Police Microanalysis

M. Edwin O'Neil

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It is interesting to note that there is a prevailing tendency, in all races, in the distribution of the pattern types among the digits. Whorls tend to be concentrated on digits I and IV, with a significant superiority of the right hand in this respect; digit II leads in the possession of arches, and radial loops also occur most commonly on digit II. Racial departures from this typical behavior must again be revealed in groups of individuals, just as the behavior itself is demonstrable only statistically. Another significant contribution by Poll to the technology of finger-print analysis is the construction of what he terms a "dactylodiagram," based on the frequencies of whorls and arches in specified finger pairs of right and left hands; for the detailed procedure reference must be made to the original publications. One expression of the result is the "lambda angle," in which racial differences are demonstrable, this being the figure cited in the press notices of Poll's work.

Among the finer traits of finger-prints which have been subjected to racial comparison, though in a limited number of peoples, may be mentioned pattern size (determined by ridge counting) and pattern form (as indicated in measurement of relative breadth and height). Here too it is evident that an existing racial difference is revealed only statistically.

In conclusion it may be emphasized that races do vary in their finger-print characteristics. It is true, further, that variations within the single race may occur as signs of sexual and constitutional differences of its components. But such differences are not diagnostic in the individual; they appear only by analysis of groups as unlike frequencies of the various finger-print traits which are discussed above.

POLICE MICROANALYSIS

II. TEXTILE FIBERS

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Most microanalysts who have written upon the subject of fiber analysis in relation to criminal investigation have devised analytical tables listing the microscopical or microchemical characteristics, by

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the application of which the investigator in hit-or-miss fashion may identify a fiber. Many of the chemical tests suggested are of little value for the reason that several different fibers respond in an identical manner to the same testing solution. Some of the tests recommended are exceedingly difficult to use because the reactions are not sufficiently clear-cut to lead to a positive conclusion, and in the hands of anyone but an expert they are extremely unreliable. The investigator who seeks help from the standard works on textile analysis, written for the textile expert, very likely becomes confused, either by the tremendous number of methods of identifying each type of fiber or by the very advanced technical procedures involved. Consequently, unless a definite and systematic course of procedure has been prepared by the investigator himself, to meet his own needs, the work of analysis will lack organization, much needless waste of time will result, and an absolute identification may not be reached.

What seems to be needed by the scientific investigator, then, is a scheme or procedure of analysis at once systematic and not too technical; a method which may be simply and easily applied without a great outlay of equipment and which will yield reliable results in the identification of an unknown. The present paper is an attempt to fill that need. The methods of analysis presented, while not intended to be the last word in fiber analysis, have been thoroughly tested and are deemed suitable for the use of the criminal investigator.

In order to simplify certain procedures the "analytical key" has been employed, since it is a convenient and readily usable form for tracing down an object or specimen to be identified. In the "analytical key" the properties of a number of objects are arranged in groups of contrasting characteristics, so that the object in question may be easily identified, more or less by the process of elimination, thereby conserving time and reducing the number of tests to a minimum.

Four different systems of analysis are presented, as follows: (1) Burning or ignition tests, (2) Fluorescence analysis, (3) Microscopic analysis, and (4) Chemical analysis. The first two are of importance only for preliminary study, the final identification being made either by microscopic examination or chemical analysis or a combination of these. As to the choice of methods, it is suggested that as many as possible be utilized, depending of course upon the amount and character of the unknown. The specimen presented for identification frequently may be very small, sometimes a short, single thread, so that if the chemical method of analysis is followed the material might be exhausted before a determination could be made.
In dealing with fibers which are artificially dyed the chemical color reactions are in general not applicable, although in some instances the fiber in question may be treated first with a bleaching agent or other chemical which will remove the dye.

The most important textile fibers from the standpoint of criminal investigation are cotton, linen, wool, natural and artificial silks, and perhaps jute, hemp and ramie. The latter three, together with a few types of lesser importance, will be discussed in a later paper.

**Burning Test**

As a preliminary to the other identification methods or as a rough physical test to determine the general group to which a fiber belongs, the burning test may be applied. The unknown fiber should be ignited by touching the end to a flame, and then noting the manner in which it burns and also the odor of the fumes. Ordinarily, the animal fibers, wool and silk, burn very slowly and emit an odor like that of burning feathers, whereas the fibers of vegetable origin burn rapidly with little accompanying odor. Other simple tests may be applied to differentiate fibers of either group, for example the sulphur-containing fibers (wool and hair) may be distinguished from those containing no sulphur (silks) by observing the effect of the fumes on lead acetate paper which is turned brown or black in the presence of hydrogen sulphide. These simple physical and chemical tests should be made according to a definite scheme or procedure to insure rapid identification with little loss of material through repetition of tests.

The necessary materials for this type of analysis are the following: Bunsen burner, fine-pointed forceps, closed-tubes or small test tubes, red and blue litmus paper, filter paper, and 5% lead acetate solution.

The tests should be made as follows: (1) Using forceps, ignite each kind of fiber by touching one end to a small gas flame. Observe the way in which the fiber burns, the appearance of the burnt end of the fiber when ignition is stopped, and the color of the ash. (2) Burn each of the distinct kinds of fibers or threads by heating in small closed-tubes over a Bunsen burner. Note (a) the odor of the fumes, (b) action of fumes on moist red and blue litmus paper, and, if an animal fiber (c) the effect of the fumes on a piece of filter paper moistened with a solution of lead acetate.

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1. If the sample to be identified is in the form of cloth, the warp and weft threads must be raveled out and tested separately.
Key to Fibers:

I. Fibers smoulder or burn slowly and give off fumes with a characteristic odor like that of burning feathers or hair. When removed from flame they do not continue to burn readily and a charred bead remains at the end of the fiber. Fumes turn red litmus blue. (Animal fibers):

A. Odor strong, disagreeable; fumes turn lead acetate paper brown or black—wool.
B. Odor not so pungent; fumes have no effect on lead acetate paper—silk.2

II. Fibers burn rapidly with a flame and give off but little smoke or fumes. Charred bead not present when fiber is removed from flame.3 Fumes turn blue litmus red. (Vegetable fibers):

A. Threads highly lustrous (Artificial Silks):
   1. Fumes with acid-like odor, very pungent, residue black—acetate silk.
   2. Fumes with little or no odor, residue white—viscose, nitrocellulose and cuprate silks.
B. Threads with little or no lustre (Cotton and Linen):
   1. Burnt end of thread curls slightly and has tufted appearance—cotton.
   2. Burnt end blunt and straight—linen.

Fluorescence Analysis

Although a great deal of progress has been made in the technique of ultra-violet fluorescence analysis, the method at the present time is not a reliable one for the positive identification of fibers. However, fluorescence analysis as a means of identification is frequently useful in ascertaining the general group to which a fiber belongs or the possible difference in origin of two or more questioned samples when it is necessary to preserve the material intact or when it is desirable to avoid a lengthy chemical or microscopic analysis.4 In general, the vegetable fibers exhibit a yellow fluorescence in ultra-violet light, whereas the animal fibers show a bluish fluorescence. The identifica-

2A slight positive test may be obtained if sulphates are present in the weighting, but the reaction is not so marked as in the case of wool.
3Acetate silk may burn slowly like the animal fibers but the characteristic odor of burnt feathers is not present. Acetate silks are dissolved by acetone; all other fibers are unaffected.
4Wynn and Donovan have shown that it is possible to distinguish between old and new cotton fibers since the fluorescence color changes from a violet to an ivory-white or brownish-white with increase in age and the probable change of cellulose to oxycellulose. Melliand Textile Monthly, 4 (5): 298ff.
tion frequently may be carried beyond this point, especially when the fibers are not dyed or treated with other dressing material during the processes of manufacture.

The following table, adapted from the results obtained by Nopitsch, gives the fluorescent colors of a number of fibers examined with a Hanan type mercury vapor arc lamp with filter, and the colors of the same fibers as seen in daylight:

<table>
<thead>
<tr>
<th>Material</th>
<th>Fluorescence Color</th>
<th>Daylight Color</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. Unbleached wool</td>
<td>Brilliant light blue</td>
<td>Light yellow</td>
</tr>
<tr>
<td>2. Bleached wool</td>
<td>Bluish-white to bluish-yellow</td>
<td>White</td>
</tr>
<tr>
<td>3. Bleached cotton</td>
<td>Light yellow</td>
<td>White</td>
</tr>
<tr>
<td>4. Mercerized cotton</td>
<td>Light yellow</td>
<td>White</td>
</tr>
<tr>
<td>5. Bleached linen</td>
<td>Brilliant yellowish-white</td>
<td>White</td>
</tr>
<tr>
<td>6. Cuprate silk</td>
<td>Reddish-white, with blue-violet</td>
<td>Brownish-white</td>
</tr>
<tr>
<td></td>
<td>shadow</td>
<td></td>
</tr>
<tr>
<td>7. Viscose silk</td>
<td>Sulphur yellow with blue shadow</td>
<td>Brownish-white</td>
</tr>
<tr>
<td>8. Nitro silk</td>
<td>Brilliant flesh color</td>
<td>Yellowish-white</td>
</tr>
<tr>
<td>9. Acetate silk</td>
<td>Bluish-violet</td>
<td>White</td>
</tr>
<tr>
<td>10. Natural silk</td>
<td>Very bright light blue, much brighter and whiter than acetate silk</td>
<td>White</td>
</tr>
</tbody>
</table>

Microscopic Analysis

Of all the various methods for identification of fibers, microscopic examination, in general, is unquestionably the most reliable and best. When combined with a few microchemical tests, carried out directly on the microscope slide, identification may be made easily and exactly, and no other type of examination is necessary. Very rarely a fiber may have been so altered in manufacturing processes or otherwise that the usual identifying peculiarities are wanting, in which case recourse must be had to some other method; again, the microanalyst may wish to apply other tests of a confirmatory nature to supplement the microscopic analysis; but in most instances microscopic identification will be found sufficient. Moreover, this type of analysis is desirable when the unknown sample is small in size, a factor which precludes the application of burning tests and chemical analysis.

Very little apparatus is required for the microscopic examination of textiles, the necessary items of equipment including a standard monocular microscope of 100 to 500 magnification, glass slides, cover glasses, a few reagents and mounting media in dropping bottles, and a small dissecting set consisting of teasing needles, fine-pointed forceps, scalpels and small scissors.

The fibers to be examined are placed on a slide, teased apart with needles, covered with a glass slip and transferred to the stage

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of the microscope. Fibers are usually mounted dry, in water, in a mixture of equal parts of glycerine and water, or sometimes in 15% alcohol. The microanalyst must experiment with various mounting media to determine the ones most suitable for bringing out various structures to be studied.

In addition to the characteristics of fibers listed below the microscopist should have for reference drawings and photographs of all types of fibers and a good reference collection of known samples.

The microscopic characteristics of the common textile fibers may be summarized as follows:

**Cotton**—Unicellular filament, flat, ribbon-like, twisted spirally to right or left on its axis; central canal or lumen broad, uniform in diameter; cell wall thick, covered by a thin, structureless, waxy cuticle. Fiber tapers gradually to a blunt or rounded point at one end.

**Mercerized cotton**—Straight, cylindrical, with occasional twists; unevenly lustrous, smooth except for occasional transverse folds or wrinkles. Cuticle mostly lacking, lumen irregular in width.

**Linen**—Multicellular filament, straight and cylindrical, not twisted and flattened, tapering to a sharp point. Cell wall thick, the lumen appearing as a narrow dark line in the center of the fiber. Filament marked by transverse lines at intervals, causing the fiber to appear jointed, resembling bamboo. Cross lines frequently intersect appearing like the letter X.

**Cultivated Silk**—Smooth, cylindrical, lustrous threads, usually single but often double, the twin-filaments held together by an envelope of gum. More or less transparent, without definite structure.

**Wild Silk**—Similar to cultivated silk but broader and less regular in outline. Marked by very fine longitudinal striations with infrequent diagonal cross-markings.

**Artificial Silk**—Cylindrical, lustrous, appearing like a glass rod. Microchemical reactions: dissolved rapidly by half-saturated chromic acid; not colored by Millon's reagent as in the case of true silk.

**Wool**—Easily distinguished by presence of flattened, overlapping epidermal scales not found on silk or any of the vegetable fibers. Fiber many-celled, cylindrical; shaft composed of three layers; central core or medulla (seldom seen), cortex, and scaly cuticle.

**Chemical Analysis**

If the sample submitted for analysis is fairly large, such as a piece of cloth or a number of large threads, it is recommended that a chemical analysis be made to supplement the microscopic examination and confirm the results obtained from that procedure. If the sample is relatively small in size and insufficient for a complete
chemical analysis, a few well chosen tests may be performed, the quantities of sample and reagent being adapted to the size of the unknown, the smallest specimens being tested directly on the microscope slide and the reaction observed under low power of the microscope.

Many of the reactions which depend upon color for differentiation of fibers are useful when dealing with white or light colored material, but cannot be employed if the fiber has been dyed. In some instances the fiber may be decolorized by boiling with such solutions as 1% hydrochloride acid, acetic acid, or dilute potassium hydroxide. No definite directions can be given for the use of these solutions, the choice depending upon the fiber; the type of dyeing material and the color test to be made.

The materials required for the tests outlined below are the following: beakers (30 cc and 100 cc); watch glasses (assorted sizes); micro-burner; dropping bottles (for reagents); small test tubes (2 cc and 10 cc);—Test solutions: sodium hydroxide (10% solution: 10 grams to 100 cc of distilled water); lead acetate (5% solution); oxalic acid (5%); chromic acid (half-saturated solution); Millon's reagent (dissolve 10 grams metallic mercury in a mixture of 25 cc concentrated nitric acid, and 25 cc warm distilled water. Dissolve 10 grams of mercury in 20 cc of fuming nitric acid. Mix the two solutions and place in a brown, glass-stoppered bottle); picric acid (0.5% solution); sulphuric acid (concentrated); ammonium hydroxide (concentrated and dilute); iodine solution (dissolve 20 grams of iodine in 100 cc of a saturated solution of potassium iodide).

Key to Fibers:

I. Place sample in a small beaker and cover with 10% sodium hydroxide solution. Cover beaker with a small watch glass and boil gently over a micro-burner for 10-15 minutes.

A. Sample dissolves—Wool or Cultivated Silk:
   1. To a portion of the alkaline solution add a few drops of 5% lead acetate solution:
      (a) Solution turns brown or black—Wool.
      (b) No brown or black coloration—SILK.

B. Sample does not dissolve—Cotton, Linen, Wild Silk, or Cellulose Silk: (Wash sample with water, place in a beaker with a quantity of 5% oxalic acid and boil for 15-20 minutes. Remove and boil in several changes of distilled water or until the sample gives no test for starch—i. e., blue color
with iodine solution. Dry thoroughly between pieces of filter paper and divide into several portions).

1. Treat one portion with half-saturated chromic acid:
   (a) Sample dissolves rapidly—ARTIFICIAL SILK.
   (b) Sample dissolves slowly or not at all—Cotton, Linen or Wild Silk.

2. To another portion in a watch glass add one or two drops of Millon’s reagent and warm slightly:
   (a) Sample colored pink or rose-red—SILK.
   (b) Sample not colored—Cotton or Linen.

3. Spot another portion with 0.5% picric acid; wash thoroughly in water:
   (a) Sample dyed yellow—SILK.
   (b) Sample loses color when washed—Cotton or Linen.

4. Immerse a sample for exactly two minutes in concentrated sulphuric acid, rinse well in distilled water and then in dilute ammonium hydroxide. Remove and dry:
   (a) Sample gelatinized; soluble in water—COTTON.
   (b) Sample little affected; not soluble—LINEN.

5. Immerse a small portion in a 1% alcoholic solution of fuchsin for 2 minutes and wash in ammonium hydroxide:
   (a) Sample slightly colored—COTTON.
   (b) Sample colored rose-red—LINEN.

6. To another portion of the washed fiber add a few drops of iodine solution, allow to remain for one minute, wash in water, and place in a watch glass of distilled water;
   (a) Sample remains blue or black—MERCERIZED COTTON.
   (b) Sample loses color in a few minutes, fading to a yellow or brown—COTTON.

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The artificial silks may be distinguished by the following test: Place fiber on microscope slide, add 2 drops of Iodine-potassium iodide solution (1% potassium iodide solution containing slight excess of iodine) and allow to soak for 2 minutes. Remove excess of solution with filter paper and add 2 drops of sulphuric-glycerol reagent (20 cc glycerine, 10 cc of water, 30 cc conc. sulphuric acid). Warm slightly over a micro-burner and examine under low power of the microscope.

(i) Fiber colored yellow.................ACETATE SILK.
(ii) Fiber colored violet..................NITROCELLULOSE SILK.
(iii) Fiber colored dark blue..............VISCOSE SILK.
(iv) Fiber colored light blue............CUFRATE SILK.

Treat (iii) and (iv) with conc. sulphuric acid:

Reddish-brown coloration ..................VISCOSE SILK.
Yellow brown (or yellow) ..................CUFRATE SILK.