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U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

**MICROSCOPIC METHODS USED
IN IDENTIFYING COMMERCIAL
FIBERS**

CIRCULAR C423

Reference book not to be
taken from the library.

U. S. DEPARTMENT OF COMMERCE

HARRY L. HOPKINS, Secretary

NATIONAL BUREAU OF STANDARDS

LYMAN J. BRIGGS, Director

CIRCULAR OF THE NATIONAL BUREAU OF STANDARDS C423

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By Thora M. Plitt

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PREFACE

The nature of the fibers utilized by everyone in textiles, papers, and countless other articles and the processes to which these fibers are subjected, frequently determine the quality of the product. This is a matter of interest to the consumer as well as the producer. The rapid strides in research and in industrial practices call for efficient methods of fiber analysis, hence microscopic methods are becoming increasingly important. Hitherto such information as has been applicable has been widely scattered. This Circular is a compilation of about 50 methods collected during a survey of the technical English, French, and German literature. All of the tests have been tried and are now employed in the fiber laboratory of the National Bureau of Standards. They have been subjected to rather stringent criteria in view of the fact that the composition of the samples is usually unknown.

The frequent inquiries received by the Bureau from research, testing, and standardizing laboratories, educational institutions, department stores, and dyers and cleaners concerning methods of identifying the numerous types of fibers examined by the Bureau, indicate that there is a real need for such knowledge. This publication has been prepared in order to make the results of the Bureau's investigation more readily available.

LYMAN J. BRIGGS, *Director.*

MICROSCOPIC METHODS USED IN IDENTIFYING COMMERCIAL FIBERS

By Thora M. Plitt

ABSTRACT

A compilation of about 50 microscopic methods for the identification of paper, textile, and cordage fibers has been made. During a survey of the literature these were selected and tried in the fiber-testing laboratory. Their relative merits are discussed.

Specifically there are included the following: General directions for the preparation of samples for examination, and for the determination of percentage of constituents present; general tests for orientation; tests for the identification of ground wood, sulfite, sulfate, coniferous, deciduous, and bleached wood pulps; of cotton, used cotton, mercerized cotton, flax, ramie, wool, damaged wool, casein fiber, silk, "wild" silk, and the different rayon fibers; also, of hemp, sisal, jute, coconut, and abacá (manila).

In addition to inspection by the aid of magnification, the means of approach include staining reactions, ashing preparations, and the use of polarized and ultraviolet light. References to original sources and standard pertinent works are appended.

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I. INTRODUCTION

This is a compilation of microscopic methods collected during a survey of the literature to assist in the identification of fibers currently in use in the industries. Much of this information is scattered and is not readily accessible. As far as practicable either direct quotations or as literal translations as possible have been rendered. All the methods given have been tried and found useful. Comments covering the experiences in their employment in this laboratory are included.

References to the originals and to other publications which include either illustrations or comprehensive considerations of the tests are given.

The tests include methods for the identification of paper and textile fibers, the quantitative determination of the composition of mixtures, and the detection of certain treatments which they may have undergone. The fibers which are most widely used, such as cotton, wool, and silk, are so readily identified by their well-known structural features that they are not dwelt upon here.

A knowledge of microscopy is assumed as comprised by such publications as the following:

E. M. Chamot and C. W. Mason, *Handbook of Microscopy*, vol. 1 and 2 (1931).

H. Molisch, *Mikrochemie der Pflanze* (1923).

O. Tunmann (L. Rosenthaler), *Pflanzenmikrochemie* (1931).

In conjunction with the tests, the morphological characters of the fibers must be taken into account. These are not illustrated here, since they have been described in the following references:

ASTM Standards on Textile Materials. American Society for Testing Materials (1937).

T. F. Hanausek, *The Microscopy of Technical Products* (1907).

P. Heermann and A. Herzog, *Mikroskopische und Mechanisch-technische Textiluntersuchungen* (1931).

W. Herzberg, *Papierprüfung* (1932).

J. M. Matthews, *The Textile Fibres* (1924).

TAPPI, *Tentative and Official Testing Methods—Recommended Practices—Specifications of the Technical Association of the Pulp and Paper Industry* (1936).

All methods found to be feasible are given in order that the results of one test, which may lack specificity, may be confirmed by information obtained by independent methods. Most fibers are biological products and as such are subject to variation within limits. Moreover, new synthetic fibers are being constantly introduced. Hence, it is frequently advisable to make comparisons with known fibers.

II. TESTS

1. PAPER FIBERS

(a) GENERAL METHODS FOR PREPARATION OF PAPER SAMPLES

(1) *Federal Specification Method* [7]¹

"A piece about 5 millimeters square shall be cut from each of 10 sheets of the delivery sample being tested. These pieces shall be placed in a 50 or 100 cubic centimeter² beaker or Erlenmeyer flask with approximately 20 cubic centimeters of 2 percent solution of potassium hydroxide, then boiled, and washed thoroughly with water. This sample shall then be rolled into a ball and worked between the index finger and thumb. The ball of paper shall then be placed in a test tube approximately 15 by 125 millimeters. Fill tube about three-fourths full with water and shake thoroughly until fibers are completely separated. After shaking, transfer about 5 cubic centimeters of the thoroughly mixed pulp to another test tube and fill tube about three-fourths full with water and shake well. As small a sample as can be conveniently

¹ Numbers in brackets indicate the literature references at the end of this paper.

² Cubic centimeter is often used for the correct term milliliter.

handled shall be removed with needles or fine forceps, placed on a glass slide, and water removed by means of hard filter or blotting paper.

"The slide shall be prepared by removing several samples of pulp from test tube as described * * * and stained in accordance with details of the respective methods * * *."

(2) TAPPI Method [45]

(Preparation of Samples)

"The specimen for test shall consist of pieces having a total area of not less than 6 square centimeters (1 square inch) cut from different portions of the test sample, so as to be representative of it.

"Place the specimen in a small beaker and completely cover it with 0.5 percent caustic soda or caustic potash solution, heat to boiling, transfer the contents of the beaker to a small 200-mesh metal sieve and wash thoroughly with water. Roll the moist pieces of paper into a ball and work between the fingers to loosen the fibers. Transfer to a test tube and shake until the fibers are completely separated. Pour a portion of the mixture into a second test tube and dilute to a fiber concentration of about 0.1 percent. Transfer the fibers to a microscope slide by means of the dropper * * *. Thoroughly mix the fibers and water, quickly insert the dropper into the mixture 5 cm (2 inches) below the surface, expel two bubbles of air from the dropper, then fill the tube to a distance of about 13 mm ($\frac{1}{2}$ inch). Transfer the contents of the dropper to the slide, making 4 drops, completely emptying it. Repeat this procedure until the slide is uniformly covered with drops of the mixture, then place the slide in an air bath until dry. When cool, add stain as specified to the dried fibers and press down on them a second slide or large cover glass. Remove excess moisture from the edges of the slides with absorbent paper. Prepare three slides in this manner."

(3) Short Method

(Preparation of Samples)

The small representative portions of paper are boiled for about a minute in a 0.5-percent solution of sodium hydroxide. After rinsing the paper with water it is rolled between the fingers into a ball sufficiently to loosen the fibers somewhat. This ball of pulp is dropped into a small beaker half full of water and stirred with a mechanical stirrer for several minutes or as long as is necessary to separate completely the individual fibers. A pipette with a rubber bulb at one end and with the discharging end left unconstricted is used to remove a portion of the fibers and to place them on a slide. The excess water is removed by the use of hard blotting paper. Then the fibers are treated according to the directions of the test selected.

a. Comments on General Methods for Preparation of Paper Samples

In order to examine the fibers in paper, coatings and sizings must be removed and the paper reduced to a pulp. The procedures given in the Federal specification (1) and in the specifications of the Technical Association of the Pulp and Paper Industry (2) are generally accepted in the trade. The short method (3) has been developed in this laboratory in order to save time, especially when large num-

bers of samples are to be analyzed. There is no opportunity for small fibers to be lost and thus affect the representative character of the material on the slide; thus the removal of the fibers with needles in method 1 might have a selective effect by retaining only the longer fibers; and some fibers of narrow diameter might conceivably slip through the meshes of the sieve used in method 2. When more than one kind of fiber is present the stirring produces as nearly homogeneous a mixture as possible for the final examination. In our experience, the results are as satisfactory as those obtained by the other methods.

(b) QUANTITATIVE ANALYSIS

(4) *Federal Specification Method* [7]

"The prepared slide shall be examined by means of the microscope, the slide being moved systematically so that the whole slide is covered. The percentage fiber determination shall be made only by thorough trained analysts familiar with the fiber analysis of paper. The determination of percentage of pulp shall be made by a recognized standard method and at least 300 fibers counted. Report the determined percentage of each kind of fiber on the basis of 100 percent fiber."

(5) *TAPPI Method* [45]

(Quantitative Analysis)

Apparatus:

"1. Microscope: A microscope capable of giving not less than 100 diameters magnification is necessary for the determination of fiber composition. It is desirable that the microscope be of the compound type and that it be equipped with a mechanical stage.

"2. Cross-Line Disk: This disk is employed to establish a dot or point for counting the fibers passing under it. The disk may be obtained in two ways:

"(a) It is possible to construct a satisfactory cross-line disk by the use of two silk fibers, approximately 0.01 mm in diameter obtained by untwisting a silk thread. The fibers are drawn across the center of a round cover glass 18 mm in diameter at right angles to each other. They are cemented to the edges of the glass with paraffin or other adhesive and the protruding ends cut off. The cover glass is then placed, with the fibers on the under side, on the diaphragm of the microscope eyepiece. The cover glass is then centered on the diaphragm of the eyepiece of the microscope and cemented in this position with a drop of paraffin.

"(b) A graticule or cross-line disk is carried in stock by many manufacturers of optical equipment. It consists of a round thick disk of optical glass on which are engraved two lines at right angles to each other, both lines running completely across the disk and intersecting at the center.

"3. Dropper: This shall consist of a glass tube 6 inches (20 cm) long and $\frac{1}{4}$ inch (6mm) internal diameter, fitted at one end with a rubber bulb and having the other end carefully smoothed but not constricted.

"The prepared slides shall be examined microscopically, using a magnification of not less than 100 diameters. Place the stained slide on the mechanical stage of the microscope, and count the fibers at

various points in a straight line, twice lengthwise and four times crosswise of the slide, starting each line of observation at a different point. The number of each kind of fiber present in at least 25 different fields and a total of not less than 600 fibers, 200 on each slide, shall be counted.

"Place a cross-line disk on the diaphragm of the eyepiece of the microscope. As each fiber passes under the dot or point formed by the intersecting lines on the disk, count it as one regardless of its size. If aggregations of fibers such as occur in ground wood are encountered, the number of single fibers in the aggregation shall be estimated and counted as if the fibers were completely separated.

"The proportion of the various fibers found shall be reported in terms of percentages of the total fiber composition, to the nearest 5 percent. The following nomenclature which covers the fibers commonly dealt with shall be used in reporting results. Chemical wood; chemical deciduous wood; chemical coniferous wood; ground wood; manila; jute; rag; linen; cotton; esparto; straw."

b. Comments on Quantitative Analysis

The percentage composition of paper obtained by the methods given above is based on a count of the individual fibers. The findings are not always in strict accord with the results of the ordinary chemical methods.

Some microanalysts prefer to use another method also given in the specifications of the Technical Association of the Pulp and Paper Industry: "Use the diameter of the field as observed through the microscope as a unit of measure, and give each fiber counted a value proportionate to this unit." However, the introductory statement of the counting method (2) in the same publication, states that it is "designed to eliminate the undesirable personal equation in the estimation of relative sizes of fibers." We believe method 5, as given above, to be the better.

(c) GENERAL STAINS

(6) *Herzberg's Stain, Federal Specification [7]*

Reagents:

"A. An aqueous solution of C. P. zinc chloride saturated at 70° F.

"B. 0.25 gram C. P. iodine and 5.25 grams C. P. potassium iodide dissolved in 12.5 cubic centimeters of distilled water.

"Mix 25 cubic centimeters of solution A, measured at 70° F, with solution B. Pour into a narrow cylinder and allow to stand until clear. Decant the supernatant liquid into an amber-colored, glass stoppered bottle and add a small piece of iodine to the solution. Thoroughly moisten the fibers with this solution and remove the excess with blotting paper. The solution should be tested with known fibers and readjusted if necessary by addition of either zinc chloride or iodine."

Application:

Excess moisture is removed from the fibers by means of hard blotting paper; then the fibers are mounted in the reagent.

Results:

"The following colors are developed by this stain:

"Red—linen, cotton, bleached manila hemp.

"Blue—chemically prepared fibers low in lignocellulose, from wood, straw, and esparto.

"Yellow—fibers high in lignocellulose such as ground wood, jute, and unbleached manila hemp."

(7) *Herzberg's Stain, Merritt's Modification* [33]

(General Stain)

Reagents:

"A. 50 g of dry zinc chloride (fused sticks), 25 cc distilled water added with a 25 cc pipette in the zinc chloride bottle, stoppered and shaken. Should be about 40 cc of solution.

"Take the specific gravity at 28° C. If the specific gravity is not 1.8 add distilled water in 1 cc pipette until the specific gravity is 1.8, then pour into a tall cylinder.

"B. Take part of 12.5 cc of the distilled water to rinse the thermometer, the hydrometer and original zinc chloride container and add to solution A. Dissolve 5.25 g of potassium iodide and 0.25 g iodine in the balance of the water.

"Add B to A, stir well and place in the dark. The following day pipette off the clear portion into a black bottle, leaving 3 or 4 cc of the solution above the sediment. Add a leaf of iodine.

"This stain will be found to be satisfactory for at least two weeks if it is to be used for routine analysis where color differentiations are important. Weights and measures must be observed carefully as Herzberg advised in his directions."

Application:

Excess moisture is removed from the fibers by means of hard blotting paper; then the fibers are mounted in the reagent.

Results:

"A correct stain should give the following colors: rag, cotton, linen, and hemp, wine red or brownish pink; chemical wood, bleached straw, and jute, colored dark blue; mechanical wood fibers, unbleached jute, and straw generally a lemon yellow; esparto bluish or reddish; manila almost any shade from blue to yellow; adansonias the same color reaction as chemical wood. The differentiation of various fibers as found in paper depends on the characteristic markings of the fibers as well as color differences which are an aid but not sufficient in themselves."

(8) *Sutermeister's Stain* [15, 42, 45]

(General Stain)

Reagents:

"A. 1.3 g iodine and 1.8 g potassium iodide in 100 cc water.

"B. A clear practically saturated solution of calcium chloride."

Application:

"Apply solution A after moistening the fibers with water, allow to remain about 1 minute, remove the excess by blotting and then add solution B."

Results:

"Red or brownish red—cotton, linen, hemp, ramie.

"Dark blue—bleached soda pulps from deciduous woods.

"Bluish or reddish violet—bleached sulfite fibers and thoroughly cooked part of unbleached sulfite fibers.

"Greenish—jute, manila hemp and the more lignified fibers in unbleached sulfite.

"Yellow—ground wood."

c. Comments on General Stains

Herzberg's stain is probably the most widely used general stain for paper analysis, because it serves to identify at once a number of the most common fibers, rag, chemical wood, and ground wood. On occasions when the colors produced by this reagent are too dark, Sutermeister's stain is advantageous, because it imparts lighter colors to the fibers.

Herzberg's stain is preferably made up according to Merritt's modification. This solution is more readily prepared by adjusting the specific gravity than by gaging the composition by colors obtained on known fibers. The stain is uniform and reproducible, the error due to the hygroscopic nature of solid zinc chloride being minimized.

According to Merritt [33], the stain will give good results for routine tests for at least 2 weeks. Minnear and Withrow [35] report that the Herzberg stain has a "life" of more than 1 year. We have been able to confirm this statement in this laboratory.

(d) BLEACHED AND UNBLEACHED FIBERS

(9) *Bright's Stain, Federal Specification* [3, 7]

Reagents:

"A. 2.7 g ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) per 100 cc distilled water.

"B. 3.29 g potassium ferricyanide ($\text{K}_3\text{Fe}(\text{CN})_6$) per 100 cc distilled water.

"C. 3 g of crude (not treated with sodium carbonate) substantive red dye per 500 cc of distilled water. The dye used shall be Benzopurpurine 4B concentrated. These solutions must all be made with cold water.

"Filter solutions A and B and keep in separate stock bottles at temperature not exceeding 20° C. Make solution C fresh each day it is used."

Application:

"For staining use tall narrow beakers, suspending the microscopic slides in the beakers from clamps. Mix 10 cc each of solutions A and B in one beaker and add an equivalent amount of solution C in another beaker. Set the beakers in a water bath, the temperature of which must be maintained constantly within plus or minus 1° of 20° C. Place a thermometer in the stains. When their temperature is 20° C dip the slide containing the dry fibers in distilled water to moisten it uniformly (so that no air bubbles will be formed when it is stained), then place the slide in stain (A-B) and allow it to remain 20 minutes. Wash by dipping in distilled water six times. Then renew the water and repeat the washing process. Dry the contents of the slide and repeat the process of moistening, washing, and drying, using the C stain."

Results:

"The colors developed by the Bright stain are:

"Red—bleached fibers or fibers practically free from lignocellulose.

"Blue—unbleached fibers or fibers containing lignocellulose."

(10) *Bright's Stain, Kantrowitz and Simmons' Modification* [24]

(Bleached and Unbleached Fibers)

Reagents:

"A. $N/10$ $FeCl_3$ (ferric chloride): dissolve 2.7 g $FeCl_3 \cdot 6H_2O$ in 100 cc distilled water.

"B. $N/10$ $K_3Fe(CN)_6$ (potassium ferricyanide): dissolve 3.29 g of this salt in 100 cc distilled water.

"C. 0.5 g benzopurpurine 4B crude in 100 cc of 50% ethyl alcohol. Warm the solution until the dye is completely dissolved."

Application:

"A few drops of the fibers are drawn from the test tube by means of dissecting needles, placed on a glass slide and the excess moisture is absorbed with a small piece of blotting paper. Two or three drops of solution "A" are placed on the fibers and then followed by an equal number of drops of solution "B". The mixture is stirred and the fibers are teased apart and then allowed to remain in contact with the stain for 1 minute. The excess stain is blotted up and two or three drops of solution "C" applied and the fibers again teased apart. The fibers are allowed to remain in contact with this stain for about 2 minutes. The excess stain is removed by blotting and then washing the fibers once with distilled water. The fibers are mounted in distilled water and a second slide applied as a cover glass. The amounts of bleached and unbleached fibers are determined by the official count method. The time required for staining is approximately 5 minutes."

Results:

The colors developed by the Bright stain [45] are:

"Red—bleached fibers or fibers practically free from lignocellulose.

"Blue—unbleached fibers or fibers containing lignocellulose."

(11) *Klemm's Malachite Green Stain* [15, p. 187; 26; 27]

(Bleached and Unbleached Fibers)

Reagents:

"A 2% solution of acetic acid is saturated with malachite green" (presumably Colour Index No. 657).

Application:

"Leave the fibers in the malachite green solution on the slide for about 1 minute. Wash off the excess dye thoroughly."

Results:

"Unbleached wood fibers whether prepared by the sulfite or the sulfate process become stained a striking deep green. On the other hand, thoroughly bleached fibers remain almost colorless. Partially bleached fibers assume intermediate shades."

d. Comments on Bleached and Unbleached Fibers

Bright's staining method, when carried out according to the directions in the Federal specification, consumes about an hour's time; whereas, the Kantrowitz and Simmons' modified method requires only about 5 minutes. This is brought about largely by the use of alcohol instead of water to dissolve the benzopurpurin, which makes it possible to greatly increase the amount of dye in solution. We

have found the colors obtained in both cases to be entirely comparable.

Klemm's malachite green stain is a very sensitive reagent which indicates, by the depth of green (value of color) imparted, the degree of lignification of the fibers. It is thus possible to gain an idea of the degree of delignification effected either by the method of cooking or by bleaching. This stain has the advantage of simplicity since only one solution is used; but for the ordinary routine determination of percentages of fibers in mixtures Bright's stain facilitates counting because of the distinctive colors, red and blue, obtained.

(e) CONIFEROUS AND DECIDUOUS WOOD FIBERS

(12) *Alexander's Stain* [4, 5]

Reagents:

"A. 0.2 g congo red" (presumably Colour Index No. 370). 300 cc distilled water.

"B. 100 g calcium nitrate. 50 cc distilled water.

"C. Herzberg's Stain."

Application:

"The sample to be tested is first stained with 2 drops of the congo red solution for 1 minute after which the excess dye is carefully blotted off and the sample allowed to dry.

"The dry stained sample is then immersed in 3 drops of the calcium nitrate solution and allowed to remain for 1 minute. At the end of this time 1 drop of the Herzberg stain is added, the whole quickly and thoroughly mixed and the cover glass placed on.

"The slide may be examined immediately but there seems to be a slightly stronger tone to the colors if it is let stand for 3 or 4 minutes before examination."

Results:

The coniferous fibers should be stained a uniform pink and the deciduous fibers a deep blue.

(13) *Alexander's Stain, Korn's Modification* [7, 28]

(Coniferous and Deciduous Wood Fibers)

Reagents:

"A. 100 g calcium nitrate in 25 cc water.

"B. chlorzinc iodide solution" (Herzberg's solution may be used).

Application:

"The fibers are floated in 3 drops of the calcium nitrate solution for one minute. Then 1 drop of chlorzinc iodide solution is added. After a few minutes the characteristic colors are established."

Results:

The fibers of coniferous wood turn pink, whereas the fibers of deciduous wood turn blue.

Remarks:

Grünsteidl calls attention to the fact that certain coniferous pulps prepared by the soda process do not give the true color. This is also mentioned by Korn. The latter also points out that the stain does not always yield reliable results with soft cooked sulfate coniferous pulps. With these exceptions, however, Alexander's stain is useful.

e. Comments on Coniferous and Deciduous Wood Fibers

Mixtures of coniferous and deciduous wood pulps are used in book papers, postage-stamp paper, and blotting paper; and at times it is desirable to know the relative amounts of each present. Alexander's stain has been found to be suitable. Korn's modification is simpler than the original method because in it the first step, the treatment with congo red, is omitted; nevertheless, the color differentiation is achieved. If artificial illumination without a daylight glass filter is used the original stain may offer some advantage, since the coniferous fibers then assume a slightly deeper pink color.

It is to be noted that the stain distinguishes between coniferous and deciduous wood pulps regardless of the cooking or bleaching treatment, with the rare exceptions noted under Korn's modification.

(f) UNBLEACHED SULFATE (KRAFT) AND UNBLEACHED SULFITE FIBERS

(14) Lofton-Merritt's Stain [7, 31]

Reagents:

"A. Malachite green, 2 grams; distilled water, 100 cubic centimeters.

"B. Basic fuchsin, 1 gram; distilled water 100 cubic centimeters.

"C. 0.1-percent hydrochloric acid."

"These shall be mixed in the proportion of 1 part A to 2 parts B. As dyes from different sources vary, it is necessary to test them by staining known fibers. * * * If any purple fibers appear in unbleached sulfate fibers, this indicates there is too much fuchsin present, and more malachite green solution must be added. The opposite is indicated if some unbleached sulfite fibers develop a green or blue color."

Application:

"Add the compound stain to the fibers and allow to remain 2 minutes. Remove excess stain by means of a hard blotting paper and add a few drops of 0.1-percent hydrochloric acid. After about 30 seconds remove excess of acid. Next, add a few drops of distilled water and remove the excess."

Results:

"Unbleached sulfate fibers are stained blue or blue green and unbleached sulfite fibers purple or lavender."

(g) MITSUMATA AND GAMPI

(15) Sodium Hydroxide [14, p. 11, 12; 22]

Reagent:

"17.5 percent aqueous sodium hydroxide solution."

Application:

"The fibers are mounted in the sodium hydroxide solution and observed under the microscope."

Results:

The swelling causes mitsumata to show a bead-like structure. Gampi fibers do not exhibit this characteristic.

2. TEXTILE FIBERS

(a) GENERAL METHODS

(16) Preparation of Samples

For the examination of a sample whose components are obviously different, it is only necessary, of course, to pull out a few fibers from

each portion. But when the whole has been dyed a solid color, it may be advisable to examine yarns of the warp and the filling separately, the pile or nap, if present, and to search for tufts woven in or applied in some other manner. Some of the latest textures are produced by using yarns composed of strands of different fibers or even by mixing fibers within a single strand.

If the color of a material is so deep as to obscure the characteristics of the fibers or to interfere with a staining reaction, it is necessary to decolorize before proceeding. Various methods will probably have to be tried because the dyes vary in their constitution. Some dyes may be removed by boiling the material in water; others may be removed by treating it with either cold or warm dilute acid or alkali (1 percent). Sodium hyposulfite³ is generally effective in discharging the color in textiles. Yet there are wool textiles in which the dye is held so tenaciously that the fiber is apt to be destroyed before the dye is released. Warming such material in a solution consisting of one volume of 20-percent titanous chloride (TiCl_3) and two volumes of concentrated hydrochloric acid usually achieves the desired results. The fibers must be washed thoroughly, of course, to remove all traces of bleaching agents, before the stains are applied.

When cross sections of fibers are to be examined, sections may be cut (by hand) in any simple manner, employing pith or cork. Hardy's [13] device has been used satisfactorily with collodion, cellulose acetate, or paraffin applied to hold the cross sections in position.

(17) Ignition

(General Test)

In the absence of a microscope, or for an occasional confirmation of microscopic findings, indications of the nature of the fibers may be obtained by burning, as shown in Table 1. A strand of similar fibers, or one fiber at a time in case a mixture is suspected, is held over a small flame. It is to be noted that various treatments, such as a casein finish, rubber impregnation, or pigmentation may affect the observations. Thus, heavily weighted silk leaves a residual ash in the form of the fiber.

TABLE 1.—*Characteristics of fibers when burned*

Rate of burning	Odor	Ash	Fiber
None.....	None.....	None.....	Asbestos. Glass.
Slow.....	Burning feathers.....	Black knob.....	Animal fibers. Casein.
Rapid.....	Burning paper.....	Little or none.....	Vegetable fibers. Rayons (except cellulose acetate).
Do.....	Acrid.....	Black knob.....	Acetate rayon.

(b) ANIMAL FIBERS (GENERAL REAGENTS)

(18) *Millon's Reagent* [1, p. 34, 36; 34; 37]

Reagent:

To 25 g of mercury add 25 g of concentrated nitric acid under a hood. Upon completion of the reaction dilute the solution by adding enough water to double the volume.

³ Sodium hyposulfite ($\text{Na}_2\text{S}_2\text{O}_4$) is known in the trade as "hydrosulfite."

Application:

The reagent is applied to the fibers; and after about 2 minutes the slide is examined.

Results:

Animal fibers assume a pink to red color. Upon standing the color deepens. Fibers of vegetable origin do not show any color.

(19) *Picric Acid* [11, p. 124]

(Animal Fibers—General Reagent)

Reagent:

1 g of picric acid in 100 ml of water.

Application:

The fibers are placed in a few drops of the reagent on a slide, left for about 3 minutes, and then washed with water.

Results:

Animal fibers are stained brilliant yellow, whereas vegetable fibers remain unstained.

b. Comments on Animal Fibers (General Reagents)

Millon's method is the simpler of the test methods given here, since the solution acts as a mounting medium as well as a reagent. Picric acid may be preferred when the dye cannot be completely removed and the result is in doubt.

(c) CASEIN FIBERS

(20) *Benzopurpurin* [2]

Reagents:

A saturated solution of benzopurpurin (presumably Colour Index No. 448) in water is prepared.

Application:

The fibers are permitted to remain in the solution for some time and are then washed.

Result:

Casein fibers become red.

(21) *Indigo Carmine* [18]

(Casein Fibers)

Reagent:

A saturated solution of indigo carmine (presumably Colour Index No. 1180) is made in water acidified with sulfuric acid.

Application:

The fibers are left in the solution for several minutes. After washing they are examined in water.

Result:

Casein fibers are stained bright blue.

Remarks:

It should be pointed out that Herzog reported the indigo carmine method only for the identification of damaged wool. However, it was found that this reagent also stains casein fibers.

c. Comments on Casein Fibers

Synthetic fibers made from casein, an animal product, are now on the market. Either of the two tests given for casein may be used, the choice of stain probably being indicated by the colors already present in the fabric. Silk and casein fibers assume similar colors but are readily distinguished by their morphological features.

(d) CORDAGE FIBERS

(22) *Abacá (Manila) and Nonabacá (Nonmanila) Fibers—Swett's Test*
[8, 43, 44]

Reagents:

Ether

Alcohol

Bleaching-powder solution

Acetic acid

Ammonia

"Bleaching powder (35 percent) shall be worked into a thick paste with water, and about six times the resultant volume of water added. The solution shall be allowed to settle about 12 hours and the clear liquid drawn off in a dark-colored glass bottle. Before using, it shall be acidified with acetic acid. A strong bleaching powder should be used since the success of this test depends upon having an excess of available chlorine present for the chlorination of the fibers."

Application:

"The specimen shall be separated into strands and the oil rinsed out with either ether or alcohol. The excess of solvent shall be evaporated by waving in the air. The fibers shall then be immersed in bleaching-powder solution * * * for 30 seconds, rinsed in water, shaken, rinsed in alcohol, and again shaken until nearly dry." They are then exposed to ammonia fumes.

Results:

The fibers are stained a characteristic color, abacá (manila) "fibers are colored brown, all adulterant fibers cherry red."

Remarks:

"This test, with careful manipulation, gives quantitative results. Considerable care is required and it is always best to run blanks on abacá and nonabacá (manila and nonmanila) to test the solution. Prolonged exposure to ammonia fumes leads to loss of the red color and should be avoided. In the case of mixed fibers, only one end of the strand should be treated, after which the different kinds of fibers may be sorted. The opposite ends of these fibers are then treated as a check upon the sorting, after which the fibers can be counted. It is important that the alcohol and ammonia be full strength and not weakened by prolonged exposure to the air."

(23) *Abacá (Manila), Coconut, and Sisal*—Ash [14, p. 174-177; 20; 46; 47; p. 405-406]

Procedure:

The fibers are ignited in a small uncovered porcelain crucible over a small flame. The resulting ash is mounted in aniline oil or phenol on a slide.

Results:

A. *Abacá*: The stegmata, or silicified cells, appear in the ash as rows of rectangular hollow bodies.

B. *Coconut*: The ash of coconut fibers yields siliceous globules of various sizes. These globules have the appearance of possessing a foamy inner structure.

C. *Sisal*: In the ash there are no siliceous skeletons; but some elongated aggregates of calcareous material are present which are characteristic.

Remarks:

If the ash is dropped into a 20-percent solution of hydrochloric acid or chromic acid the siliceous stegmata of *abacá* and of coconut are unaffected, and more readily seen because of the destruction of other parts of the ash. But the pseudomorphs after calcium oxalate in the ash obtained from *sisal* are dissolved by such treatment. When the moistened ash is treated with sulfuric acid the presence of calcium is demonstrated by the formation of needles of calcium sulfate.

(24) *Abacá (Manila) and Coconut—Stegmata Stain* [41]

Reagents:

A. 2-percent solution of potassium ferrocyanide ($K_4Fe(CN)_6$).

B. 10-percent solution of hydrochloric acid.

Application:

Ignite the sample in a porcelain crucible over a small flame. The ash is dropped into the potassium ferrocyanide solution on a slide. Hydrochloric acid is added.

Results:

The stegmata of certain plants assume a blue color. The round stegmata of coconut usually turn distinctly blue. In some samples of *abacá* the stegmata turn light blue, facilitating their detection in the ash.

(25) *Jute—Phloroglucinol* [14, p. 115, 118]

Reagents:

A. 1 g of phloroglucinol dissolved in 80 ml of alcohol.

B. concentrated hydrochloric acid.

Application:

The fibers are placed in a drop of solution A. Then a drop of solution B is added.

Result:

Unbleached jute fibers turn dark violet-red, because they are heavily lignified.

Remarks:

The phloroglucinol solution stains the lignified portions of other fibers, such as phormium and ground wood; but jute is readily identified by a consideration of the morphological characteristics in conjunction with its exceptionally heavy lignification.

(c) COTTON, MERCERIZED

(26) *Cuprammonium Solution*. [14, p 113, 115, 137-148; 38]

Reagent:

2 g of cupric hydroxide ($\text{Cu}(\text{OH})_2$).

100 ml of 25-percent ammonia.

Add a few drops of ruthenium red.

Keep in a black bottle.

Application:

The fibers are mounted in a few drops of solution and the process of swelling is observed under the microscope.

Results:

During swelling ordinary cotton exhibits marked ballooning, the constrictions occurring between the balloons where the resistant cuticle is shoved together. The balloons appear light blue, the cuticular remnants deep pink.

On the other hand mercerized cotton exhibits no such ballooning, since the cuticle has been attacked by the mercerizing process. There is an almost uniform swelling along the whole length of the fiber.

Remarks:

The individual fibers vary in their rate of swelling, as is generally the case in such reagents, so that a quantitative differentiation would prove impractical.

(27) *Hübner's Reagent* [21, 29]

(Mercerized Cotton)

Reagent:

20 g of iodine.

100 ml of a saturated solution of potassium iodide in water.

Application:

The fibers are immersed in the solution for a few seconds. They are then washed several times.

Results:

Unmercerized cotton is stained only slightly, whereas mercerized cotton becomes black or deep indigo.

e. Comments on Cotton Fibers

The color differentiation between mercerized and unmercerized cotton by means of Hübner's reagent is good, providing the fibers can be previously properly decolorized.

Although Hübner claims that the degree of mercerization of cotton can be determined, this does not appear to be generally accepted. It would seem that ordinarily only general indications can be obtained by this method.

(f) COTTON, OLD AND NEW

(28) *Ultraviolet Light* [48]*Procedure:*

"A mercury quartz arc lamp was used as the source of the ultra-violet radiations and a * * * glass filter * * * was found to be the most suitable sort for this work. A filter which allows the passage of rays from 4,047 to 2,900 Angstrom units gave the best results.

"As the range of the visible spectrum extends from about 7,000 to 3,900 Angstrom units, it will be noted that this filter allows a very small amount of the visible violet light to pass through."

Results:

New cotton fibers exhibit a violet fluorescence. "As the cotton ages the fluorescence turns from a violet to an ivory-white or brownish-white color."

Remarks:

It is advisable to have known samples of new and old cotton on hand for comparison.

"While examination of samples of material directly in the ultra-violet rays is sometimes conclusive, particularly in the case of material which is either all new or all old, this method alone should not be depended upon for correct results if new and old materials are mixed as by garneting, but tests should be conducted * * * for determination of the presence of oxycellulose, and for damage to the fibers by heat, fungi, or by mechanical means."

(g) FLAX AND HEMP

(29) *Cross Sections* [17]*Reagent and Preparation:*

Cross sections of the fibers are placed on a slide and treated with a solution of ruthenium red (1:10 000). The sections are washed after 5 or 10 minutes and examined under the microscope.

Results:

The middle lamellae and the cell contents are stained red. In flax the cell contents are apt to be the more prominently stained; in hemp, the middle lamellae. Cross sections of hemp fibers tend to be somewhat flattened and have an elongated lumen with projecting points, whereas sections of flax fibers tend toward a more nearly circular lumen and cell wall.

Remarks:

Cross sections of bundles of fibers exhibit the characteristics with sufficient clarity to be valuable diagnostically; but individual fibers vary.

(30) *Cuprammonium Solution* [14, p. 150-154; 38; 47, p. 544, 545, 555-558]

(Flax and Hemp)

Reagent:

2 g of cupric hydroxide $[\text{Cu}(\text{OH})_2]$.

100 ml of 25-percent ammonium hydroxide solution.

Keep in a dark bottle.

Application:

Fibers placed in a drop of the reagent should be immediately observed under the microscope in order to follow the stages of swelling.

Results:

As the flax fibers swell the protoplasmic remnants become prominent in the tortuous narrow central tube, whereas the inner canal of hemp exhibits prominent horizontal striations.

Remarks:

The test is a qualitative rather than a quantitative one owing to the fact that swelling takes place at different rates in the individual fibers; and moreover not all fibers exhibit the distinguishing characteristics.

If the fibers are highly bleached this test may be of no avail.

The swelling process may be arrested by substituting a glycerin solution (1 volume each of glycerin, alcohol, and water) for the cuprammonium solution.

(31) Cyanin [19]

(Flax and Hemp)

Reagent:

A saturated solution of cyanin (presumably Colour Index No. 806) is prepared at ordinary temperature. This is diluted somewhat with water. Then a third of its volume of glycerin is added.

The fibers are macerated by boiling in a 1-percent solution of sodium hydroxide and then thoroughly washed.

Application:

The fibers are heated in the reagent on a slide. The mounting medium is concentrated glycerin.

Results:

The flax fibers remain colorless, while the hemp fibers assume a greenish-blue color because the middle lamella is slightly lignified.

Remarks:

After staining the fibers, it is well to wash them carefully in a glycerin solution (1 volume each of glycerin, water, and alcohol).

(32) Moisture Test [39]

(Flax and Hemp)

Procedure and Results:

A very simple test is performed by wetting a fiber, by drawing the fiber between moist finger tips. Holding the fiber in a vertical position and viewing the tip of the fiber from above, flax may be seen to move in a clockwise direction, whereas hemp may move in either a clockwise or a counterclockwise direction, usually the latter. The motion is much weaker in hemp than in flax.

(33) Potassium Dichromate [12]

(Flax and Hemp)

Reagent:

Potassium dichromate is dissolved in an excess of sulfuric acid and then diluted somewhat with water.

Application:

The fibers are immersed in the reagent on a slide and placed at once under the microscope in order to observe the swelling.

Results:

Flax swells somewhat more readily than hemp. The central canal of flax is quite wavy, whereas that of hemp is practically straight.

Remarks:

This test is to be considered as qualitative, since the fibers swell at different rates and not all the fibers exhibit the distinguishing characteristics clearly.

(g) *Comments on Flax and Hemp*

Flax and hemp are so similar in structure and in chemical composition that positive identification is difficult; hence all available tests have been included here in order to provide independent methods of approach. Should a mixture of the two fibers occur, a quantitative analysis would probably be impossible at the present time. Usually, however, only one or the other fiber is encountered in a single sample.

The moisture test is a simple one, not requiring the use of a microscope. It is frequently used as a check.

The cross-section method is only serviceable when the fibers are in the form of bundles. Conclusions must be drawn from the appearance of the bundles as a whole, not from detached single fibers.

Swelling in cuprammonium solution has proved the clearest means of distinguishing these two fibers. But it has been reported that if the fibers have been highly bleached this method is unreliable. Potassium dichromate may be used interchangeably with cuprammonium solution.

The cyanin test is based on the presence of traces of lignin in hemp fibers and its absence in flax fibers. These differences are slight and not always easy to evaluate. Careful distinction should be made between the lignin in the middle lamellae of hemp fibers and the lignified shives in incompletely cleaned flax.

(h) **RAMIE**(34) *Ash* [36]*Procedure:*

The fibers are ashed conveniently by placing them in a porcelain crucible over a low flame until they have turned light gray or white. The ash is mounted in aniline.

Results:

Characteristic numerous spherical cystoliths with spicules are prominent.

Remarks:

Abacá (manila) also has cystoliths, which are, however, smaller. In addition abacá has characteristic rows of stegmata. Flax has no cystoliths or stegmata.

(i) **RAYONS**(35) *General Test.*—Cross Sections [1, p. 221–231; 14, p. 187–190; 40]

(Rayon)

Procedure:

Cross sections may be obtained by cutting (by hand) in any convenient manner.

Results:

Cross sections of viscose rayon fibers have irregular serrated edges. Those of cuprammonium rayon fibers are smooth and almost circular. Cellulose acetate and nitrocellulose rayon fibers have relatively smooth circumferences with a few indentations.

Remarks:

This method is of value in assisting in the identification of the rayon fibers generally on the market at present. Photomicrographs are given in the references. Some of the possible variations in the appearance of cross sections are shown in Heermann and Herzog's book [14].

(36) *General Test—Polarized Light* [14, p. 91]

(Rayon)

Procedure:

Mount the fibers in water and observe between crossed nicols.

Results:

The colors obtained are as follows (depending on the thickness of the fiber):

Viscose.....	White I to green II.
Cuprammonium.....	Light gray I to sky blue II.
Acetate.....	Gray I.
Nitrocellulose.....	Light yellow I to yellow II.

Remarks:

The authors have reported their observations in the customary terms employed in describing polarization colors.

(37) *Acetate—Acetone* [1, p. 36]

(Rayon)

Reagent:

Acetone.

Application:

The fibers are observed under a microscope while acetone is added at the edge of the cover slip.

Results:

Acetate rayon fibers swell and dissolve readily. Other fibers are not affected.

(38) *Acetate—Acidulated Iodine* [25]

(Rayon)

Reagent:

0.015 g of iodine is dissolved in 1,000 ml of a 5-percent solution of potassium iodide.

To this one or two drops of acetic acid are added.

Application:

The fibers are left for a time in the reagent. They are observed after washing.

Results:

Acetate rayon is bright yellow. Other rayons are either colorless or almost so.

(39) *Acetate and Cuprammonium—Hahn's Stain—Picric Acid and Soluble Blue* [10]

(Rayons)

Reagent:

A solution is prepared containing 1 percent of picric acid and 0.2 percent of "soluble blue 2B Extra" (presumably Colour Index No. 702).

Application:

"The sample is dipped for three minutes in a cold or lukewarm solution." It is then "washed well with cold water."

Results:

Acetate rayon fibers are stained strongly yellow. Cuprammonium rayon fibers are stained strongly blue. Viscose and nitrocellulose rayons remain colorless.

(40) *Cuprammonium—Erie Fast Orange* [10; 1, p. 36]

(Rayon)

Reagent:

A 0.2-percent solution of Erie fast orange *CG* (presumably Colour Index No. 621) in water is prepared.

Application:

The fibers are mounted on a slide in a few drops of the stain. After leaving them in the cold or lukewarm solution for 3 minutes, they are carefully washed and mounted in water for examination.

Results:

Cuprammonium rayon stains orange. Viscose and acetate rayons are colorless. Nitrocellulose rayon is pale orange.

Remarks:

If the reagent containing the fibers is heated to the boiling point, the resultant color is deeper.

(41) *Cuprammonium—Hahn's Stain—Soluble Blue and Eosin* [10]

(Rayon)

Reagent:

0.2 g of soluble blue 2B extra (presumably Colour Index No. 707).

0.1 g of eosin *J* (presumably Colour Index No. 768).

1 g of tannic acid.

These three materials are dissolved in 100 ml of hot water. After cooling, 0.2 ml of 10-percent hydrochloric acid is stirred in.

Application:

"The sample is dipped for 3 minutes in the cold solution and washed well with cold water."

Results:

Cuprammonium rayon is stained blue; viscose, acetate, and nitrocellulose rayons are stained lavender.

(42) *Nitrocellulose—Diphenylamine Solution* [1, p. 33, 36]

(Rayon)

Solution:

20 ml of concentrated sulfuric acid.

10 ml of glacial acetic acid.

0.3 g of diphenylamine.

Application:

The fibers are mounted dry on a glass slide and observed under a microscope while the reagent is added at the edge of the cover slip.

Results:

Nitrocellulose rayon fibers give a deep-blue color and dissolve. Other fibers do not exhibit this color.

(43) *Viscose—Dark Field Illumination* [14, p. 12; 16]

(Rayon)

Procedure:

The fibers are examined at a magnification of about 350 diameters by means of a dark field condenser.

Results:

Viscose rayon fibers reveal such numerous minute white particles within the fiber mass as to give a cloudy effect. In this respect the fibers of other rayons show but scattered particles, if any. Larger and more brilliantly reflecting particles may be present on or in all rayons. They are probably particles of materials used as delustrants; they are not apt to be confused with the white fine particles which characterize viscose.

(44) *Viscose and Cuprammonium—Wright's Stain* [23]

(Rayons)

Reagent:

"Precipitate a water suspension of methylene blue 2B" (presumably Colour Index No. 922) "with about one-half the quantity of eosin yellowish" (presumably Colour Index No. 768), "stirring thoroughly to insure the completion of the reaction, separating the precipitate from the liquor by centrifugal force, followed by decantation, or by filtering (plain or with a Büchner funnel and suction flask)."

Application:

"Employing a white or light colored, *dry* sample of fiber for identification, cover it with a cold alcoholic solution of Wright's stain and bring in a few seconds just to a boil. Pour the reagent back into the bottle and wash the stained sample thoroughly with water."

Results:

Viscose rayon is stained blue; cuprammonium rayon is stained violet. Acetate rayon is also violet but partially disintegrated. Nitrocellulose rayon is stained deep blue.

Remarks:

"Rayon samples when wet with water before staining all give a violet coloration. Hence, the dry condition when making the test."

(45) *Viscose and Nitrocellulose—Silver Nitrate Solution* [30]

(Rayons)

Reagent:

The solution is "composed of 1 percent silver nitrate, 4 percent sodium thiosulfate, and 4 percent sodium hydroxide. This test solution is prepared by dissolving the silver nitrate and the sodium thiosulfate separately and adding the first solution to the second until the

cloudiness disappears. The sodium hydroxide solution is then added to this mixture, and the same brought up to the correct volume, boiled and filtered."

Application:

The sample is immersed in this solution.

Results:

Viscose rayon stains to a brown or reddish brown color. Nitrocellulose rayon also develops a brown color. Cuprammonium and acetate rayons remain colorless.

Remark:

It has been found advisable to bring the solution with the fibers in it to a boil.

(i) Comments on Rayons

Table 2 is designed as a survey of the reactions of the different rayons to the various tests. The authors of these methods have generally developed a test which would distinguish one type of rayon from all others or which would differentiate between two given types of rayon. These particular characteristics are set in *italic* in the table. It was thought that when examining unknown fibers a knowledge of the reactions of the rayons not mentioned by the authors would prove a valuable guide to direct further analysis. With this in mind, each test was applied to every type of rayon, and the other results are noted in table 2 in roman type.

TABLE 2.—Identification of rayons

Test and method number	Type of rayon			
	Cellulose acetate	Viscose	Cuprammonium	Nitrocellulose
Acetone (37).....	<i>Dissolves</i>	Unaltered.....	Unaltered.....	Unaltered.
Diphenylamine (42)....	Colorless; <i>dis-</i> <i>solves</i>	Colorless; <i>dis-</i> <i>solves</i>	Colorless; <i>dis-</i> <i>solves</i>	<i>Deep blue</i> ; <i>dis-</i> <i>solves</i>
Erie fast orange (40)....	Colorless or pale orange.....	Colorless or pale orange.....	<i>Deep orange</i>	Colorless or pale orange.....
Wright's stain (44)....	Violet (partially disintegrated). <i>Yellow</i>	<i>Blue</i>	<i>Violet</i>	Deep blue.
Hahn's; Picric acid+ soluble blue (39).....	<i>Yellow</i>	Colorless.....	<i>Blue</i>	Colorless.
Hahn's; Soluble blue+ eosin (41).....	Lavender.....	Lavender.....	<i>Blue</i>	Lavender.
Acidulated iodine (38).. Dark field illumina- tion (43).....	<i>Yellow</i> Almost transpar- ent.	Almost colorless.. <i>Innumerable very</i> <i>fine white parti-</i> <i>cles</i>	Almost colorless.. Almost transpar- ent.	Almost colorless.. Almost transpar- ent.
Silver nitrate solution (45).....	Colorless.....	<i>Brown</i>	Colorless.....	Brown.
Polarized light (36)....	<i>Silver gray</i>	Streaked.....	<i>Uniform yellow or</i> <i>green</i>	Streaked.
Cross sections (35)....	Few identations...	<i>Serrated edges</i>	<i>Circular</i>	Few indentations.

These tests are applicable to the rayons commonly on the market at present. It should be borne in mind that special treatments, such as delustering and "iron proofing", sometimes cause deviations in the results indicated. The results obtained with experimental rayons are not always reliable, possibly because of their "purity" or because of unusual treatments.

For quantitative determinations, Wright's and Hahn's stains are probably the most satisfactory.

(j) SILK "WILD"

(46) *Nickel Hydroxide Ammonia* [14, p. 115, 181]*Reagent:*

25 g of crystalline nickel sulfate are dissolved in 500 ml of water. By the addition of sodium hydroxide a precipitate is formed. This is washed and subsequently dissolved in a solution of 125 ml of concentrated ammonia and 125 ml of water.

Application:

Fibers are placed in a drop of this solution on a slide and their behavior is observed under the microscope.

Results:

Ordinary silk fibers swell and go into solution at ordinary temperatures, whereas "wild" (tussah) silk fibers remain unaffected. However, the latter will swell and begin to go into solution on warming.

(k) WOOL, DAMAGED

(47) *Indigo Carmine* [18]*Reagent:*

A saturated solution of indigo carmine (presumably Colour Index No. 1180) is made in water acidified with sulfuric acid (prepared at ordinary temperature).

Application:

The fibers are left in the reagent for a short time and then mounted in concentrated glycerin.

Results:

The damaged portions of the wool take a bright blue stain, but the undamaged portions are not affected.

Remarks:

Herzog points out that if the treatment with indigo carmine is followed by picric acid, the undamaged parts assume a yellow color, the damaged portions become green.

An examination of several samples of wool, which had been subjected to various treatments, showed that appreciable damage caused by mechanical means, alkali treatment, or exposure to light could be detected by this method.

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