

ANALYSIS OF WOOL-COTTON TEXTILES

By Ralph T. Mease and Daniel A. Jessup

ABSTRACT

The results of a study of several methods for the determination of cotton and wool in mixtures are presented. The inadequacy of some of the methods is shown, and a satisfactory procedure is described. In this method, sizing, finishing materials, and natural nonfibrous constituents of the textiles are removed by solvent extraction followed by digestion with a starch-hydrolyzing enzyme and washing. Wool is determined directly by weighing after removal of the cotton by carbonization with aluminum chloride. Cotton is determined directly by weighing after removal of the wool with potassium hydroxide. Results accurate within 1 percent of the amount of total dry fiber present are readily obtainable by the method.

CONTENTS

	Page
I. Introduction.....	75
II. Materials.....	77
III. Determination of sizing, finishing, and other nonfibrous materials..	77
IV. Removal of cotton from mixtures with wool.....	79
V. Removal of wool from mixtures with cotton.....	82
VI. Proposed method for the analysis of wool-cotton mixtures.....	83
1. Drying and weighing.....	83
2. Total sizing, finishing, and other nonfibrous materials.....	83
3. Determination of cotton cellulose.....	84
4. Determination of wool.....	84
VII. Analysis of mixtures of known composition by the proposed method..	85

I. INTRODUCTION

As a result of the trade-practice conference of the knit-underwear industry held on May 26, 1930, a ruling was adopted by the Federal Trade Commission requiring that "the word 'wool' shall not be used in any way in the labeling, advertising, merchandising, or selling of knit underwear unless the percentage by weight of wool contained in the garment is stated."¹ Further, a commercial standard for "Wool and part-wool blankets" was established by the joint action of producers, distributors, and users, and became effective April 1, 1933. It provides for the labeling of part-wool blankets with the guaranteed minimum wool content.² These standardization activities suggested the need for a precise quantitative method of analysis for part-wool textiles.

¹ Statement by the Commission, Trade Practice Conference, Knit Underwear Industry. Mimeographed. July 7, 1930. Federal Trade Commission, Washington, D. C.
² CS39-32. Wool and Part-Wool Blankets. Government Printing Office, Washington, D. C., price 5 cents.

In general, textiles made from mixtures of cotton and wool are composed of: (1) Wool fiber (protein); (2) cotton fiber (cellulose); (3) natural oils, waxes, and other constituents of the raw fibers which have not been removed completely in the course of manufacture of the textile; (4) oils, soaps, waxes, starches, clay, and other materials which have been added, collectively termed sizing and finishing materials; and (5) degraded protein and cellulose resulting from chemical deterioration of the fibers before or during processing. The materials named under (3), (4), and (5) are included in the term "nonfibrous materials" subsequently used in this paper.

The usual procedure of the analyst called upon to determine the cotton and wool content of the mixture is to remove nonfibrous material from a weighed specimen of the textile, then to remove one of the fibers by chemical means, and finally to weigh the residue. The weight of the fiber which has been removed from the mixture is then determined by difference. Sometimes allowance is made for materials which are considered to be a normal or allowable part of the fiber but which are removed by the reagents.

Two procedures are in general use for the removal of nonfibrous constituents: One consisting in successive treatments with hydrochloric acid and sodium carbonate, here designated the "scouring" method; the other consisting in a treatment with a hydrolytic enzyme, or the "malting" method. The scouring method was prescribed until recently by the American Society for Testing Materials and for the testing of U.S. Government purchases of wool-cotton textiles.^{3,4} The malting method is the tentative-standard procedure of the American Association of Textile Chemists and Colorists for the determination of sizing, loading, and finishing materials in cotton textiles.⁵ It has been shown to give more reliable results than the scouring method.⁶

Two methods are in use for the quantitative separation of the fibers after removal of nonfibrous materials. One depends upon the removal of the cotton either by solution in strong sulphuric acid or by carbonization with aluminum chloride. The other depends upon the removal of the wool by treatment with sodium- or potassium-hydroxide solution.

Few quantitative data have been published to show the effect upon wool protein or cotton cellulose of the various reagents used to effect separation of wool and cotton fibers. It is known that cotton which has been treated with hydrochloric acid loses weight in the potassium-hydroxide solution used to remove wool. It is also known that wool absorbs sulphuric acid and other substances from solutions in which it is immersed. It seemed essential, therefore, to make a quantitative study of the methods for the analysis of wool-cotton textiles in order to establish on an experimental basis a procedure satisfactory for the new demands. That is the purpose of the work reported here.

³ A.S.T.M. D276-31T. Tentative Methods for Identification of Textile Fibers and Their Quantitative Determination in Mixed Goods. Proc. Am. Soc. Testing Materials, vol. 30, part I, p. 1027, 1931.

⁴ U.S.G.M.S. no. 345a. General Specification for Textile Materials (Methods of Physical and Chemical Tests), 1929. Government Printing Office, Washington, D.C. Superseded by F.S. no. CCC-T-191, Textiles; Test Methods. Government Printing Office, Washington, D.C.

⁵ Tentative Method for Determination of Total Sizing and Finishing Materials in Textiles. 1931 Year Book Am. Assoc. Textile Chemists and Colorists, pp. 136-137. Howes Publishing Co., New York.

⁶ D. A. Clibbens and A. Geake, The Determination of Total Size or Filling in Cotton Goods. Jour. Textile Inst., vol. 22, p. T465, October 1931.

II. MATERIALS

The cotton used in most of the experiments to be described was prepared from "grey" cotton yarn, unbleached airplane cloth, and commercial sheeting, by the method of Corey and Gray for the preparation of high-grade cellulose.⁷

The cotton was extracted 6 hours in a soxhlet apparatus with ether and 16 hours with alcohol. It was allowed to dry in air at room temperature and was then extracted with a boiling 1-percent solution of sodium hydroxide. This extraction was carried out in a 500 ml erlenmeyer flask fitted with a siphon, and the extraction rate was so regulated that 10 l of solution was used in 4 hours. During the treatment the cotton was not allowed to come in contact with the air and the solution in the flask was kept boiling. After the caustic treatment the material was rinsed with water, the residual alkali was neutralized with 5 percent acetic acid, and the material was then rinsed with water until neutral to methyl red.

The wool was prepared from raw wool, commercial yarns, and cloth. The visible foreign material was removed from the raw fiber by hand. The wool was then agitated in a 1-percent solution of a neutral soap for $\frac{1}{2}$ hour at 70 C, rinsed well with distilled water at the same temperature, extracted in a soxhlet extraction apparatus with alcohol for 16 hours and then with ether for 6 hours. It was then air dried, rinsed once with distilled water at 70 C to remove residual traces of alcohol,⁸ and dried in a well-ventilated oven at 60 C for about 2 hours.

For some of the experiments, the wool and cotton yarns and cloths were used as received from the manufacturer.

III. DETERMINATION OF SIZING, FINISHING, AND OTHER NONFIBROUS MATERIALS

A consideration of the variety and ever-increasing number of materials applied to textiles in the form of sizing, filling, or coating for the purpose of improving their appearance or "feel", increasing their weight, or rendering them resistant to water, organisms, fire, etc., leads to the conclusion that no practicable method is possible for the quantitative removal of all nonfibrous materials that may be present. The analyst must adapt any standard method to the material in hand. However, for the more usual wool-cotton textiles that come to the laboratory for analysis, particularly for blankets and underwear, a satisfactory standard method seems possible.

Adequate methods for the quantitative determination of materials added to the fiber in the course of manufacture or for nonfibrous natural constituents which have not been completely removed in the processing, are still to be developed. For this reason, and also because it makes little difference to the consumer of the finished article whether the nonfibrous materials are natural or added, it is logical to consider all such substances as a group for the purpose of analysis.

Specimens weighing about 10 g each of the yarns and cloths were dried in an oven at 105 to 110 C to constant weight. They were

⁷ A. B. Corey and H. Le B. Gray, Notes on the Preparation of Standard Cellulose. *Ind. Eng. Chem.*, vol. 16, p. 853, August 1924.

⁸ R. T. Mease, Adsorption of Alcohol by Fibrous Materials. *Ind. Eng. Chem., Anal. Ed.*, vol. 5, p. 317 September 15, 1933.

then treated by the method for the removal of sizing and other non-fibrous materials, dried as before, and the change in weight determined.

The scouring method involved the following steps: (1) Extracting the fibers with ethyl ether in a soxhlet apparatus for 1 hour; (2) immersion in boiling distilled water for 10 minutes; (3) immersion in 0.5-percent solution of sodium carbonate (Na_2CO_3) at 90° to 95° C. for 30 minutes; (4) rinsing with distilled water; (5) immersion in 0.5-percent solution of hydrochloric acid for 30 minutes; (6) rinsing in distilled water; (7) rinsing in 1-percent solution of sodium carbonate; and (8) rinsing in distilled water.

In the malting method, the specimens were extracted with chloroform (some with carbon tetrachloride) in a soxhlet apparatus for 2 hours, allowed to dry in the air, and washed with hot water. They

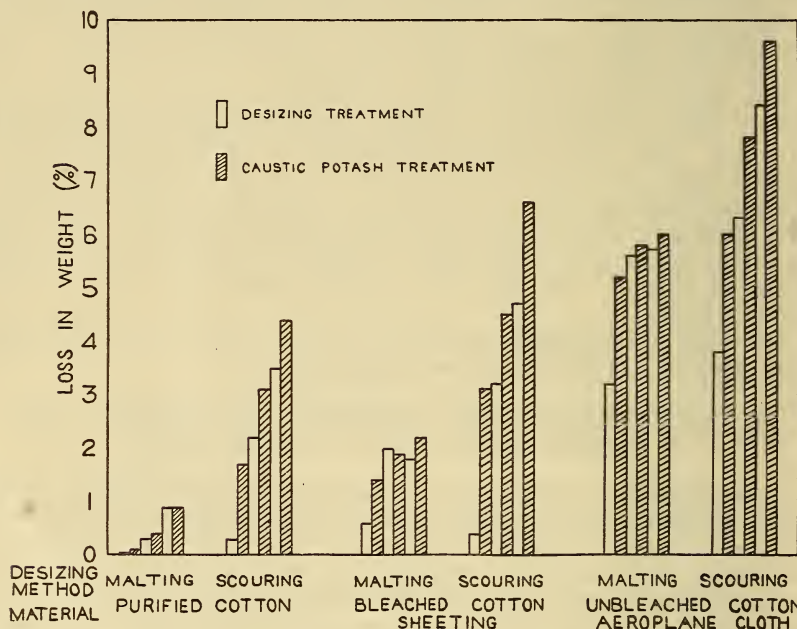


FIGURE 1.—Change in weight of cotton specimens subjected to desizing and caustic treatments.

[The loss in weight of a specimen after desizing, desizing and treatment with caustic potash, and repetition of these treatments 1 and 2 times is shown.]

were then placed in a 0.5-percent water solution of a starch-hydrolyzing enzyme at 50° C, and squeezed to remove air and insure wetting of the fibers. The solution was then heated to 70° C. and the specimens kept immersed in it at this temperature for 15 minutes.

The effects of these treatments on the weight of cotton when applied 1, 2, and 3 times in succession are shown in figure 1 and their effects on the weight of wool shown in figure 2. Each desizing treatment of the cotton was followed by a caustic-potash boil required by one method of analysis for the removal of wool. The total loss in weight of the specimens caused by the treatments is shown in figure 1. It is evident that the scouring method leaves cotton cellulose more susceptible to attack in the caustic treatment necessary to remove wool than does the malting method of desizing. The

malting method has much less effect on wool than the scouring method. Neither purified cotton nor purified wool loses more than a few tenths of a percent of its weight in a single treatment. Figure 2 shows that successive malting treatments produce a small increase in weight of wool cloth that has not been subjected to the purification process. Though the increase in weight of the commercial wool cloth and the decrease in weight of the purified wool when subjected to the malting treatment are unimportant for ordinary analytical purposes, they may be significant for other work.

IV. REMOVAL OF COTTON FROM MIXTURES WITH WOOL

One published method for the removal of cotton from mixtures with wool calls for treatment of the specimen with a solution of sulphuric acid prepared by mixing 80 ml of the concentrated laboratory reagent with 25 ml of water and weighing the wool residue after it is washed and dried.⁹ This method may be applied to the fiber mixture free from sizing and finishing materials or, if these materials are removed by the sulphuric-acid solution, it may be applied directly to a specimen of the original material. A similar method for separating cotton and rayon from wool is recommended by Kraiss and Markert.¹⁰

Though cotton is readily dissolved

by the acid, it was thought necessary to determine whether the wool is recovered quantitatively. Skeins of purified wool were dried, weighed, and then immersed for 15 minutes in 80 to 100 times their weight of sulphuric-acid solution at room temperature. The wool was then washed until the wash water gave no test for acid with methyl red indicator or for sulphate with barium-chloride solution. It was finally dried and weighed.

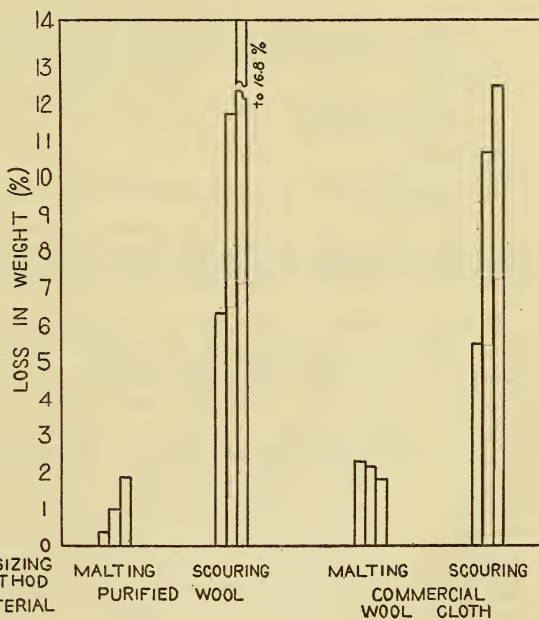


FIGURE 2.—Change in weight of wool specimens subjected to desizing treatments.

[The change in weight of a specimen after desizing 1, 2, and 3 times is shown.]

⁹ Roger Castle Griffin, *Technical Methods of Analysis*, p. 506, 2nd ed. 1927. McGraw-Hill Book Co., Inc., New York.

¹⁰ P. Kraiss and H. Markert, *Quantitative Determination of Cotton, Wool, Silk, and Artificial Silks in Mixed Textiles*. *Jour. Textile Inst.*, vol. 23, p. 214, August 1932.

TABLE 1.—Changes in weight of cleaned wool treated with sulphuric acid

Sample no.	Treatment	Gain in weight	Loss in weight when corrected for absorbed SO ₄
		Percent	Percent
1	Washed with water at room temperature until water gave no test for acid or SO ₄ .	7.0	-----
2		5.1	-----
3		5.7	-----
4		6.8	-----
5		6.5	-----
6		6.2	-----
7		5.5	-----
8		7.8	2.5
9		7.4	2.3
10		8.4	3.0
11		8.7	2.6
12		8.8	2.4
13	Washed and then extracted ¹ with water for 10 hours.....	5.9	-----
14	Same as 13, then extracted with 0.2 percent NH ₄ OH, followed by water.....	5.7	-----
15	Same as 14, followed successively with soap solution, an alcohol, and an ether rinse.....	5.4	-----
16	Immersed for 16 hours in dilute NH ₄ OH and extracted 10 hours with water.....	6.9	-----
17	Immersed for 16 hours in 1 percent sodium-carbonate solution and extracted 10 hours with water.....	6.0	-----
18	Immersed for 16 hours in 1 percent triethanolamine and extracted 10 hours with water.....	9.9	-----
19		8.9	-----
20		7.6	-----
21		Immersed for 16 hours in 2 percent triethanolamine and extracted 10 hours with water.....	7.4

¹ Each extraction was made at room temperature in a modified soxhlet apparatus, the extraction chamber filling with fresh liquid after each discharge, except with the ether and alcohol—these were made with the regular soxhlet extractor in the usual manner.

It is seen from the results given in table 1, for samples numbered 1 to 12, that the wool weighs from 5 to 8 percent more after this treatment. The amount of sulphate left in the wool was determined for samples numbered 8 to 12 by dissolving the yarn in a boiling 5-percent solution of potassium hydroxide, cooling, acidifying with hydrochloric acid, and filtering; the filtrate was heated and the sulphate ion precipitated with barium chloride; the precipitate filtered, washed, ignited, and weighed. After allowing for the sulphate, the weight of the wool is 2 to 3 percent less than that of the original, cleaned, untreated sample, as shown in the last column of the table. It appears that there are at least 2 sources of error inherent in the method. One, the result of absorption and retention of acid by the wool; the other, the result of partial decomposition of the wool in the acid treatment.

An effort was made to eliminate the absorbed sulphate by various treatments without serious decomposition of the wool itself, but none was successful. The results of these experiments are also given in table 1, samples 13 to 21. They show that the sulphuric-acid method as commonly applied to the quantitative separation of wool and cotton or rayon gives inaccurate results because a proper correction is not made for the absorbed acid which is not removed from the wool. The results for wool may be high by as much as 8 percent of the amount actually present.

A modification of the commercial process of carbonization with aluminum chloride has been adopted by some industrial laboratories for the removal of cotton from mixtures with wool. The laboratory method first brought to our attention included the following steps:

1. Boil about 1 g of the fiber mixture for 10 minutes in a 5-percent aqueous solution of aluminum chloride.

2. Remove the fibers from the aluminum chloride solution without squeezing and bake them for 2 hours on a watch glass in an oven at a temperature of 100 to 105 C.

3. Rub the fibers well between the hands over a piece of smooth glazed paper to break up the hydrated cellulose and to separate as much of it as possible from the wool.

4. Collect the hydrated cellulose in a flask and shake it with an aqueous solution of hydrochloric acid. Let the suspension stand for a few minutes and then wash it through a no. 100 sieve with tap water. The hydrated cellulose passes through and any wool fibers mixed with the cellulose will remain on the sieve. Collect the wool fibers and wash them with 10-percent hydrochloric-acid solution. Filter through the sieve and rinse well with tap water.

TABLE 2.—Changes in weight of cleaned wool treated by the different steps of the aluminum-chloride carbonization method¹

Sample no.	Treatment	Change in weight
		<i>Percent</i>
20	Baked at 100° to 105° C.; washed with 3.5 percent HCl, 2 percent Na ₂ CO ₃ , and water..	-0.6
21		- .5
22		- .5
23		- .6
24		- .8
25	Baked at 105° to 110° C.; washed with 3.5 percent HCl, 2 percent Na ₂ CO ₃ , and water..	- .7
26		- .5
27	Baked at 105° to 110° C.; washed with water only-----	2.3
28		1.8
29		2.4
30	Treated with 3.5 percent HCl and washed-----	.1
31		.0
32		.0
33	Baked at 105° to 110° C; washed with 3.5 percent HCl, and water-----	- .2
34		- .3
35		.0

¹ Values are in percentages of the oven-dried wool and represent increases except where preceded by a minus sign.

5. Dry or condition the wool fibers and weigh them.

Purified wool was subjected to treatment by this method and the change in weight determined. The results are recorded in table 2, samples 20 to 24. Modifications of the method were tried to determine whether any of the steps could be omitted without causing greater changes in the weight of cleaned wool treated. The results on samples 25 and 26 show that the baking temperature may be raised to 105 to 110 C so that the same oven used by laboratories for drying fibers to constant weight at this temperature may also be used for baking the specimen for carbonization. Washing the wool fibers with hydrochloric-acid solution is necessary for the removal of decomposition products of the aluminum salt which may otherwise be retained by the wool fibers, producing errors in the weight for wool. This is illustrated by the results obtained on samples 27 to 29, which were not washed with acid. Wool fibers given the treatment received by this group of samples yield a considerable ash on ignition. It seems, however, that the treatment with 2-percent sodium-carbonate solution may be successfully omitted, samples 30 to 32. It is probable that this treatment was introduced with the object of neutralizing any hydrochloric acid that might be held by the wool so that it could be more readily removed. Wool treated at room temperature with

approximately 3.5-percent solution of hydrochloric acid (prepared by diluting 1 part of the concentrated laboratory reagent acid (35 to 36 percent) with 9 parts of distilled water), to remove the decomposition products of aluminum chloride, can readily be washed free of acid with practically no change in weight of the wool treated. This is shown by the results obtained on samples 30 to 32.

These results show that the aluminum-chloride carbonization method for removing cotton from wool, as given above, produces changes in weight of purified wool of less than 1 percent of the weight of the wool treated; that the method may be modified by baking at 105 to 110 C instead of 100 to 105 C to suit the convenience of the analyst; and that the treatment of the wool residue with 2-percent sodium-carbonate solution may be omitted.

No difficulty was experienced in collecting the wool fibers during the rubbing-out process to remove the carbonized cellulose. With the aid of a no. 40 wire sieve placed between the hands and glazed paper the dry wool and cotton can be easily separated and the wool obtained in the form of a tangled mass or mat. Washing the cellulose with hydrochloric acid as described in the first part of step 4 can be omitted. It was further observed that when wet wool fibers are placed upon a no. 100 sieve, some of the ends of the fibers extend to the opposite side and become quite firmly held in the meshes. This screen is not considered satisfactory. A small büchner funnel or gooch crucible was found very convenient and effective for holding the wool fibers while washing them to remove aluminum salts.

With the modifications suggested, the method is convenient and applicable for the accurate determination of wool in mixtures with cotton and regenerated cellulose rayons, but not with cellulose acetate. The regenerated cellulose rayons, like cotton, are carbonized by the treatment. Many of the common sizing and finishing materials are likewise removed.

V. REMOVAL OF WOOL FROM MIXTURES WITH COTTON

The fact that wool is readily dissolved by hot caustic solution has been the basis of quantitative methods for separating wool from cotton. The residual cotton is weighed and the amount of wool is estimated from this weight and that of the untreated specimen. It has been customary to make an arbitrary correction on the weight of cotton residue obtained to allow for natural noncellulosic materials likely to be present in it. It seems preferable for purposes of analysis to consider "cotton" and "cotton cellulose" as synonymous terms. Any allowances that may be desired may conveniently be based upon this weight.

Specimens of purified cotton—some of it in the form of yarns and some as unspun fibers—were subjected to the treatment for removal of wool; that is, immersion for 15 minutes in a boiling 5-percent solution of potassium hydroxide. Care was taken to keep the specimen submerged and increased concentration of the solution by evaporation was avoided by use of a reflux condenser. The cotton was then rinsed with water, then with 1-percent aqueous solution of acetic acid and again with water until neutral to methyl-red indicator. Figure 1 shows the loss in weight of purified cotton, unbleached and bleached cotton when subjected to desizing treatments followed by a boiling

caustic-potash solution to remove wool. It was found that purified cotton cellulose lost no appreciable weight when subjected to a malting followed by a caustic-potash treatment. Bleached and unbleached cotton lost much more. This can only be accounted for by the presence of materials other than cotton cellulose. The amount lost indicates the magnitude of error for wool when it is determined by difference.

When cotton cellulose only is to be determined, a preliminary extraction and malting treatment to remove size and finishing materials may not be necessary; the results show, however, that a specimen used for determination of size and finishing materials by the malting method may also be used for the determination of cotton cellulose.

VI. PROPOSED METHOD FOR THE ANALYSIS OF WOOL-COTTON MIXTURES

Wool and cotton fibers may be separated mechanically and determined with the aid of a microscope and proper auxiliary equipment.^{11 12} Such separation is easily made if the wool and cotton are in separate yarns in a fabric, but is tedious and less reliable if the fibers are spun together into yarns. If a method of counting the fibers is used, computations must be made to take into account the relative diameters and densities of wool and cotton fibers if the results are to be expressed on either a volume or weight percentage basis. The following method of chemical analysis is proposed for use where mechanical methods for separation of the fibers are inconvenient or impractical.

1. DRYING AND WEIGHING

Dry 5 g of the material in an oven maintained at 105 to 110 C (221 to 230 F) to constancy ± 0.1 percent of the weight of the specimen. A satisfactory procedure is to dry the specimen for 1½ hours, remove it from the oven, place it while warm in a tared weighing bottle, cover, cool in a desiccator, and weigh; return the specimen to the oven and repeat the drying and weighing until constant weight is attained. Let the weight of the oven-dried sample be called *A*.

2. TOTAL SIZING, FINISHING, AND OTHER NONFIBROUS MATERIALS

(a) The purpose of this procedure is to remove nonfibrous natural constituents and materials added by the manufacturer, such as starch, china clay, soap, some waxes, and some nondrying oils. However, general directions for the removal of all substances which may be present cannot be given. When it is necessary to modify the procedure in order to remove nonfibrous constituents completely, the analyst should make certain that purified fibers do not lose weight, or are not made susceptible to loss when analysed for fiber content according to the modified procedure.

(b) Extract the dried specimen from 1 in a soxhlet apparatus for 2 hours with carbon tetrachloride. Allow the specimen to dry and then wash it by alternate immersion in hot distilled water and by squeezing.

¹¹Edward R. Schwarz. Microscope is valuable asset to textile manufacturer and finisher. *Textile World* vol. 80, p. 770, Aug. 29, 1931.

¹²Heermann, Paul, und Herzog, Alvis, *Mikroskopische und Mechanischtechnische Textiluntersuchungen*, p. 216, 3d ed., 1931. Julius Springer, Berlin.

(c) Immerse the specimen in a 0.5-percent aqueous solution of a starch-hydrolyzing enzyme at 50 C (122 F), squeeze the specimen while it is immersed, then remove and squeeze it again.¹³ This procedure is repeated with the same solution for a total of three times to saturate the fibers. The enzyme solution is then maintained at 70 ± 1 C (158 F) and the specimen kept immersed in it for a period of 15 minutes. Rinse the specimen 12 times in fresh portions of hot distilled water, squeeze it after each rinse, and dry it to constant weight as before. This gives the weight of dry fiber, called *B*. The percentage of sizing, finishing, and other nonfibrous materials in terms of the weight of the oven-dried specimen is $100 \frac{A-B}{A}$. Care must be taken at all times not to wash away any fibers.

3. DETERMINATION OF COTTON CELLULOSE

Place the specimen from 2 (c) in a boiling 5-percent aqueous solution of potassium hydroxide¹⁴ (previously boiled to remove dissolved air) for 10 minutes; sufficient solution is taken to cover the specimen continually. Decant or filter the solution and rinse the cotton residue with hot distilled water, then with a 5-percent aqueous solution of acetic acid, and finally with water until the rinse water is neutral to blue litmus paper. The residue is dried and weighed, giving weight *C*. This is the weight of dry cotton cellulose in the specimen. Then the percentage of cotton in the dried specimen is $100 \frac{C}{A}$.

4. DETERMINATION OF WOOL

(a) This method is applicable to the determination of wool in mixture with cotton, regenerated cellulose rayons, or both. It is not applicable to mixtures containing cellulose acetate or silk.

(b) Treat a specimen of the material as described under 1, 2 (b), and 2 (c), but omit the weighing under 2 (c). Immerse the specimen from 2 (c) for 10 minutes in a boiling solution of aluminum chloride prepared by dissolving 9 g of the hydrated salt ($\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$) in 100 ml of water. Remove the specimen from the solution; allow the excess liquid to drain or the specimen may be lightly squeezed. Heat it at 105 to 110 C (221 to 230 F) until the cotton has become brown and brittle—2 hours is usually sufficient. Hold the specimen over a piece of glazed paper and work it with the hands in order to pulverize the carbonized cellulose and separate it from the wool. By suitable manipulation, all of the wool is obtained as a mat and the cellulose is left on the paper, mostly in the form of a powder. The separation is facilitated by the use of a wire screen, approximately no. 40, between the hands and the glazed paper. Place the wool in a beaker of warm water, agitate it thoroughly to wet it, and form a mat of the coarser fibers in a gooch crucible or büchner funnel; apply gentle suction and pour the remaining fibers and filtrate on the pad. Remove the fibers and agitate them with a solution of hydrochloric acid prepared by diluting 1 part of the concentrated laboratory acid

¹³ The effectiveness of the hydrolytic action of the enzyme should always be tested when in doubt. This may be done by treating fabric or yarn, known to contain starch sizing, with the enzyme solution according to the method prescribed and then testing for starch with iodine. This may be done by placing the fibers in a straw-colored aqueous solution of iodine. The fibers should not be blue after 5 minutes immersion.

¹⁴ Although potassium hydroxide is preferred for critical work, sodium hydroxide is probably satisfactory for ordinary purposes.

with 9 parts of distilled water. Repeat the operation using several portions of water instead of acid until the wool is freed from powdered cellulose and the wash water is free from chlorides. The wool is dried to constant weight, called *D*. Then the percentage of wool in the specimen is $100 \frac{D}{A}$.

A less reliable value for wool may be obtained by difference after the removal of wool according to paragraph 3, by use of the formula $100 \frac{B-C}{A}$; similarly, cotton or cellulosic material may be determined

by the formula $100 \frac{B-D}{A}$.

VII. ANALYSIS OF MIXTURES OF KNOWN COMPOSITION BY THE PROPOSED METHOD

Table 3 gives the results of analyses to determine the amount of wool in known mixtures of purified cotton and wool. The samples were prepared in the laboratory by drying purified wool and cotton, some as yarns and others as loose fibers, and accurately weighing and then mixing them. They were then analyzed according to the method described in section VI.

TABLE 3.—Analyses of prepared wool-cotton mixtures for wool content

Wool content (percent)			
Present	Found	Present	Found
87.9	88.2	49.4	49.1
83.8	83.8	40.1	40.0
78.3	79.0	29.4	29.1
72.8	72.3	20.0	20.3
69.4	69.7	9.9	9.9
57.8	57.7	5.3	5.3

Other samples, prepared similarly, were analyzed for cotton content by the method recommended for cotton by two analysts working independently, one inexperienced in its use. The results of these analyses are given in table 4.

TABLE 4.—Analyses of wool-cotton mixtures for cotton content

Cotton content (percent)					
Analyst	Present	Found	Analyst	Present	Found
G.....	53.0	52.6	J.....	48.3	47.3
	55.4	55.1		49.3	48.9
	80.1	79.5		18.0	18.0
	80.5	80.0		18.5	18.4
	19.8	19.5		85.4	85.2
	23.6	23.4		83.8	83.6

When mixtures to be analyzed consist of comparatively pure cotton and wool, either one of these may be determined directly as described, and the other by difference, with a fair degree of accuracy. In actual practice, however, fabrics may contain other materials which may introduce an error in the figure for fiber content obtained by difference. This error is usually eliminated if the fiber which is to be determined is weighed directly.

It is not always required that sizing and finishing materials be determined. The aluminum-chloride carbonization method for determining wool and the potassium-hydroxide method for determining cotton may be applied directly to specimens of the original mixture provided extraneous materials are removed by the treatments.

Although the fibers used in these tests did not contain size, they were given the treatment recommended for its removal. Therefore, the results given include the possible error produced. The method gives the amount of cotton and wool in mixtures of these fibers to within 1 percent of the total weight of the fiber present.

WASHINGTON, October 11, 1933.