotplate, while slight pressure was applied to the covering glass slide, until the film became clear. The slides

were allowed to cool, and were then separated. The film containing the fibers was then viewed under a stereomicroscope, while cross-sections were cut through the film, with a sharp razor blade, perpendicular to the fiber axis. A series of sections were cut for each fiber. Several of these were transferred to a clean microscope slide, mounted in 1.530 +/-0.0002 Cargille refractive index liquid, and covered with a cover slip. The cross-sectional shape was then observed and recorded using the comparison microscope in the laboratory (an Olympus DH II), which was equipped with a digital camera. A digital photograph was then taken of a fiber from each sample lengthwise, visualized under 20 X magnification, along with its corresponding cross-sectional cut, visualized under 40X magnification. Thus, cross-sections were evaluated in much the same manner as the delusterance properties—using light microscopy initially, and digital photography as a permanent record of each sample's characteristics for future comparisons. These photographs can be viewed in Appendix F. (Note: These images in this thesis are presented in color.)

In addition to the above analyses, microspectrophotometry was employed to analyze each fiber sample. The same fiber mounts were used for this analysis that were used for the optical and morphological analyses described previously. Microspectrophotometric analysis was conducted on the S.E.E. 1100 (version 2.0), with Grams 32 software, in the forensic science laboratory at Michigan State University. The initial calibration of the instrument had been conducted prior to the initiation of this study, and involved measuring each filter 50 times over a period of 25 days. This was done in order to establish a baseline average for the specific microspectrophotometer used. After this initial calibration, the instrument was calibrated daily, and all

wavelength measurements were required to stay within +/- 3.0 nm of the NIST (National Institute of Standards and Technology) values on the calibration certificate under the same measurement conditions. All subsequent photometric measurements were expected to stay within the range specified on the certificate when the same measurement conditions were used as well. The instrument was also calibrated prior to each day's analyses using the NIST Traceable Filter Set from S.E.E. Incorporated. Calibration results were stored daily on the computer's hard drive for future instrument evaluation.

Another level of calibration was conducted every time a new sample slide was viewed. The Köhler illumination – the most desirable illumination for a wide variety of applications— of the microspectrophotometer was re-established, and the instrument's autogain function was performed [17]. In addition, a dark scan; a scan with the light source shutter closed which was conducted in order to ensure that no stray light would be affecting the results, and a reference scan; a scan on an area within the field of view that does not contain any of the sample, were performed on each new sample slide prior to taking any sample scans. [A reference area.] Once calibration was completed, ten scans were taken of each sample, using the transmittance mode. The scans were collected randomly from two fibers of a sample, scanned five times each, at two different locations each. The spectra from the scans for each fiber sample were then overlaid and printed for further examination.

RESULTS AND DISCUSSION

Upon comparison of the data obtained in this study, it became apparent that it was possible to distinguish several of the fiber samples utilizing an analytical scheme with fewer tests involved. For the purposes of this study, however, all of the tests were performed on all of the fiber samples in an attempt to discern the utility of a microspectrophotometry analysis step in an analytical scheme for carpet fiber comparisons. Results of these examinations were compared until final differentiation of the fiber samples could be achieved, regardless of where this occurred in the analytical scheme. Additional results that were not used to differentiate the fibers, but that were noted can be viewed in Appendix A.

The fiber samples analyzed in this study were initially sub-divided into three general fiber class types—polyester, olefin, and nylon. This level of classification was possible after the optical properties of the fiber samples, including their refractive index measurements in parallel and perpendicular polarized light, were noted. These classes were then further subdivided utilizing the analytical scheme outlined above in an attempt to differentiate the 72 fiber samples. Level of delusterance, diameter measurements, ultraviolet fluorescence properties, cross-sectional profile, and microspectrophotometry visible absorption spectra were compared among the samples of each fiber class.

Three delusterance categories were established in this study—those fiber samples exhibiting no delusterance (also termed "bright"), those exhibiting light delusterance, and those exhibiting heavy delusterance. The maximum diameter measurement and the color and relative brightness of the ultraviolet fluorescence of each fiber sample were also

compared. The microspectrophotometry absorption spectra were independently compared and separated into four spectral classes, based on peak maxima, and overall shape of the spectral curve. Examples of spectra from each of the four microspectrophotometry classes established in this study are shown in Figures 1 - 5. (Note: These images in this thesis are presented in color.) The remainder of the spectra for each class can be viewed in Appendices B - E.

















In this study, less weight was placed on the discriminating capabilities of crosssectional analysis as compared to the other techniques. This was largely due to the fact that all of the fibers used in this study had a similar perceived trilobal cross-sectional appearance while laying lengthwise on the slides. The similar cross-sectional appearance of the fibers was confirmed after comparison of the cut cross sections. However, for the purposes of this study, specifically that of determining the utility of microspectrophotometry, less emphasis was placed on the actual cut cross-sections. With limited sample (sometimes only one questioned fiber is available) the opportunity to not have to manipulate and destroy part of the sample by cutting cross sections is very desirable. Microspectrophotometry is a useful technique in situations such as these, as it is a non-destructive method of analysis that can be performed on very small samples. Limited sample size is often a consideration in forensic fiber analysis, exemplifying the utility of microspectrophotometry in fiber comparisons. Cross-sectional analysis does provide differentiating capabilities, however, and should be conducted if a large enough sample is available to the analyst. Manufacturers have even patented some crosssectional shapes in the past, but many of these patents have expired, making it difficult to identify the manufacturer by cross-section alone [18]. However, identifying manufacturer would not have provided any additional information in this study as the manufacturers were known at the time of sampling, and any single manufacturer was the source of several fiber samples.

Polyester Fiber Samples

Ten polyester fiber samples were analyzed in this study. Based on level of delusterance alone, it was possible to establish two groups of polyester fibers—samples 21 and 22 displayed heavy delusterance, whereas the remaining polyester fiber samples were bright, or lacking in any delusterance. Following the aforementioned scheme, it was then possible to differentiate samples 21 and 22 based on their diameter measurements.

The bright polyester fiber group was then sub-categorized as well, based on the diameters of the fibers in that group. Three fiber groups were established among the bright polyester fibers at this point. The first group, consisting of fiber samples 55 and 59, were differentiated by their diameter measurements from all other bright polyester fibers in the study. A second group—one pair of indistinguishable fiber samples, samples 8 and 49, was distinguishable from a third group of four other indistinguishable fiber samples, namely 7, 50, 56, and 60. These remaining six fiber samples were ultimately individualized based on their ultraviolet fluorescence microscopy properties. The results of these comparisons are summarized in Table 2.

Olefin Fiber Samples

All of the olefin carpet samples analyzed in the current study were determined to be polypropylene in composition, based upon their refractive index measurements taken both parallel and perpendicular to the plane of polarized light. (There are also polyethylene olefin fibers that are produced, but this variety of olefin fiber was not observed in the current study.) Two fiber samples, samples 9 and 10 were classified as

olefin fibers for the purposes of this study, despite the information printed on the manufacturer's tag, which claimed that the fibers were polyester. The classification used in this study was based on the optical information obtained using polarized light microscopy, including refractive index data.

Two groups of olefin fibers could initially be established based on delusterance properties. The lightly delustered olefin fiber samples (9, 10, 11, 12, 15, and 16) could all be further differentiated based on diameter data, whereas only six of the heavily delustered fiber samples could be differentiated based on diameter data (samples 2, 4, 17, 19, 44, and 62). The remaining heavily delustered olefin fiber samples required further comparisons. See Table 3 and Table 4 for lightly delustered and heavily delustered olefin fiber data.

Ultraviolet fluorescence comparisons for the heavily delustered olefin fiber samples were able to distinguish eight of the samples that appeared similar to the point of diameter comparison (samples 1, 13, 14, 20, 38, 39, 40, and 61). The remaining olefin fiber samples were subjected to further comparisons using cross-sectional analysis and microspectrophotometry data, as ultraviolet fluorescence microscopy data provided no additional discriminatory information for these fiber samples. Samples 3 and 6 were slightly different from one another in their cross-sectional shape, and were also in different spectral classes. This was also the case with samples 45 and 46, which were slightly different from one another in cross-sectional shape, and in different spectral classes. Although samples 37 and 43 had very similar cross-sectional shapes, they were
Sample Number	Delusterance	Diameter (µm)	UV Fluorescence
21	Heavy	56	Not Applicable
22	Heavy	54	Not Applicable
55	None	56	Not Applicable
59	None	44	Not Applicable
8	None	51	Bright green-yellow center/ Mod orange outline
49	None	51	Bright orange center/ Mod orange outline
7	None	49	Faint green center/ Mod orange outline
50	None	49	Bright orange center/ Mod orange outline
56	None	49	Mod green center/ Mod orange outline
60	None	49	Bright pale green

Table 2 – Morphological and Optical Properties of Polyester Fiber Samples

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Abbreviations: Mod = moderate, UV = ultraviolet

in different spectral classes as well, so these fiber samples could also be differentiated from one another. Thus, it was possible to individualize these six fiber samples unequivocally using microspectrophotometry. However, samples 5 and 18 had similar cross-sectional shapes, and were also in the same spectral class and remained indistinguishable from one another at the conclusion of this study. (Data for these samples are in bold in Table 4.)

Sample Number	Diameter (µm)
9	80
10	85
11	112
12	88
15	95
16	102

Table 3 - Diameter Measurements of Lightly Delustered Olefin Fiber Samples

Sample Number	Diameter (µm)	UV Fluorescence	Microspec. Class
2	59	Not Applicable	Not Applicable
4	68	Not Applicable	Not Applicable
17	61	Not Applicable	Not Applicable
19	78	Not Applicable	Not Applicable
44	85	Not Applicable	Not Applicable
62	88	Not Applicable	Not Applicable
1	71	Dull Dark Orange	Not Applicable
13	63	Faint Pale Green	Not Applicable
14	71	Faint Pale Green	Not Applicable
20	66	No Fluorescence	Not Applicable
38	63	Dull Dark Orange	Not Applicable
39	71	Faint Orange-White	Not Applicable
40	63	Faint Orange-White	Not Applicable
61	80	MG-WC/PGO	Not Applicable
3	66	Dull Dark Orange	2
6	66	Dull Dark Orange	1
37	76	Dull Dark Orange	1
43	76	Dull Dark Orange	2
45	80	Dull Pale Orange	2
46	80	Dull Pale Orange	1
5	73	Dull Dark Orange	1
18	73	Dull Dark Orange	1

Table 4 – Morphological and Optical Properties of Heavily Delustered Olefin Fiber Samples

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Abbreviations: M = moderate, G = green, W = white, C = center, P = pale, O = outline, UV = ultraviolet, Microspec. = Microspectrophotometry

Nylon Fiber Samples

Two groups of nylon fiber samples were established based upon comparisons of level of delusterance – bright nylon fibers, and heavily delustered nylon fibers. Diameter comparisons of the fiber samples in each group were able to distinguish several fiber samples. Among the bright nylon fibers, samples 26, 47, and 69 were individualized. Among the heavily delustered nylon fibers, samples 27, 33, 63, 64, 66, and 72 were individualized. See Tables 5 and 6 for a summary of the data obtained from the nylon samples.

Further comparisons were then conducted on the bright nylon fiber samples in an attempt to differentiate them as has been previously described for the polyester and olefin fibers. Seven of the bright nylon fiber samples were distinguished after ultraviolet fluorescence data was compared—samples 31, 32, 48, 51, 52, 54, and 70. Samples 57 and 58, however, were indistinguishable from one another in their diameters, ultraviolet fluorescence properties, and cross-sections. In addition, these two fiber samples presented excessive difficulty in microspectrophotometry analysis due to their lack of delusterance. This caused them to appear clear using the microspectrophotometer, and they were not detectable. Several attempts to scan each of these fiber samples on the microspectrophotometer did not produce any useable spectra. Thus, these two fiber samples remained indistinguishable from one another at the conclusion of this study. Two other bright nylon fiber samples 25 and 53. These fiber samples were indistinguishable from one another in their diameters, ultraviolet fluorescence properties, and cross-

sections, and were also in the same spectral class. (Data for the bright nylon samples that remained undifferentiated at the conclusion of the study are in **bold** in Table 5).

Sample Number	Diameter (µm)	UV Fluorescence	Microspec. Class
26	68	Not Applicable	Not Applicable
47	78	Not Applicable	Not Applicable
69	63	Not Applicable	Not Applicable
31	73	Br B-G C/Br P G O	Not Applicable
32	66	Br B-G C/ P B-G O	Not Applicable
48	76	Pale Green-Blue	Not Applicable
51	76	No Fluorescence	Not Applicable
52	80	No Fluorescence	Not Applicable
54	80	Pale Green-Blue	Not Applicable
70	66	Br B-G C/Br P B-G Outline	Not Applicable
57	90	Mod G C/ P G O	None Available
58	90	Mod G C / P G O	None Available
25	73	Br B-G C/PGO	3
53	73	Br B-G C/P G O	3

Table 5 – Morphological and Optical Properties of Bright Nylon Fiber Samples

Abbreviations: Br = bright, Mod = Moderate, P = pale, B = blue, G = green, O = outline, C = center, UV = ultraviolet, Microspec. = Microspectrophotometry Further comparisons among the heavily delustered nylon fiber samples were conducted producing the following results. Samples 28, 41, 67, and 71 were distinguishable from all of the other heavily delustered nylon fiber samples in the study based upon their ultraviolet fluorescence properties. Four pairs of fiber samples were indistinguishable from one another to the point of microspectrophotometry (samples 23 and 30, 24 and 29, 34 and 42, 65 and 68). Comparison of the microspectrophotometric class of these fiber samples enabled differentiation that would have otherwise not been possible, for each member of each previously indistinguishable pair, was in a different spectral class. Samples 35 and 36 were indistinguishable from one another in their diameter measurements, ultraviolet fluorescence properties, and cross-sectional profiles, and were also in the same spectral class. Thus, these two fiber samples remained indistinguishable from one another at the conclusion of this study. (Data for these samples are in bold in Table 6.)

Sample Number	Diameter (µm)	UV Fluorescence	Microspec. Class
27	51	Not Applicable	Not Applicable
33	85	Not Applicable	Not Applicable
63	61	Not Applicable	Not Applicable
64	80	Not Applicable	Not Applicable
66	73	Not Applicable	Not Applicable
72	44	Not Applicable	Not Applicable
28	71	Br B-G C/Mod B-G O	Not Applicable
41	66	Br G C/Br P G O	Not Applicable
67	66	Bright Green	Not Applicable
71	59	Br B-G C/P B-G O	Not Applicable
23	71	Br G C / P G O	2
30	71	Br G C / P G O	3
24	66	Br G C / P G O	2
29	66	Br G C / P G O	3
34	83	Br G C / P G O	2
42	83	Br G C / P G O	4
65	68	Br G C / P G O	3
68	68	Br G C / P G O	2
35	59	Br G C / P G O	4
36	59	Br G C / P G O	4

Table 6 – Morphological and Optical Properties of Heavily Delustered Nylon Fiber Samples

Abbreviations: Br = bright, P = pale, Mod. = moderate, G = green, B = blue,

C = center, O = outline, UV = ultraviolet

Microspec. = Microspectrophotometry

CONCLUSION

The current study reveals the utility of incorporating the technique of microspectrophotometry into an analytical scheme for forensic fiber analysis. Utilizing such a scheme in the present study enabled individual differentiation of 64 beige carpet fiber samples. In addition, of the seventy-two beige carpet fibers analyzed, fourteen were ultimately differentiated based on their microspectrophotometry visible absorption spectra. This represents approximately 20% of the original pool of fibers sampled, which would not have been distinguished without microspectrophotometry.

Nevertheless, the ambiguity of beige carpet fibers was not completely eliminated for the fiber samples examined in the current study, exemplifying the difficulties that such items could present to a forensic analyst. There remained four pairs of fiber samples that although distinguishable between the pairs, as well as among the other fiber samples, were indistinguishable within their pairings at the completion of the current study. It is also important to note that in only two instances were fiber samples taken from the same carpet square indistinguishable from one another. This was observed in samples 35 and 36 (obtained from one nylon carpet square) and in samples 57 and 58 (from another, different nylon carpet square). A summary of the analytical data obtained for the fiber samples that remained undifferentiated at the conclusion of this study is located in Table

7.

Sample #/ Fiber Type	Cross Section	Retardation Colors	Sign of Elongation	Refractive Indices (n ₁₁ / n ₁)	Diameter (µm)	UV Fluorescence	M.S. Class
5 / Olefin	Trilobal	Bright Rainbow	+	<1.53/<1.53	73	Dull Dark Orange	1
18 / Olefin	Trilobal	Bright Rainbow	+	<1.53/<1.53	73	Dull Dark Orange	1
25 / Nylon	Trilobal	Bright Rainbow	+	>1.53/<1.53	73	Br B-G C / P G O	3
53 / Nylon	Trilobal	Bright Rainbow	+	>1.53/<1.53	73	Br B-G C / P G O	3
35 / Nylon	Trilobal	Bright Rainbow	+	>1.53/<1.53	59	BrGC/ PGO	4
36 / Nylon	Trilobal	Bright Rainbow	+	>1.53/<1.53	59	BrGC/ PGO	4
57 / Nylon	Trilobal	Pale Rainbow	+	>1.53/<1.53	90	Mod G C / P G O	NA
58 / Nylon	Trilobal	Pale Rainbow	+	>1.53/<1.53	90	Mod G C / P G O	NA

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Table 7 - Summary of Data for Undifferentiated Fiber Samples

Abbreviations: Br = bright, B = blue, G = green, C = center, P = pale, O = outline NA = none available, UV = Ultraviolet, M.S. = Microspectrophotometery It can be seen in Table 7 that two false positive associations were made for samples 5 and 18, and for samples 25 and 53. False positive associations could have resulted in this study for two reasons. Two different manufacturers could have obtained the fibers used in their carpets from the same bulk fiber source. In addition, differences in the quantity of dye present on any given fiber could cause two fibers of different source to appear as if they were from the same source using these analytical techniques. A trace evidence analyst highly skilled in microscopy would have re-analyzed those fiber samples that were falsely associated. Using comparison microscopy, an analyst would re-evaluate the optical and morphological properties of each sample that was falsely associated with another, in an attempt to find minute differentiating qualities. In addition, Fourier Transform Infrared Spectrophotometry could have been conducted on those samples that were falsely associated in an attempt to differentiate them. These procedures however, are beyond the scope of this study, the purpose of which was to determine the utility of microspectrophotometry in a fiber analysis scheme.

Variation in dye dispersion across any carpet sample could also create false negative associations for individual fiber samples taken from that carpet. It would be expected that members of each consecutive pair of fiber samples (i.e. 5 and 6, 7 and 8, etc.) would be indistinguishable from one another, since each member originated from the same carpet square. (The only exceptions to this were in samples 1-4 and 13-16, in which cases four samples were taken from different regions of two separate carpet squares.) Thus, fiber samples originating from the same carpet square, could be distinguished from one another in most cases using this analytical scheme, causing them to appear as if they were from different sources. However, there is a tendency among

carpet manufacturers to blend fibers, so that any one carpet could include fibers of different cross-sectional shapes, or diameters [18]. This could potentially create false negative results in case work analysis of carpet fibers for a forensic scientist.

The forensic fiber analyst must always remain cognizant of the fact that a single fiber source, such as a single carpet square, can possess fibers that appear to be from different sources. Prior to excluding a suspect fiber as originating from a particular source, a comprehensive sampling of the fibers of known source should be analyzed. This also creates the potential for multiple-fiber consistencies to be found in a particular case. The probative value of finding several suspect fibers that are consistent with several fibers of known source is much more significant than finding a single suspect fiber that is consistent with a single known fiber, as is evidenced by the data generated in the current study.

The reasons for excluding Fourier Transform Infrared Spectrophotometry (FTIR) in the current study should be noted at this point. FTIR analysis of fibers requires the sample fibers to be flattened prior to analysis. It was unknown as to how this would affect the microspectrophotometry results of this study. In addition, the purpose of this study was focused on determining the utility of the technique of microspectrophotometry, and it was useful to evaluate the discriminating capabilities of this technique in an analytical scheme that did not involve FTIR analysis.

Future research should be conducted in the interest of the advancement of the forensic sciences. Specifically, the current project should be expanded to include other manufacturers of carpet fibers. It would also be of use to conduct further research of this

nature on new fiber types, and on carpets consisting of blends of current fiber types, as they are developed and disseminated to the general public.

Additional research efforts could also focus on the effects of flattening and wear on fibers, and how this manipulation alters their performance in the current analytical scheme. Carpet fibers that are not new are subjected to a range of conditions that could alter their performance in the current analytical scheme. These conditions include exposure to detergents that could fluoresce, exposure to ultraviolet radiation that could fade the dyes on the fibers, exposure to dirt and other contaminants, and the degrading effects of time. The results obtained using these manipulated fibers could then be compared to those of the un-altered fibers in an attempt to determine if fiber manipulation alters analytical results.

The ability to distinguish beige carpet fibers in a criminal investigation is extremely beneficial to the forensic science community. If a forensic scientist can conclude that one or more fibers obtained from a suspect are consistent with the type of fibers found at a crime scene, this can lend useful information in a criminal investigation. Indeed, positive fiber associations could be the ultimate connection between a suspect and a crime. Furthermore, if an analyst can eliminate a fiber found on a given suspect as potentially originating from fibers located at the scene of a crime, investigative efforts can be focused on other physical evidence, or can be directed toward other suspects in the case.

APPENDICES

APPENDIX A

Additional Morphological and Optical Properties of the 72 Carpet Fiber

Samples Analyzed

Sample Number /	Cross Section	Retardation	Sign of	Refractive Indices
Fiber Type		Colors	Elongation	n ₁₁ / n_
1 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
2 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
3 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / <1.53
4 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
5 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
6 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
7 / polyester	Trilobal	Green / White	+	> 1.53 / > 1.53
8 / polyester	Trilobal	Green / White	+	> 1.53 / > 1.53
9 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
10 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
11 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
12 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
13 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
14 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
15 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
16 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
17 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
18 / olefin	Trilobal	Bright Rainbow	.+	< 1.53 / < 1.53
19 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
20 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
21 / polyester	Trilobal	Green / White	+	> 1.53 / > 1.53
22 / polyester	Trilobal	Green / White	+	> 1.53 / > 1.53
23 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
24 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
25 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
26 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
27 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
28 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
29 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
30 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
31 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
32 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
33 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
34 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
35 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
36 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
37 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
38 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53

Table 8 – Additional Morphological and Optical Properties of Fiber Samples Analyzed

Table 8 (cont'd)

Sample Number /	Cross Section	Retardation	Sign of	Refractive Indices
Fiber Type		Colors	Elongation	n _{ii} / n_
39 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
40 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
41 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
42 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
43 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
44 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
45 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
46 / olefin	Trilobal	Pale Rainbow	+	< 1.53 / < 1.53
47 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
48 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
49 / polyester	Trilobal	Green / White	+	> 1.53 / > 1.53
50 / polyester	Trilobal	Green / White	+	> 1.53 / > 1.53
51 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
52 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
53 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
54 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
55 / polyester	Trilobal	Green / White	+	> 1.53 / > 1.53
56 / polyester	Trilobal	Green / White	+	> 1.53 / > 1.53
57 / nylon	Trilobal	Pale Rainbow	. +	> 1.53 / < 1.53
58 / nylon	Trilobal	Pale Rainbow	+	> 1.53 / < 1.53
59 / polyester	Trilobal	Green / White	+	> 1.53 / > 1.53
60 / polyester	Trilobal	Green / White	+	> 1.53 / > 1.53
61 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
62 / olefin	Trilobal	Bright Rainbow	+	< 1.53 / < 1.53
63 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
64 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
65 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
66 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
67 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
68 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
69 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
70 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
71 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53
72 / nylon	Trilobal	Bright Rainbow	+	> 1.53 / < 1.53

Sample Number	UV Fluorescence	Microspec. Class
7	See Table 2	3
8	See Table 2	3
21	Green Center/Dull Orange Outline	3
22	Green Center/Dull Orange Outline	2
49	See Table 2	3
50	See Table 2	3
55	Mod Green Center/Mod Orange Outline	3
56	See Table 2	3
59	Bright Pale Green	3
60	See Table 2	3

Table 9 – Additional Optical Properties of Polyester Fiber Samples

Abbreviations: Mod = Moderate, UV = ultraviolet, Microspec. = Microspectrophotometry

Sample Number	UV Fluorescence	Microspec. Class
1	See Table 4	1
2	Dull Dark Orange	1
4	Dull Dark Orange	2
9	No Fluorescence	1
10	No Fluorescence	3
11	No Fluorescence	1
12	No Fluorescence	1
13	See Table 4	1
14	See Table 4	1
15	No Fluorescence	3
16	No Fluorescence	3
17	Dull Dark Orange	1
19	No Fluorescence	1
20	See Table 4	2
38	See Table 4	1
39	See Table 4	2
40	See Table 4	2
44	Dull Pale Orange	2
61	See Table 4	2
62	Mod Green-White Center/Pale Green Outline	2

Table 10 – Additional Optical Properties of Olefin Fiber Samples

Abbreviations: Mod = Moderate, UV = ultraviolet, Microspec. = Microspectrophotometry

Sample Number	UV Fluorescence	Microspec. Class
26	Br G C / P G O	3
27	Br G-B C/Mod G-B O	3
28	Br G-B C/Mod G-B O	3
31	See Table 5	3
32	See Table 5	3
33	Br G C / P G O	2
41	See Table 6	4
47	Pale Green-Blue	3
48	See Table 5	3
51	See Table 5	3
52	See Table 5	3
54	See Table 5	3
63	Br G C/P G O	4
64	Br G C / P G O	4
66	Br G C / P G O	4
67	See Table 6	2
69	Mod B-G C / P B-G O	3
70	See Table 5	3
71	See Table 6	3
72	Br B-G C / P B-G O	4

Table 11 – Additional Optical Properties of Nylon Fiber Samples

Abbreviations: Mod = moderate, Br = bright, P = pale, G = green, B = blue, C = center, O = outline, UV = ultraviolet, Microspec. = Microspectrophotometry

APPENDIX B

Microspectrophotometry Spectra - Spectral Class 1





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Figure 11 – Fiber Sample 12

















Absorbance









APPENDIX C

Microspectrophotometry Spectra - Spectral Class 2












Figure 22 – Fiber Sample 23







Figure 24 – Fiber Sample 33































APPENDIX D

Microspectrophotometry Spectra - Spectral Class 3

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Absorbance












Absorbance









Figure 58 – Fiber Sample 59





















APPENDIX E

Microspectrophotometry Spectra - Spectral Class 4



























APPENDIX F

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Figure 71 – Heavily Delustered Polyester Fiber Samples

(cross-sectional and lengthwise views)





Figure 72 – Bright Polyester Fiber Samples (cross-sectional and lengthwise views)



Figure 73 – Lightly Delustered Olefin Fiber Samples



Figure 74 – Heavily Delustered Olefin Fiber Samples





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Figure 75 – Bright Nylon Fiber Samples (cross-sectional and lengthwise views)







(cross-sectional and lengthwise views)





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