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Standard Test Methods for Quantitative Analysis of Textiles[1]

This standard is issued under the fixed designation D 629; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Methods D 629-59 T, Quantitative Analysis of Textiles, were discontinued in 1969 because the responsible subcommittee failed to recommend their adoption as a standard after several years of publication as a tentative. The subcommittee action was based on the members' knowledge that the standard did not include several fiber types introduced to the textile trade after the method was published, and that the techniques required for their identification were lacking in the text, allowing it to become out of date. The procedures included in the text, however, are believed to be reliable for the fiber types named and the techniques described are currently being used in the trade and are referenced by other standards sponsored by Committee D-13 on Textiles. Reinstatement as a standard using the previously assigned number was requested since the listed procedures were reliable and the text considered to be the best available, though not all inclusive. Extensive editorial changes were made in various sections in 1972, and the methods were reinstated as D 629-72. Editorial changes have again been made throughout the text, and statements on precision and bias and suitability for acceptance testing have been added.

The text of Methods D 629-59 T was published by the American Association of Textile Chemists and Colorists in that society's Technical Manual as "Test Method 20A-1959" issued in the years 1959 through 1974. The AATCC Method was revised completely in 1975 and since published as "Test Method 20A-1975."

1. Scope

1.1 These test methods cover procedures for the determination of the fiber blend composition of mixtures of the fibers listed in 1.2. Procedures for quantitative estimation of the amount of moisture and certain nonfibrous materials in textiles are also described, for use in the analysis of mixtures, but these are not the primary methods for the determination of moisture content for commercial weights.

1.2 These test methods cover procedures for the following fiber types:

1.2.1 *Natural Fibers*:

1.2.1.1 *Cellulose-Base Fibers*:

Cotton
Hemp
Flax
Ramie

1.2.1.2 *Protein-Base Fibers*:

Animal hairs (other than wool)
Silk, cultivated
Silk, Tussah
Wool

1.2.2 *Man-Made Fibers*:

1.2.2.1 *Cellulose-Base Fibers*:

Acetate (secondary)
Rayon, viscose or cuprammonium
Triacetate

1.2.2.2 *Synthetic-Base Fibers*:

Acrylic
Aramid
Modacrylic
Nylon 6, Nylon 6-6, others
Olefin
Polyester
Spandex

1.3 These test methods include the following sections and tables:

	Section
Referenced Documents	2
Terminology	3
Summary of Methods	4
Uses and Significance	5
Sampling	7
Purity of Reagents	6
Moisture Content or Moisture Regain	9
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[1] These test methods are under the jurisdiction of ASTM Committee D-13 on Textiles and are the direct responsibility of Subcommittee D13.51 on Chemical Conditioning and Performance.

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1.4 The analytical procedures described in the test methods are applicable to the fibers listed in 1.2. The test methods are not satisfactory for the separation of mixtures containing fibers that fall within the same generic class but differ somewhat, either physically or chemically, from each other. These test methods are not satisfactory for the determination of bicomponent fibers.

NOTE 1—For other methods of analysis covering specific determinations, refer to: Test Methods D 461, Test Method D 584, Methods D 885, Test Method D 1113, Test Method D 1334, and Test Method D 2130. Methods for moisture are covered in Methods D 885, Test Method D 1576, Test Method D 2462, Test Method D 2495 and Test Methods D 2654. For the determination of commercial weight, refer to Test Method D 2494.

1.5 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 123 Terminology Relating to Textiles[2]

D 276 Test Methods for Identification of Fibers in Textiles[2]

D 461 Test Methods for Felt[2]

D 584 Test Method for Wool Content of Raw Wool—Laboratory Scale[2]

D 885 Methods of Testing Tire Cords, Tire Cord Fabrics, and Industrial Filament Yarns Made From Man-Made Organic Base Fibers[2]

D 1113 Test Method for Vegetable Matter and Other Alkali-Insoluble Impurities in Scoured Wool[2]

D 1193 Specification for Reagent Water[3]

D 1334 Test Method for Wool Content of Raw Wool—Commercial Scale[2]

D 1576 Test Method for Moisture in Wool by Oven Drying[2]

D 1909 Table of Commercial Moisture Regains for Textile Fibers[2]

D 2130 Test Method for Diameter of Wool and Other Animal Fiber by Microprojection[2]

D 2462 Test Method for Moisture in Wool by Distillation With Toluene[2]

D 2494 Test Method for Commercial Mass of a Shipment of Yarn or Man-Made Staple Fiber or Tow[2]

D 2495 Test Method for Moisture in Cotton by Oven-Drying[2]

D 2654 Test Methods for Moisture Content and Moisture Regain of Textiles[2]

D 2816 Test Method for Cashmere Coarse-Hair Content in Cashmere[2]

D 2817 Specification for Maximum Cashmere Coarse-Hair Content in Cashmere[2]

2.2 *AATCC Method:*

20 A Test Method for Fiber Analysis: Quantitative[4]

3. Terminology

3.1 *Definitions:*

3.1.1 *textile*, *n*—originally a woven fabric, now generally applied to: (*1*) staple fibers and filaments suitable for conversion to or use as yarns, or for the preparation of nonwoven fabrics, (*2*) yarns made from natural or man-made fibers, (*3*) fabrics and other manufactured products made from fibers as defined above and from yarns, and (*4*) garments and other articles fabricated wholly from one or more of the above elements, and articles made principally from the above when the products retain the characteristic flexibility and drape of the original fabrics.

3.1.1.1 *Discussion*—Typical examples (but not a complete listing) of materials included in the above definitions are: (*1*) Natural fibers such as cotton, wool, sisal, and asbestos, also silk filaments, after these materials have been subjected to their first preparatory process, and man-made fibers such as rayon, nylon, glass, and metals in the form of spinnable fibers, monofilaments, multifilament yarns, or tows; (*2*) Yarn intermediates such as sliver, roving, and top; yarn in single or plied, spun or filamentous form; threads, cords, and ropes but not metallic cables; yarn substitutes including twisted paper or foil, slit foil, or slit laminated sheets, metallic or plastic monofilaments; (*3*) Products made from materials included in (*1*) or (*2*), or both, by interlacing in any manner including weaving, knitting, tufting and braiding, bonding, knotting, or felting and including such products as cloths, woven goods, knitted goods, lace, embroidery, nets, webbing, batts, and nonwoven fabrics. Reinforced plastics and papers are not included; (*4*) Wearing

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[2] *Annual Book of ASTM Standards,* Vol 07.01.

[3] *Annual Book of ASTM Standards,* Vol 11.01.

[4] AATCC Technical Manual, available from the American Association of Textile Chemists and Colorists, P.O. Box 12215, Triangle Park, NC 27709.

apparel, household, and industrial articles fabricated principally from one or more of the materials listed in (*1*), (*2*), and (*3*).

3.1.2 *bicomponent fiber*, *n*—a fiber consisting of two polymers which are chemically different, physically different, or both.

3.2 For definitions of other textile terms used in these test methods, refer to Terminology D 123.

4. Summary of Test Methods

4.1 Summaries of the specific methods used for different tests are given in the appropriate sections.

4.2 The methods for fiber analysis are grouped under three headings, as follows: Mechanical Separation or Dissection, Chemical Test Methods, and Microscopical Analysis.

NOTE 2—It is assumed that the analyst knows from qualitative tests (as directed in Test Methods D 276) what fibers are present and, therefore, which method of analysis is applicable. The choice of method will depend upon the nature of the material to be analyzed and, in some cases, on the accuracy required.

5. Significance and Use

5.1 Test Methods D 629 for the determination of quantitative analysis of textiles may be used for acceptance testing of commercial shipments but caution is advised since information on between-laboratory precision is lacking. Comparative tests as directed in 5.1.1 or by Table D 1909 may be advisable.

5.1.1 In case of a dispute arising from differences in reported test results using Test Methods D 629 for acceptance testing of commercial shipments, the purchaser and the supplier should conduct comparative tests to determine if there is a statistical bias between their laboratories. Competent statistical assistance is recommended for the investigation of bias. As a minimum, the two parties should take a group of test specimens which are as homogeneous as possible and which are from a lot of material of the type in question. The test specimen should then be randomly assigned in equal numbers to each laboratory for testing. The average results from the two laboratories should be compared using Student's *t*-test and an acceptable probability level chosen by the two parties before the testing began. If a bias is found, either its cause must be found and corrected or the purchaser and supplier must agree to interpret future test results in the light of the known bias.

5.2 The effects of the various reagents used in the chemical methods on the residual fibers in a blend depend upon the history of the fibers and, unless otherwise stated, are generally too small or too uncertain to warrant the application of correction factors.

5.3 Fiber composition is generally expressed either on the oven-dry mass of the original sample or the oven-dry mass of the clean fiber after the removal of nonfibrous materials. If nonfibrous materials are not first removed from the textile before the fiber analysis is carried out, or if the treatments described in Section 8 are incapable of removing them, any such materials present will increase the percentage of the fiber constituent with which they are removed during the analysis, assuming they are soluble in the solvent used.

5.4 The analytical methods are intended primarily for the separation of binary mixtures of fibers. These procedures may also be used for the analysis of mixtures containing more than two types of fibers by selecting the best combination of methods to use (Table 1). Since a sequence of solvents on a given fiber may produce different results than the expected

TABLE 1 Chemical Methods for Analysis of Fiber Mixtures^A

	Wool	Spandex	Silk	Rayon	Polyester	Olefin	Nylon	Mod-acrylic	Cellulosic, Natural	Aramid	Acrylic	Triacetate
Acetate	1		1	1	1	1	1	1	1		1	1
Triacetate	3		(5)	(7^5)^B	9	3^9	(5)	9	(5)		3	
Acrylic	(6)		(6)	(7^5)	10	(8)	10	10	(5^7)			
Aramid	(6)											
Cellulosic, Natural	(6)^5	(10)	(6)	(4)	5	5	(3)	(2)				
Modacrylic	2	2	2	2	2	2	2					
Nylon	3	(10)	(6)	3	9	(8)						
Olefin	(6)		(6)	(7^5)	8							
Polyester	(6)	(10)	(6)	(7)								
Rayon	5		(6)									
Silk		6										
Spandex	(6)											

^{A}Key to Methods and Reagents:
Method No. 1—80 % acetone(cold)
Method No. 2—*N*-Butyrolactone
Method No. 3—90 % formic acid
Method No. 4—59.5 % sulfuric acid
Method No. 5—70 % sulfuric acid
Method No. 6—Sodium hypochlorite solution
Method No. 7—Curpammonia solution
Method No. 8—Hot xylene
Method No. 9—90 % formic acid
Method No. 10—N,N-dimethylacetamide

^{B}Each analytical method is identified by a number and where possible, two methods of analysis are provided for each binary mixture of fibers. The number or numbers inside parentheses refers to the method that dissolves the fiber shown at the top of the diagram. The number or numbers outside the parentheses indicates the method that dissolves the fiber listed at the left side of the diagram. Where two methods are listed for a specific binary mixture, the non-superscript method number represents the method of choice.

results from a single solvent, it is advisable to determine the results of such sequential effects when testing multiple fiber blends. It is sometimes more convenient to separate mechanically the yarns in a textile which are of similar types, and then use the appropriate chemical method to analyze each of the components. Table 2 shows the solubilities of the various fibers in different chemical reagents.

6. Purity of Reagents

6.1 Use reagent-grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

6.2 Unless otherwise indicated, references to water shall be understood to mean water conforming to Type I grade of Specification D 1193.

7. Sampling

NOTE 3—It is not possible to provide specific instructions for all types of textile materials to which these analytical methods may be applicable, but a few general recommendations shall be followed.

7.1 *Sampling for Acceptance Testing*:

7.1.1 *Lot Sample*—As a lot sample for acceptance testing, take at random the number of shipping containers directed in an applicable material specification or other agreement between the purchaser and the supplier. Consider shipping containers to be the primary sampling units.

NOTE 4—An adequate specification or other agreement between the purchaser and the supplier requires taking into account the variability between and within shipping containers so as to provide a sampling plan with a meaningful producer's risk, consumer's risk, acceptable quality level, and limiting quality level.

7.1.2 *Laboratory Sample*—As a laboratory sample for acceptance testing, proceed as follows:

7.1.2.1 *Fiber in Bulk*—Take one composite sample from each bale or other shipping container in the lot sample with each such composite sample being taken from at least five places in the shipping container.

7.1.2.2 *Textile Strands*—Take one package at random from each shipping container in the lot sample.

7.1.2.3 *Fabric*—Take a full width swatch 1 m long or a swatch long enough to contain one pattern repeat, whichever is longer, from the end of each roll of fabric in the lot sample, after first discarding a minimum of 1 m of fabric from the very outside of the roll.

7.1.2.4 *Garments*—Take one garment at random from each shipping container in the lot sample.

7.1.2.5 *Other Textiles*—Take one laboratory sample from each shipping container in the lot sample as agreed between the purchaser and the supplier.

7.1.3 *Test Specimens*—Unless otherwise directed in the test method(s) of interest, prepare a test specimen having a mass of 1.0 to 1.5 g by placing it in a weighing bottle. Place the weighing bottle and specimen in an oven and heat for 1 h at a temperature of 105° to 110°C. Transfer the weighing bottle to a desiccator and allow it to cool to room temperature. For fabrics containing a repeating weave pattern, include all yarns in the pattern within the test specimen. Take the specimen from other units when more than one unit is being tested or analyzed.

7.2 *Sampling for Other Purposes*—In general, follow the principles specified for samples for acceptance testing. Insofar as the amount of material available and the purposes of the analysis dictate, take a lot sample, laboratory sample, and test specimens in such a manner that all fibers present have a

TABLE 2 Solubilities of Fibers in Solvents Used in Chemical Methods[A]

	Method No. (1)	(2)		(3) and (9)	(4)	(5)	(6)	(7)	(8)	(10)
	80 % Acetone	Butyrolactone		90 % Formic Acid	59.5% H_2SO_4	70% H_2SO_4	NaOCl Solution	Curpammonia Solution	Hot Xylene	N,N-dimethyl acetamide
		(A) RT	(B) 75°C							
Acetate	S	S	PS	S	S	S	I	I	...	...
Triacetate	I	PS	PS	S	I	I		I	...	S
Acrylic	I	S	S	I	I	I	I	I	I	S
Aramid										
Cellulosic, Natural	I	I	I	I	SS	S	I	S	...	I
Modacrylic	I	S	S	I	I	I	I	I	...	I
Nylon	I	I	I	S	S	S	I	I	I	I
Olefin	I	I	I	I	I	I	I	I	S	I
Polyester	I	I	I	I	I	I	I	I	I	I
Rayon	I	I	I	I	S	S	I	S	I	I
Silk	I	I	I	PS	S	S	S	...	...	S
Spandex	...	I	I	PS	I	I	I	...	...	S
Wool and Hair Fibers	I	I	I	I	I	I[B]	S	...	...	...

[A] Key to Symbols:
S = Soluble
PS = Partially Soluble
SS = Slightly Soluble (a correction factor may be applied)
I = Insoluble

[B] Reworked wools are soluble in 70 % H_2SO_4 depending upon their previous history.

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[5] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

proportional probability of being sampled.

8. Nonfibrous Materials

8.1 *Scope*—These procedures are intended for the removal of the nonfibrous natural constituents of the fiber and substances added by the manufacturer. Starch, China-clay, soaps, some waxes, some nondrying oils, certain resins, and the usual natural constituents are in this category and are removed by the procedures described. However, general directions for the removal of all possible substances which may be present cannot be included. Inorganic pigments which are frequently used as delustrants in the manufacture of man-made fibers and types of dyes are not removed by the procedures described in 8.4. Some of the newer finishes present special problems and the analyst will have to deal with these cases as they arise. When it is necessary to modify the procedure to remove nonfibrous constituents, the analyst shall make sure that cleaned (Note 5) specimens of the fibers under consideration do not lose weight when subjected to the same treatment. The treatments described in 8.4 will allow removal of most, but not all, nonfibrous materials that may be present on textiles. The total amount of nonfibrous materials removed by these treatments may be determined by subjecting the specimen in turn to each of the treatments described. When the type of finish present is known, only the appropriate treatment need be used.

8.2 *Reagents*:

8.2.1 *Ethyl Alcohol*—Pure ethyl alcohol or ethyl alcohol denatured with methyl alcohol.

8.2.2 *Solution,* of 5 % urea/1.5 % phosphoric acid (85 %).

8.2.3 *Trichlorotrifluoroethane.*[6]

8.3 *Test Specimen*—Take a specimen having a mass of approximately 5 g. Take care to prevent the loss of fibers in the treatments. Dry the specimen at 105 to 110°C to constant mass to obtain the oven-dry mass of the original specimen, mass *C*, and then subject the specimen to one or more of the treatments described in 8.4.

8.4 *Procedure*:

8.4.1 *Solvent Extraction* (for removal of oils, fats, waxes, certain thermoplastic resins, etc.)—Extract the dried specimen for 2 h with trichlorotrifluoroethane (Note 5) in a Soxhlet or similar extractor, siphoning over a minimum of six times. Air dry the specimen and then dry in an oven at 105 to 110°C to constant mass.

NOTE 5—Trichlorotrifluoroethane is nonflammable, has a very low level of toxicity, and a high degree of stability. It is recommended in preference to carbon tetrachloride, which has been used historically for its excellence as a solvent for oils, fats, and the like. This preference is based on the toxicity of carbon tetrachloride by absorption or inhalation and its tendency to decompose in the presence of moisture, with the formation of hydrochloric acid.

8.4.2 *Alcohol Extraction* (for removal of soaps, cationic finished, etc.)—Extract the dried specimen for 2 h with ethyl alcohol in a Soxhlet or similar extractor, siphoning over a minimum of six times. Air-dry the specimen and then dry in an oven at 105 to 110°C to constant mass.

8.4.3 *Aqueous Treatment* (for removal of water-soluble materials)—Immerse the dried specimen for ½ h in water at 50°C using a liquid to fabric ratio of approximately 100:1. Agitate the specimen occasionally by stirring with a glass rod or by mechanical means. Rinse three times in fresh portions of warm (50°C) water and dry in an oven at 105 to 110°C to constant mass.

8.4.4 *Enzyme Treatment* (for removal of starch, gelatin, etc.)—Immerse the specimen in an aqueous solution of a starch and protein-solubilizing enzyme preparation using the concentration, liquid to fabric ratio, temperature, and time of immersion recommended by the supplier of the enzyme preparation. After this treatment, thoroughly rinse the specimen with fresh portions of hot water. Thorough rinsing is necessary to remove China-clay or similar material from heavily filled fabrics. Dry the rinsed specimen in an oven at 105 to 110°C to constant mass.

8.4.5 *Removal of Amino-Aldehyde Resins*— Treat the specimen with 1.5 % phosphoric acid (85 %) and 5 % urea at 75°C for 15 to 30 min with occasional stirring. Rinse thoroughly in hot (80°C) water, and dry the specimen in an oven at 105 to 110°C to constant mass, mass *D.*

NOTE 6—The fiber mass remaining after being subjected to the treatments described in 8.4.1-8.4.5 is referred to as "clean fiber" in the following sections.

8.5 *Calculation*:

8.5.1 Calculate the total percentage of nonfibrous materials in the specimens using Eq 1:

$$\text{Nonfibrous materials, \%} = 100\,(C - D/C) \tag{1}$$

where:

C = oven-dry mass of the original specimen, g, and
D = oven-dry mass of the specimen after acid treatment, g.

8.5.2 The percentage of nonfibrous materials removed by any of the individual treatments described in 8.4 may also be calculated using the dry mass of the specimen determined after specific stages.

8.6 *Report*:

8.6.1 State that the amount of nonfibrous material was determined as directed by Test Methods D 629.

8.6.2 Describe the material(s) or product(s) sampled and the method of sampling used.

8.6.3 State the type of extraction used.

8.6.4 Report the individual and the average values of percent nonfibrous materials.

8.7 *Precision and Bias*:

8.7.1 *Precision*—The precision of the procedure for nonfibrous materials in Test Methods D 629 is being established.

8.7.2 *Bias*—The procedure in Test Methods D 629 for measuring the nonfibrous materials in textiles has no known bias because the value of the nonfibrous materials in textiles can be defined only in terms of a test method.

9. Moisture Content or Moisture Regain

9.1 *Scope*—This test method may be used to determine the amount of moisture in a textile either as it is received or when it is in moisture equilibrium for testing in the standard

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[6] The solvent grade of the trichlorotrifluoroethane is available under various trademarks including Freon, Isotron, Ucon, and also Grenesolve.

atmosphere for testing textiles, as defined in Terminology D 123.

9.2 *Summary of Method*—Specimens are heated until they reach constant mass; the loss in mass in considered moisture.

NOTE 7—When textiles are heated under the conditions described in 9.5, volatile materials in addition to moisture may be removed. If this possibility is known or suspected, it should be reported that the percentage loss in mass of the textile does or may include volatile substances as well as moisture.

9.3 *Apparatus*:

9.3.1 *Weighing Bottle*, glass, approximately 100-mL capacity, fitted with a ground-glass cover, or an aluminium weighing can, approximately 100-mL capacity, and having a tight-fitting cover.

9.3.2 *Desiccator*, containing anhydrous calcium sulfate ($CaSO_4$) or other suitable dehydrating agent.

9.3.3 *Analytical Balance*, capable of weighing to 0.1 mg.

9.3.4 *Oven*, maintained at 105 to 110°C.

9.3.5 *Special Equipment*, for drying specimens to constant mass, generally available in textile laboratories (conditioning ovens, etc.) may also be used. The apparatus listed in 9.3.1-9.3.4 and the procedure described in 9.5 are provided for laboratories not so equipped and shall be used in cases of dispute, with regard to the fiber composition of mixtures.

9.4 *Test Specimen*—Take a specimen having a mass of approximately 1 g and weigh it to the nearest 0.01 g.

9.5 *Procedure*:

9.5.1 Place the weighing bottle and cover separately in the oven. After heating for 1 h at a temperature of 105 to 110°C, replace the cover, transfer the weighing bottle to the desiccator, and allow it to cool to room temperature. Remove the cover momentarily to equalize the pressure, and with the cover in place, weigh the container. Repeat the heating, cooling, and weighing until the mass of the empty weighing bottle is constant to within± 0.001 g.

9.5.2 Place the specimen to be tested in the container, cover, and weigh. Subtract the mass of the empty container (9.5.1) from this mass to obtain the as-received mass of the original specimen, mass A.

9.5.3 Place the uncovered weighing bottle and specimen in the oven for 1.5 h at a temperature of 105 to 110°C. Cover the container and transfer it and the specimen to a desiccator. When the container has cooled to room temperature, remove the cover momentarily to adjust the pressure, replace the cover, and weigh the container and the specimen. Repeat the heating for periods of not less than 20 min, cooling and weighing until the mass is constant to within ±0.001 g. Subtract the mass of the empty container (9.5.1) from this mass to obtain the oven-dry mass of the original specimen, mass B (Note 7).

9.6 *Calculation*:

9.6.1 Calculate the moisture content of the specimen using Eq 2 and the moisture regain of the specimen using Eq 3:

$$\text{Moisture content, \%} = 100\ (A - B/A) \quad (2)$$

$$\text{Moisture regain, \%} = 100\ (B - B/B) \quad (3)$$

where:
A = as-received mass of the specimen, g, and
B = oven-dry mass of the specimen, g.

9.7 *Report*:

9.7.1 State that the moisture content of the specimen was determined as directed by Test Methods D 629.

9.7.2 Describe the material(s) or product(s) sampled and the method of sampling used.

9.7.3 Report the individual and the average values.

9.8 *Precision and Bias*:

9.8.1 *Precision*—The precision of the procedure for determining moisture content in Test Methods D 629 is being established.

9.8.2 *Bias*—The true value of the moisture content in textiles can only be defined in terms of a specific test method. Within this limitation, the procedure for determining the moisture content in Test Methods D 629 has no known bias.

MECHANICAL SEPARATION OR DISSECTION

10. Fiber Analysis by Dissection

10.1 *Scope*—This method is intended for mechanical separation of yarns in a textile, provided the different fibers are segregated.

10.2 *Summary of Method*—A specimen is carefully selected to include all the yarns in a complete repeat of the pattern. The component yarns are mechanically separated, dryed, and weighed separately after heating at 105 to 110°C to constant mass. The percentage composition is calculated for each fiber type present.

10.3 *Apparatus*:

10.3.1 *Analytical Balance*, accurate to 0.1 mg.

10.3.2 *Weighing Bottle*, approximately 25 mL capacity and having a tight-fitting cover.

10.3.3 *Oven*, maintained at 105 to 110°C.

10.3.4 *Desiccator*, with anhydrous calcium sulfate or equivalent.

10.4 *Test Specimen*—Take a specimen having a mass of approximately 1 g.

10.5 *Procedure*:

10.5.1 Select the specimen carefully to include all the yarns in a complete repeat of the pattern.

10.5.2 Place the entire specimen in a weighing container preweighed to the nearest 0.001 g.

10.5.3 Place the weighing container and specimen in an oven at 105 to 110°C for 1.5 h.

10.5.4 Remove the weighing container and specimen from the oven and transfer them to a desiccator. When the container has cooled to room temperature, weigh the container and the specimen to the nearest 0.001 g. Subtract the mass of the empty container from this mass to obtain the oven-dry mass of the specimen, mass B.

10.5.5 Separate the component yarns and place each of the fiber types into separate preweighed weighing containers.

10.5.6 Place the weighing containers with specimens in an oven for 1.5 h at 105 to 110°C.

10.5.7 Remove the weighing container and specimen from the oven and transfer to a desiccator. Allow the container to cool to room temperature and then weigh the containers and specimens to the nearest 0.001 g. Subtract the mass of the empty containers from these masses to obtain the oven-dry mass of the specimen, mass A_1, A_2. etc.

10.6 *Calculation*:

10.6.1 Calculate the percent composition of each fiber type, to the nearest 0.1 %, of dry sample using the following equation:

$$\text{Fiber type } A_1\text{, \% dry} = 100(A_1/B) \quad (4)$$

where:

A_1 = separate fiber type dry mass from 10.5.7, and
B = original dry specimen mass from 10.5.4.

10.7 *Report*:

10.7.1 State that the specimens were tested as directed by mechanical separation or dissection of Test Methods D 629.

10.7.2 Describe the material(s) or product(s) sampled and the method of sampling used.

10.7.3 Report the individual and the average values of percent of each fiber type, based on dry sample.

10.8 *Precision and Bias*:

10.8.1 *Precision*—An interlaboratory study has been organized. The analysis data will be completed within the year 1999 and the precision statement will be balloted for inclusion in the test method by the end of the year 2000.

10.8.2 *Bias*—The procedure in Test Methods D 629 for measuring the fiber content of textiles by mechanical separation or dissection has no known bias because the values of fiber content of textiles by mechanical separation or dissection can be defined only in terms of a test method.

CHEMICAL TEST METHODS

11. Summary of Methods

11.1 A diagrammatic arrangement of binary mixtures of fibers and appropriate methods of analysis are shown in Table 1. Each analytical method is identified by a number and, where possible, two methods of analysis are provided for each binary mixture of fibers. The number or numbers inside parentheses refers to the method that dissolves the fiber shown at the top of the diagram. The number or numbers outside the parentheses indicates the method that dissolves the fiber listed at the left side of the diagram. Where two methods are listed for a specific binary mixture, the nonsubscript method number represents the method of choice.

11.2 An indication of the solubilities of various textile fibers in the solvents specified in the chemical methods of separation herein described is given in Table 2. These solubilities may be used as a guide in selecting appropriate methods for analyzing mixtures that contain more than two types of fibers.

11.3 In these methods of analysis, provision is made for expressing fiber composition either on the oven-dry mass of clean fiber after the removal of nonfibrous materials or on the moisture regain basis.

12. Specimens and Symbols

12.1 For each of the methods of analysis described in Sections 13-22, take a specimen having a mass of approximately 1 g. Treat the specimen as directed in 8.4 and determine the mass of the clean fiber after oven-drying, mass F.

12.2 If requested, determine the mass of the original specimen after oven-drying (see 9.4), to the nearest 0.001 g, mass E.

12.3 Use the symbols E and F in the number equations in Sections 13-22, as follows:

E = mass of the original specimen after oven−drying, g, and (5)
F = mass of original dry clean specimen (after oven−drying), g. (6)

13. Chemical Test Method No. 1—Acetate Mixed With Other Fibers

13.1 *Scope*—This test method is intended primarily for the separation of acetone-soluble types of acetate, and should be used for mixtures of acetate (secondary) fibers and some other fibers including modacrylic fibers.

13.2 *Summary of Method*—Regular secondary acetate is dissolved in 80 % cold acetone and the remaining fiber is determined quantitatively by gravimetric methods. Percent composition is calculated on oven-dry or commercial moisture regain basis.

13.3 *Apparatus*:

13.3.1 *Analytical Balance*, accurate to 0.1 mg.

13.3.2 *Fritted Glass Filtering Crucible*, of medium porosity.

13.3.3 *Weighing Bottle*, large enough to hold the fritted glass crucible.

13.3.4 *Erlenmeyer Flask*, glass-stoppered, having a capacity of 500 mL.

13.3.5 *Ice Bath*, in container large enough to submerge the Erlenmeyer flask.

13.3.6 *Suction Flask*, with adapter.

13.3.7 *Oven*, maintained at 105 to 110°C.

13.3.8 *Desiccator*, with anhydrous calcium sulfate ($CaSO_4$) or equivalent.

13.4 *Reagents and Materials*:

13.4.1 *Acetone − Water Mixture*—Mix 80 volumes of acetone with 20 volumes of water at 0 to 2°C in the 500-mL flask, cooling by means of an ice bath.

NOTE 8—**Precaution:** In addition to other precautions, acetone is flammable and should be handled away from any ignition source. It is also very volatile and breathing the vapors should be avoided. The use of a hood is strongly recommended.

NOTE 9—Some types of acetate are not completely soluble in an acetone-water mixture, but are soluble in glacial acetic acid.

NOTE 10—If there are no modacrylic fibers present, the analysis may be carried out using 100 % acetone at room temperature (15 to 25°C).

13.5 *Procedure*:

13.5.1 Weigh the dried specimen and weighing container to the nearest 0.001 g. Label as Tare and Sample.

13.5.2 Place the specimen in an Erlenmeyer flask and cover with 100 mL of the acetone − water mixture at 0 to 2°C (Note 10).

13.5.3 Agitate vigorously the flask containing the specimen for 15 min at a room temperature of 15 to 30°C.

13.5.4 Transfer the contents of the flask to a fritted glass funnel fitted into a suction flask.

13.5.5 Filter out the residual fiber.

13.5.6 Repeat 13.5.2-13.5.5 two additional times.

13.5.7 Dry the residue on the funnel by suction and wash with several portions of 70°C water.

13.5.8 Collect the residue from above and blot dry on paper towels. Exercise care not to lose any fibers in the process. Examine for complete extraction and, if necessary, repeat 13.5.2-13.5.7.

13.5.9 Place the specimen in an open weighing container and dry at 105 to 110°C for at least 1.5 h. Do not put wet specimens in the oven while other specimens are drying.

13.5.10 Remove the weighing container from the oven and immediately place it uncovered into a desiccator over $CaSO_4$, allowing it to cool for at least 30 min.

13.5.11 Place cover on specimen container, remove the assembly from desiccator, and weigh to the nearest 0.001 g. Label as Tare and Treated.

13.5.12 Remove the specimen and weigh the empty container to the nearest 0.001 g. Label as Tare.

13.6 *Calculations*:

13.6.1 Calculate the percent composition, to the nearest 0.1 %, of dry sample using the following equations:

$$\text{Fiber dissolved, \% } dry = 100\,(F - G/F) \tag{7}$$

where:
F = original dry clean specimen mass, g, (Tare and Sample) − (Tare) from 13.5.1 and 13.5.12, and
G = extracted dry clean residual specimen mass, g, (Tare and Treated) − (Tare) from 13.5.11 and 13.5.12.

$$\text{Fiber undissolved, \% dry} = 100 - \text{Fiber dissolved, \% dry} \tag{8}$$

13.6.2 If requested, calculate the percent composition, to the nearest 0.1 % on a commercial moisture regain basis (MRB) using the following equations:

$$G_1 = [(\text{Fiber dissolved, \% dry})(100 + MR_1)/100] \tag{9}$$

$$G_2 = [(\text{Fiber undissolved, \% dry})(100 + MR_2)/100] \tag{10}$$

where:
MR_1 = moisture regain % for the dissolved fiber, and
MR_2 = moisture regain % for the undissolved fiber.

$$\text{Fiber dissolved, \% } MRB = 100\,G_1/(G_1 + G_2) \tag{11}$$

$$\text{Fiber undissolved, \% } MRB = 100 - \text{\%Fiber dissolved, \% } MRB$$

13.7 *Report*:

13.7.1 State that the specimens were tested as directed in Chemical Test Method 1 of Test Methods D 629.

13.7.2 Describe the material(s) or product(s) sampled and the method of sampling used.

13.7.3 Report the individual and the average values of percent dissolved and undissolved fiber, based on the clean, dry sample.

13.7.4 Report the calculated composition, moisture regain basis, if requested.

13.8 *Precision and Bias*:

13.8.1 *Precision*—An interlaboratory study has been organized. The analysis data will be completed within the year 1999 and the precision statement will be balloted for inclusion in the test method by the end of the year 2000.

13.8.2 *Bias*—The procedure in Test Methods D 629 for measuring the fiber content of textiles by chemical test methods has no known bias because the values of the fiber content of textiles by chemical test methods can be defined only in terms of a test method.

14. Chemical Test Method No. 2—Modacrylic Mixed With Cellulosic Fibers or Wool

14.1 *Scope*—This test method is applicable to mixtures of most modacrylic with cellulosic fibers or wool.

14.2 *Summary of Method*—Modacrylic fibers are dissolved in γ-butyrolactone leaving cellulosic fibers or wool. Percent composition is calculated on oven-dry or commercial regain basis.

14.3 *Apparatus*:

14.3.1 *Analytical Balance*, accurate to 0.1 mg.

14.3.2 *Weighing Container*, glass or metal.

14.3.3 *Beakers and Flasks*, and other laboratory glassware, as required.

14.3.4 *Oven*, maintained at 105 to 110°C.

14.3.5 *Sieve*, stainless steel, 180 µm (80 mesh).

14.3.6 *Desiccator*, with anhydrous calcium sulfate ($CaSO_4$) or equivalent.

14.4 *Reagents and Materials*:

14.4.1 *γ-Butyrolactone*, American Chemical Society reagent.

NOTE 11—**Precaution:** In addition to other precautions, avoid contact of γ-butyrolactone with eyes, skin, or clothing. γ-butyrolactone causes irritation. Wear safety equipment when using this solvent.

14.5 *Procedure*:

14.5.1 Weigh the dried specimen and weighing container to the nearest 0.001 g. Label as Tare and Sample.

14.5.2 Place the specimen in a 250-mL beaker and cover with 100 mL of γ-butyrolactone.

14.5.3 Let stand for 60 ± 5 min at a room temperature of 15 to 30°C, stirring every 5 min.

14.5.4 Decant the liquid through the 550-µm (30-mesh) strainer returning the specimen to the beaker.

14.5.5 Repeat 14.5.2-14.5.4 allowing the specimen to remain in γ-butyrolactone for 30 ± 5 min.

14.5.6 Wash the specimen in running water for at least 10 min. If the specimen is fabric, wash it in the original beaker. If the specimen is fibrous or in small pieces, wash it in the 180-µm (80-mesh) sieve.

14.5.7 Collect the residue from above and blot dry on paper towels. Exercise care not to lose any loose fibers in the process. Examine for complete extraction and, if necessary, repeat 14.5.2-14.5.6.

14.5.8 Place the specimen in an open weighing container and dry at 150 to 110°C for at least 1.5 h. Do not put wet specimens in the oven while other specimens are drying.

14.5.9 Remove the weighing container from the oven and immediately place it uncovered into a desiccator over $CaSO_4$, allowing it to cool for at least 30 min.

14.5.10 Place cover on specimen container, remove assembly from desiccator and weigh to nearest 0.001 g. Label as Tare and Treated.

14.5.11 Remove the specimen and weigh the empty container to the nearest 0.001 g. Label as Tare.

14.6 *Calculations*:

14.6.1 Calculate the percent composition, to the nearest 0.1 %, of dry sample using the following equations:

$$\text{Fiber dissolved, \% dry} = 100\,(F - G/F) \tag{13}$$

where:
F = original dry clean specimen mass, g, (Tare and Sample) − (Tare) from 14.5.1 and 14.5.11.
G = extracted dry clean residual specimen mass, g, (Tare and Treated) − (Tare) from 14.5.10 and 14.5.11.

$$\text{Fiber undissolved, \% dry} = 100 - \text{Fiber dissolved, \% dry} \qquad (14)$$

14.6.2 If requested, calculate the percent composition, to the nearest 0.01 % on a commercial moisture regain basis (MRB) using the following equations:

$$G_1 = [(\text{Fiber dissolved, \% dry}) \cdot (100 + MR_1)/100] \qquad (15)$$

$$G_2 = [(\text{Fiber undissolved, \% dry}) \cdot (100 + MR_2)/100] \qquad (16)$$

where:

MR_1 = moisture regain % for dissolved fiber, and

MR_2 = moisture regain % for the undissolved fiber.

$$\text{Fiber dissolved, \% } MRB = 100\ G_1/(G_1 + G_2) \qquad (17)$$

$$\text{Fiber undissolved, \% } MRB = 100 - \%\ \text{Fiber dissolved, \% } MRB \qquad (18)$$

14.7 *Report*:

14.7.1 State that the specimens were tested as directed in Chemical Test Method No. 2 of Test Methods D 629.

14.7.2 Describe the material(s) and product(s) sampled and the method of sampling used.

14.7.3 Report the individual and average values of percent dissolved and undissolved fiber, based on the clean, dry sample.

14.7.4 Report the calculated composition, moisture regain basis, if requested.

14.8 *Precision and Bias*:

14.8.1 *Precision*—An interlaboratory study has been organized. The analysis data will be completed within the year 1999 and the precision statement will be balloted for inclusion in the test method by the end of the year 2000.

14.8.2 *Bias*—See 13.8.2

15. Chemical Test Method No. 3—Nylon 6 or Nylon 6–6 Mixed With Natural Fiber or Rayon

15.1 *Scope*—This test method is applicable to mixtures of Nylon 6 or Nylon 6–6 with cotton, rayon, or wool.

15.2 *Summary of Method*—Nylon fibers are dissolved in 90 % formic acid, and the other fibers present (rayon, wool, or cotton) are determined quantitatively by gravimetric methods. Percent composition is calculated either on oven dry or commercial moisture regain basis.

15.3 *Apparatus*:

15.3.1 *Analytical Balance*, accurate to 0.1 mg.

15.3.2 *Weighing Container*, glass or metal.

15.3.3 *Beakers and Flasks*, and other laboratory glassware, as required.

15.3.4 *Oven*, maintained at 105 to 110°C.

15.3.5 *Sieve*, stainless steel, 180 µm (80 mesh).

15.3.6 *Desiccator*, with anhydrous calcium sulfate ($CaSO_4$) or equivalent.

15.4 *Reagents and Materials*:

15.4.1 *Formic acid*, (HCOOH), 90 % purified.

NOTE 12—**Precaution:** In addition to other precautions, formic acid is corrosive to human tissue. Wear protective equipment and handle with care. Work with adequate ventilation.

15.5 *Procedure*:

15.5.1 Weigh the dried specimen and weighing container to the nearest 0.001 g. Label as Tare and Sample.

15.5.2 Place the specimen in a 250-mL beaker and cover with 100 to 150 mL of HCOOH (See Note 12).

15.5.3 Let stand for 30 ± 5 min at a room temperature of 15 to 30°C, stirring every 10 min.

15.5.4 Decant the liquid through the 550-µm (30 mesh) strainer, returning the specimen to the beaker.

15.5.5 Repeat 15.5.2-15.5.4.

15.5.6 Wash the specimen in running water for at least 10 min. If the specimen is fabric, wash it in the original beaker. If the specimen is fibrous or in small pieces, wash it in the 180-µm (80-mesh) sieve.

15.5.7 Collect the residue from above and blot dry on paper towels. Exercise care not to lose any loose fiber in this process. Examine for complete extraction and, if necessary, repeat 15.5.2-15.5.6.

15.5.8 Place the specimen in an open weighing container and dry at 105 to 110°C for at least 1.5 h. Do not put wet specimens in the oven while other specimens are drying.

15.5.9 Remove the weighing container from the oven and immediately place it uncovered into a desiccator over $CaSO_4$, allowing it to cool for at least 30 min.

15.5.10 Place cover on specimen container, remove the assembly from desiccator, and weigh to the nearest 0.001 g. Label as Tare and Treated.

15.5.11 Remove the specimen and weigh the empty container to the nearest 0.001 g. Label as Tare.

15.6 *Calculation*:

15.6.1 Calculate the percent composition, to the nearest 0.1 %, of dry sample using the following equations:

$$\text{Fiber dissolved, \% dry} = 100\ (F - G/F) \qquad (19)$$

where:

F = original dry clean specimen mass, g, (Tare and Sample) − (Tare) from 15.5.1 and 15.5.11, and

G = extracted dry clean residual specimen mass, g, (Tare and Treated) − (Tare) from 15.5.10 and 15.5.11.

$$\text{Fiber undissolved, \% dry} = 100 - \text{Fiber dissolved, \% dry} \qquad (20)$$

15.6.2 If requested, calculate the percent composition, to the nearest 0.1 %, on a commercial moisture regain basis (MRB) using the following equations.

$$G_1 = [(\text{Fiber dissolved, \% dry}) \cdot (100 + MR_1)/100] \qquad (21)$$

$$G_2 = [(\text{Fiber undissolved, \% dry}) \cdot (100 + MR_2)/100] \qquad (22)$$

where:

MR_1 = moisture regain, %, for the dissolved fiber, and

MR_2 = moisture regain, %, for the undissolved fiber.

$$\text{Fiber dissolved, \% } MRB = 100\ G_1/(G_1 + G_2) \qquad (23)$$

$$\text{Fiber undissolved, \% } MRB = 100 - \%\ \text{Fiber dissolved, \% } MRB \qquad (24)$$

15.7 *Report*:

15.7.1 State that the specimens were tested as directed in Chemical Test Method No. 3 of Test Methods D 629.

15.7.2 Describe the material(s) or product(s) sampled and the method of sampling used.

15.7.3 Report the individual and average values of percent dissolved and undissolved fiber, based on the clean, dry sample.

15.7.4 Report the calculated composition, moisture regain basis, if requested.

15.8 *Precision and Bias*:

15.8.1 *Precision*—An interlaboratory study has been organized. The analysis data will be completed within the year 1999 and the precision statement will be balloted for inclusion in the test method by the end of the year 2000.

15.8.2 *Bias*—See 13.8.2

16. Chemical Test Method No. 4—Rayon Mixed With Cotton

16.1 *Scope*—This test method is applicable to mixtures of rayon with cotton.

16.2 *Summary of Method*—Rayon fibers are dissolved in 59.5 % sulfuric acid leaving cotton fibers. Percent composition is calculated either on oven-dry or commercial moisture regain basis.

16.3 *Apparatus*:

16.3.1 *Analytical Balance*, accurate to 0.1 mg.

16.3.2 *Weighing Container*, glass or metal.

16.3.3 *Beakers and Flasks*, and other laboratory glassware, as required.

16.3.4 *Oven*, maintained at 105 to 110°C.

16.3.5 *Sieve*, stainless steel, 180 µm (80 mesh).

16.3.6 *Desiccator*, with anhydrous calcium sulfate ($CaSO_4$) or equivalent.

16.4 *Reagents and Materials*:

16.4.1 *Sulfuric Acid* (H_2SO_4)—American Chemical Society grade, 59.5 ± 0.5 % weight mass, specific gravity 1.493 ± 0.027 Mg/m^3 at 20°C. Add sulfuric acid (density 1.84 Mg/m^3) slowly to water in the ratio of 1 mL acid to 1.25 mL water.

16.4.2 *Sodium Bicarbonate* ($NaHCO_3$)—Two percent prepared by adding 20 g of $NaHCO_3$ to 500 mL water and diluting to 1 L.

NOTE 13—**Precaution:** In addition to other precautions, sulfuric acid is corrosive to human tissue. Wear protective equipment and handle with care.

16.5 *Procedure*:

16.5.1 Weigh the dried specimen and weighing container to the nearest 0.001 g. Label as Tare and Sample.

16.5.2 Place the specimen (weighing approximately 1.0 g) in a 500-mL beaker and cover with 100 to 150 mL of 59.5 % H_2SO_4.

16.5.3 Let stand for 15 ± 2 min at a room temperature of 15 to 30°C, stirring every 5 min.

16.5.4 Decant the liquid through the 550-µm (30-mesh) strainer returning the specimen to the beaker.

16.5.5 Repeat 16.5.2-16.5.4 but allow the specimen to remain in the acid for 30 ± 5 min.

16.5.6 Repeat 16.5.2-16.5.4 but allow the specimen to remain in the acid for 5 ± 1 min.

16.5.7 Wash the specimen in running water for at least 10 min. If the specimen is fabric, wash it in the original beaker. If the specimen is fibrous or in small pieces, wash it in the 180-µm (80-mesh) sieve.

16.5.8 Place the specimen in a 2 % $NaHCO_3$ solution and allow it to stand for at least 5 min, then repeat 16.5.7.

16.5.9 Collect the residue from above and blot dry on paper towels. Exercise care not to lose any loose fiber in the process. Examine for complete extraction.

16.5.10 Place the specimen in an open weighing container and dry at 105 to 110°C for at least 1.5 h. Do not put wet specimens into the oven while other specimens are drying.

16.5.11 Remove the weighing container from the oven immediately and place it uncovered into a desiccator over $CaSO_4$, allowing it to cool for at least 30 min.

16.5.12 Place cover on specimen container, remove the assembly from desiccator, and weigh to nearest 0.001 g. Label as Tare and Treated.

16.5.13 Remove the specimen and weigh the empty container to the nearest 0.001 g. Label as Tare.

16.6 *Calculations*:

16.6.1 Calculate the percent composition, to the nearest 0.1 %, of dry sample using the following equations:

$$\text{Fiber dissolved, \% dry} = 100\ (F - G/F) \qquad (25)$$

where:

F = original dry clean specimen mass, g, (tare and sample) − (Tare) from 16.5.1 and 16.5.12, and
G = original dry clean specimen mass, g, (tare and treated) − (Tare) from 16.5.11 and 16.5.12.

$$\text{Fiber undissolved, \% dry} = 100 - \text{Fiber dissolved, \% dry} \qquad (26)$$

16.6.2 If requested, calculate the percent composition to the nearest 0.1 % on a commercial regain basis (MRB) using the following equations:

$$G_1 = [(\text{Fiber dissolved, \% dry})\cdot (100 + MR_1/100] \qquad (27)$$

$$G_2 = [(\text{ Fiber undissolved, \% dry}) \cdot (100 + MR_2/100] \qquad (28)$$

where:

MR_1 = moisture regain % for the dissolved fiber, and
MR_2 = moisture regain % for the undissolved fiber.

$$\text{Fiber dissolved, \% } MRB = 100\ G_1/(G_1 + G_2) \qquad (29)$$

$$\text{Fiber undissolved, \% } MRB = 100 - \text{\% Fiber dissolved, \% } MRB \qquad (30)$$

16.6.3 Cotton is not completely insoluble in H_2SO_4 (59.5 %). Furthermore, a small amount of rayon remains undissolved in this solvent. To allow for this bias, correct the observed composition using Eq 30 or Eq 31.

$$\text{Corrected cotton, \%} = (100\ a\ \ G/F) - 1.6 \qquad (31)$$

where:

For a raw cotton, a = 1.062.
For bleached cotton, a = 1.046.

$$\text{Corrected regenerated cellulose rayon, \%} = 100 - \text{cotton, \% (corrected)} \qquad (32)$$

16.7 *Report*:

16.7.1 State that the specimens were tested as directed in Chemical Test Method No. 4 of Test Methods D 629.

16.7.2 Describe the material(s) or product(s) sampled and the method of sampling used.

16.7.3 Report the individual and average values of percent dissolved and undissolved fiber, based on the clean, dry sample.

16.7.4 Report the calculated composition, moisture regain basis, if requested.

16.8 *Precision and Bias*:

16.8.1 *Precision*—An interlaboratory study has been organized. The analysis data will be completed within the year 1999 and the precision statement will be balloted for inclusion in the test method by the end of the year 2000.

16.8.2 *Bias*—See 13.8.2

17. Chemical Test Method No. 5—Wool or Polyester Mixed With Cellulosic Fibers or Silk

17.1 *Scope*—This test method is applicable to mixtures of wool or polyester mixed with cotton, linen, acetate, silk, and rayon.

17.2 *Summary of Method*—Cellulosic fibers and silk are dissolved in 70 % sulfuric acid, leaving wool or polyester as a residue. Percent composition is calculated either on oven-dry or commercial moisture regain basis.

17.3 *Apparatus*:

17.3.1 *Analytical Balance*, accurate to 0.1 mg.

17.3.2 *Weighing Container*, glass or metal.

17.3.3 *Beakers and Flasks*, and other laboratory glassware, as required.

17.3.4 *Oven*, maintained at 105 to 110°C.

17.3.5 *Sieve*, stainless steel, 180 µm (80 mesh).

17.3.6 *Desiccator*, with anhydrous calcium sulfate ($CaSO_4$) or equivalent.

17.4 *Reagents and Materials*:

17.4.1 *Sulfuric Acid* (H_2SO_4)—American Chemical Society grade, 70 ± 1 % by mass, density 1.6105 ± 0.0116 Mg/m^3 at 20°C. Add sulfuric acid (density 1.84 Mg/m^3) slowly to water in the ratio of 1 mL acid to 0.77 mL of water.

17.4.2 *Sodium Bicarbonate* ($NaHCO_3$)—Two percent prepared by adding 20 g of $NaHCO_3$ to 500 mL water and diluting to 1 L.

NOTE 14—**Precaution:** In addition to other precautions, sulfuric acid is corrosive to human tissue. Wear protective equipment and handle with care.

17.5 *Procedure*:

17.5.1 Weigh the dried specimen and weighing container to the nearest 0.001 g. Label as Tare and Sample.

17.5.2 Place the specimen in a 250-mL beaker and cover with 100 to 150 mL to 70 % H_2SO_4.

17.5.3 Let stand for 15 ± 2 min at a room temperature of 15 to 30°C, stirring every 5 min.

17.5.4 Decant the liquid through the 550-µm (30-mesh) strainer, returning the specimen to the beaker.

17.5.5 Repeat 17.5.2-17.5.4 and allow the specimen to remain in the acid for 30 ± 5 min.

17.5.6 Wash the specimen in running water for at least 10 min. If the specimen is fabric, wash it in the original beaker. If the specimen is fibrous or in small pieces, wash it in the 180-µm (80-mesh) sieve.

17.5.7 Place the specimen in a 2 % $NaHCO_3$ and allow it to stand for at least 5 min, then repeat 17.5.6.

17.5.8 Collect the residue from above and blot dry on paper towels. Exercise care not to lose any loose fiber in this process. Examine for complete extraction and, if necessary, repeat 17.5.2-17.5.7.

17.5.9 Place the specimen in an open weighing container and dry at 105 to 110°C for at least 1.5 h. Do not put wet specimens in the oven while other specimens are drying.

17.5.10 Remove the weighing container from the oven immediately and place it uncovered into a desiccator over $CaSO_4$, allowing it to cool for at least 30 min.

17.5.11 Place a cover on the specimen container, remove the assembly from desiccator, and weigh to nearest 0.001 g. Label as Tare and Treated.

17.5.12 Remove the specimen and weigh the empty container to the nearest 0.001 g. Label as Tare.

17.6 *Calculations*:

17.6.1 Calculate the percent composition, to the nearest 0.1 %, of dry sample using the following equations (See Note 15):

$$\text{Fiber dissolved, \% dry} = 100\ (F - G/F) \tag{33}$$

where:

F = original dry clean specimen mass, g, (Tare and Sample) − (Tare) from 17.5.1 and 17.5.12, and

G = extracted dry clean specimen mass, g, (Tare and Treated) − (Tare) from 17.5.11 and 17.5.12.

$$\text{Fiber undissolved, \% dry} = 100 - \text{Fiber dissolved, \% dry} \tag{34}$$

17.6.2 If requested, calculate the percent composition to the nearest 0.1 % on a commercial moisture regain basis (MRB) using the following equations (See Note 15):

$$G_1 = [(\text{Fiber dissolved, \% dry}) \cdot (100 + MR_1)/100] \tag{35}$$

$$G_2 = [(\text{Fiber undissolved, \% dry}) \cdot (100 + MR_2)/100] \tag{36}$$

where:

MR_1 = moisture regain % for the dissolved fiber, and

MR_2 = moisture regain % for the undissolved fiber.

$$\text{Fiber dissolved, \% } MRB = 100\ G_1/(G_1 + G_2) \tag{37}$$

$$\text{Fiber undissolved, \% } MRB = 100 - \text{\% Fiber dissolved, \% } MRB \tag{38}$$

NOTE 15—Investigation had disclosed that when reprocessed or re-used wool, as defined in the Wool Products Labeling Act of 1939[7], is present in the textile the amount of wool determined by this method may be lower than the actual amount present by as much as 4 % of the amount of the reprocessed or re-used wool present. The effect of the acid treatment appears to depend upon the previous history of the wool fibers and is too uncertain to apply a uniform correction factor. In the case of virgin wool, no correction factor is required.

17.7 *Report*:

17.7.1 State that the specimens were tested as directed in Chemical Test Method No. 5 of Test Methods D 629.

17.7.2 Describe the material(s) or product(s) sampled and the method of sampling used.

17.7.3 Report the individual and average values of percent dissolved and undissolved fiber, based on the clean, dry sample.

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[7] Act of Congress, "Wool Products Labelling Act of 1939," 76th Congress, Third Session, approved Oct. 14, 1939.

17.7.4 Report the calculated composition, moisture regain basis, if requested.

17.8 *Precision and Bias*:

17.8.1 *Precision*—An interlaboratory study has been organized. The analysis data will be completed within the year 1999 and the precision statement will be balloted for inclusion in the test method by the end of the year 2000.

17.8.2 *Bias*—See 13.8.2.

18. Chemical Test Method No. 6—Polyester or Acrylic Mixed With Wool

18.1 *Scope*—This test method is applicable to mixtures of polyester or acrylic and wool.

18.2 *Summary of Method*—Wool fibers are dissolved in dilute sodium hypochlorite. Percent composition is calculated either on oven-dry or commercial moisture regain basis.

18.3 *Apparatus*:

18.3.1 *Analytical Balance*, accurate to 0.1 mg.

18.3.2 *Weighing Container*, glass or metal.

18.3.3 *Oven*, maintained at 105 to 110°C.

18.3.4 *Sieve*, stainless steel, 180 µm (80 mesh).

18.3.5 *Desiccator*, with anhydrous calcium sulfate ($CaSO_4$) or equivalent.

18.4 *Reagents and Materials*:

18.4.1 *Sodium Hypochlorite* (NaOCl), 5.0 % available chlorine by weight.

18.4.1.1 Since NaOCl solutions lose strength on standing, they should be standardized frequently. Determine the available chlorine content of NaOCl solutions as follows: dilute a 10-mL sample of the NaOCl solution to be tested to 250 mL with water in a volumetric flask. Add 25 mL of this solution to an Erlenmeyer flask with 3 to 5 mL of a 10 % solution of potassium iodide (KI) and 2 to 3 mL of acetic acid (CH_3COOH). Then titrate this solution with 0.1 *N* sodium thiosulfate ($Na_2S_2O_3$) solution until the yellow color of the iodine is nearly destroyed. Add 5 mL of starch solution and titrate until the blue color entirely disappears. Calculate the percentage of available chlorine by weight using the following equation:

$$\text{Available chlorine, \%} - [(A \times 3.5)/B] \tag{39}$$

where:

A = total millilitres of 0.1 N $Na_2S_2O_3$ solution required for titration of the sample, and
B = grams of sample used.

NOTE 16—**Precaution:** In addition to other precautions, sodium hypochlorite is corrosive to the eyes. Wear protective equipment and handle with care. Work with adequate ventilation.

18.5 *Procedure*:

18.5.1 Weigh the dried specimen and weighing container to the nearest 0.001 g. Label as tare and sample.

18.5.2 Place the specimen in a 250-mL beaker and cover with 100 to 150 mL of NaOCl.

18.5.3 Let stand for 30 ± 5 min at a room temperature of 15 to 30°C, stirring every 10 min.

18.5.4 Decant the liquid through the 550-µm (30-mesh) strainer returning the specimen to the beaker.

18.5.5 Repeat 18.5.2-18.5.4.

18.5.6 Wash the specimen in running water for at least 10 min. If the specimen is fabric, wash it in the original beaker. If the specimen is fibrous or in small pieces, wash it in the 180-µm (80-mesh) sieve.

18.5.7 Collect the residue from above and blot dry on paper towels. Exercise care not to lose any loose fibers in this process. Examine for complete extraction and, if necessary, repeat 18.5.2-18.5.6.

18.5.8 Place the specimen in an open weighing container and dry at 105 to 110°C for at least 1.5 h. Do not put wet specimens in the oven while other specimens are drying.

18.5.9 Remove the weighing container from the oven and immediately place it uncovered into a desiccator over $CaSO_4$, allowing it to cool for at least 30 min.

18.5.10 Place the cover on the specimen container, remove the assembly from the desiccator, and weigh to the nearest 0.001 g. Label as tare and treated.

18.5.11 Remove the specimen and weigh the empty container to the nearest 0.001 g. Label as tare.

18.6 *Calculation*:

18.6.1 Calculate the percent composition, to the nearest 0.1 %, of dry sample using the following equations:

$$\text{Fiber dissolved, \% dry} = 100\,(F - G/F) \tag{40}$$

where:

F = original dry clean specimen mass, g, (Tare + Sample) − (Tare) from 18.5.1 and 18.5.11, and
G = extracted dry clean residual specimen mass, g, (Tare + Treated) − (Tare) from 18.5.10 and 18.5.11.

$$\text{Fiber undissolved, \% dry} = 100 - \text{Fiber dissolved, \% dry} \tag{41}$$

18.6.2 If requested, calculate the percent composition, to the nearest 0.1 %, on a commercial moisture regain basis (MRB) using the following equations.

$$G_1 = [(\text{Fiber dissolved, \% dry}) \cdot (100 + MR_1)/100] \tag{42}$$

$$G_2 = [(\text{ Fiber undissolved, \% dry}) \cdot (100 + MR_2)/100] \tag{43}$$

where:

MR_1 = moisture regain % for the dissolved fiber, and
MR_2 = moisture regain % for the undissolved fiber.

$$\text{Fiber dissolved, \% } MRB = 100\,G_1/(G_1 + G_2) \tag{44}$$

$$\text{Fiber undissolved, \% } MRB = 100 - \text{\% Fiber dissolved, \% } MRB \tag{45}$$

18.7 *Report*:

18.7.1 State that the specimens were tested as directed in Test Method No. 6 of Test Methods D 629.

18.7.2 Describe the material(s) or product(s) sampled and the method of sampling used.

18.7.3 Report the individual and average values of % dissolved and undissolved fiber, based on the clean, dry sample.

18.7.4 Report the calculated composition, moisture regain basis, if requested.

18.8 *Precision and Bias*:

18.8.1 *Precision*—The precision of Chemical Test Method No. 6 in Test Methods D 629 is being established.

18.8.2 *Bias*—See 13.8.2.

19. Chemical Test Method No. 7—Natural Cellulosic Material and Rayon Mixed with Acrylic, Modacrylic, and Polyester

19.1 *Scope*—This test method is intended for the separation of natural cellulosic material and rayon from mixtures containing acrylic, modacrylic, or polyester.

19.2 *Summary of Method*—Cellulosic materials are dissolved in a cuprammonia solution leaving acrylic, modacrylic, or polyester as a residue. Percent composition is calculated either on oven dry or commercial regain basis.

19.3 *Apparatus*:

19.3.1 *Analytical Balance*, accurate to 0.1 mg.

19.3.2 *Weighing Container*, glass or metal.

19.3.3 *Beakers and Flasks*, and other laboratory glassware, as required.

19.3.4 *Oven*, maintained at 105 to 110°C.

19.3.5 *Sieve*, stainless steel, 180 µm (80 mesh).

19.3.6 *Desiccator*, with anhydrous calcium sulfate ($CaSO_4$) or equivalent.

19.4 *Reagents and Materials*:

19.4.1 *Cupric Sulfate* ($CuSO_4 \cdot 5H_2O$)—American Chemical Society grade; sodium hydroxide (NaOH), analytical reagent; and ammonium hydroxide (NH_4OH), American Chemical Society grade.

19.4.2 Dissolve 7.8 g of cupric sulfate in 100 mL of boiling water, and slowly add 1.5 % aqueous sodium hydroxide until precipitation is complete (noted by a clear liquor). Wash the light green precipitate well, and dissolve it in a minimum quantity of ammonium hydroxide or until all the solid is just dissolved. A bluish-purple color solution should develop.

NOTE 17—**Precaution:** In addition to other precautions, cuprammonia solution is corrosive to human tissue. Wear protective equipment and handle with care.

19.5 *Procedure*:

19.5.1 Weigh the dried specimen and weighing container to the nearest 0.001 g. Label as Tare and Sample.

19.5.2 Place the specimen in a covered 250-mL beaker and cover with 100 mL of cuprammonia solution.

19.5.3 Let stand for 30 ± 5 min at a room temperature of 15 to 30°C, stirring every 5 min.

19.5.4 Decant the liquid through the 550-µm (30-mesh) strainer, returning the specimen to the beaker.

19.5.5 Rinse the specimen in 75 mL of ammonia hydroxide and blot on paper towels.

19.5.6 Repeat 19.5.2-19.5.5.

19.5.7 Wash the specimen in running water for at least 10 min. If the specimen is fabric, wash it in the original beaker. If the specimen is fibrous or in small pieces, wash it in the 180-µm (80-mesh) sieve.

19.5.8 Collect the residue from above and blot dry on paper towels. Examine for complete extraction and, if necessary, repeat 19.5.2-19.5.6.

19.5.9 Place the specimen in an open weighing container and dry at 105 to 110°C for at least 1.5 h. Do not put wet specimens in the oven while other specimens are drying.

19.5.10 Remove the weighing container from the oven and immediately place it uncovered into a desiccator over $CaSO_4$, allowing it to cool for at least 30 min.

19.5.11 Place cover on specimen container, remove the assembly from the desiccator, and weight to the nearest 0.001 g. Label as Tare and Treated.

19.5.12 Remove the specimen and weigh the empty container to the nearest 0.001 g. Label as Tare.

19.6 *Calculation*:

19.6.1 Calculate the percent composition, to the nearest 0.1 %, of dry sample using the following equations:

$$\text{Fiber dissolved, \% dry} = 100\,(F - G/F) \tag{46}$$

where:

F = original clean dry specimen mass, g (Tare and Sample) − (Tare) from 19.5.1 and 19.5.12.

G = extracted clean residual specimen mass, g, (Tare and Treated) − (Tare) from 19.5.11 and 19.5.12.

$$\text{Fiber undissolved, \% dry} = 100 - \text{Fiber dissolved, \% dry} \tag{47}$$

19.6.2 If requested, calculate the percent composition, to the nearest 0.01 %, on a moisture regain basis (MRB) using the following equations:

$$G_1 = [(\text{Fiber dissolved, \% dry}) \cdot (100 + MR_1)/100] \tag{48}$$

$$G_2 = [(\text{ Fiber undissolved, \% dry}) \cdot (100 + MR_2)/100] \tag{49}$$

where:

MR_1 = moisture regain % for the dissolved fiber, and

MR_2 = moisture regain % for the undissolved fiber.

$$\text{Fiber dissolved, \% } MRB = 100G_1/(G_1 + G_2) \tag{50}$$

$$\text{Fiber undissolved, \% } MRB = 100 - \text{\% Fiber dissolved, \% } MRB$$

19.7 *Report*:

19.7.1 State that the specimens were tested as directed in Chemical Test Method No. 7 of Test Methods D 629.

19.7.2 Describe the material(s) or product(s) sampled and the method of sampling used.

19.7.3 Report the individual and average values of percent dissolved and undissolved fiber, based on the clean dry sample.

19.7.4 Report the calculated composition, moisture regain basis, if requested.

19.8 *Precision and Bias*:

19.8.1 *Precision*—An interlaboratory study has been organized. The analysis data will be completed within the year 1999 and the precision statement will be balloted for inclusion in the test method by the end of the year 2000.

19.8.2 *Bias*—See 13.8.2.

20. Chemical Test Method No. 8—Polyester Mixed With Olefin

20.1 *Scope*—This test method is applicable to mixtures of polyester and olefin.

20.2 *Summary of Method*—Olefin fibers are dissolved in boiling xylene. Percent composition is calculated either on oven-dry or commercial moisture regain basis.

20.3 *Apparatus*:

20.3.1 *Analytical Balance*, accurate to 0.1 mg.

20.3.2 *Weighing Container*, glass or metal.

20.3.3 *Oven*, maintained at 105 to 110°C.

20.3.4 *Sieve*, stainless steel, 180 µm (80 mesh).

20.3.5 *Desiccator*, with anhydrous calcium sulfate ($CaSO_4$) or equivalent.

20.3.6 *Hot plate*, low surface temperature type.

20.4 *Reagents and Materials*:

20.4.1 *Xylene*—Meta [$C_6H_4(CH_3)_2$], purified.

NOTE 18—**Precaution:** In addition to other precautions, xylene is a flammable defatting agent and also harmful if inhaled. Wear protective equipment and handle with care. Use only with adequate ventilation.

20.5 *Procedure*:

20.5.1 Weigh the dried specimen and weighing container to the nearest 0.001 g. Label as Tare and Sample.

20.5.2 Place the specimen in a 250-mL beaker and cover with 100 to 150 mL of xylene. Use only under a hood.

20.5.3 Heat to the boil and hold for 30 ± 5 min, stirring every 10 min.

20.5.4 Decant the liquid through the 550-µm (30-mesh) strainer returning the specimen to the beaker.

20.5.5 Repeat 20.5.2-20.5.4.

20.5.6 Wash the specimen in clear xylene. If the specimen is fabric, wash it in the original beaker. If the specimen is fibrous or in small pieces, wash it in the 180-µm (80-mesh) sieve.

20.5.7 Collect the residue from above and blot dry on paper towels. Exercise care not to lose any loose fabrics in this process. Examine for complete extraction and, if necessary, repeat 20.5.2-20.5.6. Allow to air dry, in a hood or on a steam table, until all traces of xylene have evaporated from the residue.

20.5.8 Place the specimen in an open weighing container and dry at 105 to 110°C for at least 1.5 h. Do not put wet specimens in the oven while other specimens are drying.

20.5.9 Remove the weighing container from the oven and immediately place it uncovered into a desiccator over $CaSO_4$, allowing it to cool for at least 30 min.

20.5.10 Place cover on specimen container, remove the assembly from desiccator, and weigh to the nearest 0.001 g. Label as Tare and Treated.

20.5.11 Remove the specimen and weigh the empty container to the nearest 0.001 g. Label as Tare.

20.6 *Calculation*:

20.6.1 Calculate the percent composition, to the nearest 0.1 %, of dry sample using the following equations.

$$\text{Fiber dissolved, \% dry} = 100\,(F - G/F) \tag{51}$$

where:

F = original dry clean specimen mass, g, (Tare and Sample) − (Tare) from 20.5.1 and 20.5.11.

G = extracted dry clean residual specimen mass, g, (Tare and Treated) − (Tare) from 20.5.10 and 20.5.11.

$$\text{Fiber undissolved, \% dry} = 100 - \text{Fiber dissolved, \% dry} \tag{52}$$

20.6.2 If requested, calculate the percent composition, to the nearest 0.1 % on a commercial moisture regain basis (MRB) using the following equations:

$$G_1 = [(\text{Fiber dissolved, \% dry})\cdot(100 + MR_1)/100] \tag{53}$$

$$G_2 = [(\text{Fiber undissolved, \% dry})\cdot(100 + MR_2)/100] \tag{54}$$

where:

MR_1 = moisture regain % for the dissolved fiber, and

MR_2 = moisture regain % for the undissolved fiber.

$$\text{Fiber dissolved, \% } MRB = 100\,G_1/(G_1 + G_2) \tag{55}$$

$$\text{Fiber undissolved, \% } MRB = 100 - \text{\% Fiber dissolved, \% } MRB \tag{56}$$

20.7 *Report*:

20.7.1 State that the specimens were tested as directed in Chemical Test Method No. 8 of Test Methods D 629.

20.7.2 Describe the material(s) or product(s) sampled and the method of sampling used.

20.7.3 Report the individual and average values of percent dissolved and undissolved fiber, based on the clean, dry sample.

20.7.4 Report the calculated composition, moisture regain basis, if requested.

20.8 *Precision and Bias*:

20.8.1 *Precision*—An interlaboratory study has been organized. The analysis data will be completed within the year 1999 and the precision statement will be balloted for inclusion in the test method by the end of the year 2000.

20.8.2 *Bias*—See 13.8.2.

21. Chemical Test Method No. 9—Polyester Mixed With Acetate or Nylon 6, 66

21.1 *Scope*—This method is applicable to mixtures of polyester with triacetate, acetate, or Nylon 6 or 66.

21.2 *Summary of Method*—Nylon or acetate fibers, or both, are dissolved in 90 % formic acid. Percent composition is calculated either on oven-dry or commercial moisture regain basis.

21.3 *Apparatus*:

21.3.1 *Analytical Balance*, accurate to 0.1 mg.

21.3.2 *Weighing Container*, glass or metal.

21.3.3 *Oven*, maintained at 105 to 110°C.

21.3.4 *Sieve*, stainless steel, 180 µm (80 mesh).

21.3.5 *Desiccator*, with anhydrous calcium sulfate ($CaSO_4$), or equivalent.

21.4 *Reagents and Materials*:

21.4.1 *Formic acid* (HCOOH), 90 % purified.

NOTE 19—**Precaution:** In addition to other precautions, formic acid is corrosive to human tissue. Wear protective equipment and handle with care. Work with adequate ventilation.

21.5 *Procedure*:

21.5.1 Weigh the dried specimen and weighing container to the nearest 0.001 g. Label as Tare and Sample.

21.5.2 Place the specimen in a 250-mL beaker and cover with 100 to 150 mL of HCOOH.

21.5.3 Let stand for 30 ± 5 min at a room temperature of 15 to 30°C, stirring every 10 min.

21.5.4 Decant the liquid through the 550-µm (30-mesh) strainer returning the specimen to the beaker.

21.5.5 Repeat 21.5.1-21.5.3.

21.5.6 Wash the specimen in running water for at least 10 min. If the specimen is fabric, wash it in the original beaker. If the specimen is fibrous or in small pieces, wash it in the 180-µm (80-mesh) sieve.

21.5.7 Collect the residue from above and blot dry on paper towels. Exercise care not to lose any loose fiber in the process. Examine for complete extraction and, if necessary, repeat 21.5.2-21.5.6.

21.5.8 Place the specimen in an open weighing container and dry at 105 to 110°C for at least 1.5 h. Do not put wet specimens in the oven while other specimens are drying.

21.5.9 Remove the weighing container from the oven and immediately place it uncovered into a desiccator over $CaSO_4$, allowing it to cool for at least 30 min.

21.5.10 Place cover on specimen container, remove the assembly from desiccator, and weigh to the nearest 0.001 g. Label as Tare and Treated.

21.5.11 Remove the specimen and weigh the empty can to the nearest 0.001 g. Label as Tare.

21.6 *Calculation*:

21.6.1 Calculate the percent composition, to the nearest 0.1 %, of dry sample using the following equations.

$$\text{Fiber dissolved, \% dry} = 100\,(F - G/F) \tag{57}$$

where:

F = original dry clean specimen mass, g, (Tare + Sample) − (Tare) from 21.5.1 and 21.5.11, and

G = extracted dry clean residual specimen mass, g, (Tare + Treated) − (Tare) from 21.5.10 and 21.5.11.

$$\text{Fiber undissolved, \% dry} = 100 - \text{Fiber dissolved, \% dry} \tag{58}$$

21.6.2 If requested, calculate the percent composition, to the nearest 0.1 % on a commercial moisture regain basis (MRB) using the following equations:

$$G_1 = [(\text{Fiber dissolved, \% dry})\cdot(100 + MR_1)/100] \tag{59}$$

$$G_2 = [(\text{ Fiber undissolved, \% dry})\cdot(100 + MR_2)/100] \tag{60}$$

where:

MR_1 = moisture regain % for the dissolved fiber, and

MR_2 = moisture regain % for the undissolved fiber.

$$\text{Fiber dissolved, \% } MRB = 100\,G_1/(G_1 + G_2) \tag{61}$$

$$\text{Fiber undissolved, \% } MRB = 100 - \text{\% Fiber dissolved, \% } MRB \tag{62}$$

21.7 *Report*:

21.7.1 State that the specimens were tested as directed in Chemical Test Method No. 9 of Test Methods D 629.

21.7.2 Describe the material(s) or product(s) sampled and the method of sampling used.

21.7.3 Report the individual and average values of percent dissolved and undissolved fiber, based on the clean dry sample.

21.7.4 Report the calculated composition, moisture regain basis, if requested.

21.8 *Precision and Bias*:

21.8.1 *Precision*—An interlaboratory study has been organized. The analysis data will be completed within the year 1999 and the precision statement will be balloted for inclusion in the test method by the end of the year 2000.

21.8.2 *Bias*—See 13.8.2.

22. Chemical Test Method No. 10—Acrylic Fiber or Linear Spandex Mixed With Nylon or Polyester

22.1 *Scope*—This test method is applicable to mixtures of acrylic fiber or linear spandex with nylon or polyester.

22.2 *Summary of Method*—Linear spandex fibers and acrylic fibers are dissolved in dimethylacetamide. Percent composition is calculated either on oven dry or commercial moisture regain basis.

22.3 *Apparatus*:

22.3.1 *Analytical Balance*, accurate to 0.1 mg.

22.3.2 *Weighing Container*, glass or metal.

22.3.3 *Oven*, maintained at 105 to 110°C.

22.3.4 *Sieve*, stainless steel, 180 µm (80 mesh).

22.3.5 *Desiccator*, with anhydrous calcium sulfate ($CaSO_4$) or equivalent.

22.3.6 *Steam bath*, on low temperature hot plate.

22.4 *Reagents and Materials*:

22.4.1 *N,N-Dimethylacetamide* (DMAC), reagent grade. (Note 21)

NOTE 20—**Precaution:** In addition to other precautions, *N,N*-Dimethylacetamide is a developmental toxin hazard. (Note 21). Wear protective equipment and handle with care. Work with adequate ventilation.

22.5 *Procedure*:

22.5.1 Weigh the dried specimen and weighing container to the nearest 0.001 g. Label as Tare and Sample.

22.5.2 Place the specimen in a 250-mL beaker and cover with 100 to 150 mL of DMAC.

22.5.3 Using a low temperature hot plate or steam bath, bring the DMAC to 65 to 70°C.

22.5.4 Let stand for 30 ± 5 min, stirring every 10 min.

22.5.5 Decant the liquid through the 550-µm (30-mesh) strainer returning the specimen to the beaker.

22.5.6 Repeat 22.5.2-22.5.5.

22.5.7 Wash the specimen in hot running water for at least 10 min. If the specimen is fabric, wash it in the original beaker. If the specimen is fibrous or in small pieces, wash it in the 180-µm (80-mesh) sieve.

22.5.8 Collect the residue from above and blot dry on paper towels. Exercise care not to lose any loose fibers in the process. Examine for complete extraction and, if necessary, repeat 22.5.2-22.5.7.

22.5.9 Place the specimen in an open weighing container and dry at 105 to 110°C for at least 1.5 h. Do not put wet specimens in the oven while other specimens are drying.

22.5.10 Remove the weighing container from the oven and immediately place it uncovered into a desiccator over $CaSO_4$, allowing it to cool for at least 30 min.

22.5.11 Place cover on specimen container, remove the assembly from desiccator, and weigh to the nearest 0.001 g. Label as Tare and Treated.

22.5.12 Remove the specimen and weigh the empty container to the nearest 0.001 g. Label as Tare.

22.6 *Calculations*:

22.6.1 Calculate the percent composition, to the nearest 0.1 %, of dry sample using the following equations.

$$\text{Fiber dissolved, \% dry} = 100\,(F - G/F) \tag{63}$$

where:

F = original dry clean specimen mass, g, (Tare and Sample) − (Tare) from 22.5.1 through 22.5.12, and

G = extracted dry clean residual specimen mass, g, (Tare and Treated) − (Tare) from 22.5.11 and 22.5.12.

$$\text{Fiber undissolved, \% dry} = 100 - \text{Fiber dissolved, \% dry} \tag{64}$$

22.6.2 If requested, calculate the percent composition, to the nearest 0.1 %, on a commercial moisture regain basis (MRB) using the following equations:

$$G_1 = [(\text{Fiber dissolved, \% dry}) \cdot (100 + MR_1)/100] \tag{65}$$

$$G_2 = [(\text{ Fiber undissolved, \% dry}) \cdot (100 + MR_2)/100] \tag{66}$$

where:

MR_1 = moisture regain % for the dissolved fiber, and

MR_2 = moisture regain % for the undissolved fiber.

$$\text{Fiber dissolved, \% } MRB = 100\ G_1/(G_1 + G_2) \tag{67}$$

$$\text{Fiber undissolved, \% } MRB = 100 - \text{\% fiber dissolved, \% } MRB \tag{68}$$

22.7 *Report*:

22.7.1 State that the specimens were tested as directed in Chemical Test Method No. 10 of Test Methods D 629.

22.7.2 Describe the material(s) or product(s) sampled and the method of sampling used.

22.7.3 Report the individual and average values of percent dissolved and undissolved fiber, based on the clean dry sample.

22.7.4 Report the calculated composition, moisture regain basis, if requested.

22.8 *Precision and Bias*:

22.8.1 *Precision*—An interlaboratory study has been organized. The analysis data will be completed within the year 1999 and the precision statement will be balloted for inclusion in the test method by the end of the year 2000.

22.8.2 *Bias*—See 13.8.2.

NOTE 21—Cross-linked spandex will not dissolve completely in DMAC. Cyclohexanone at the boil may be used with cross-linked spandex nylon combinations. Mechanical separation is recommended with cross-linked spandex polyester combinations as cyclohexanone partially dissolves polyester.

MICROSCOPICAL ANALYSIS

23. Scope

23.1 The following procedures may be used for the quantitative analysis of textiles containing two or more types of fibers that cannot be readily separated chemically. If the sample contains both animal and vegetable fibers, a mechanical separation (Section 10) or a chemical analysis (Sections 11-22) shall first be made. The percentages of the different types of fibers in these two groups shall then be determined by the appropriate method as directed in Sections 24 and 26. The accuracy obtainable with microscopical methods depends to a considerable extent upon the ability of the analyst to identify the different fibers present in the mixture and also upon the number of fibers examined and the number measured.

NOTE 22—Photomicrographs of common textile fibers are appended to AATCC Method 20.

24. Summary of Method

24.1 Mixtures of fibers such as cotton, hemp, linen, ramie, regular viscose rayon, also wool, camel hair, mohair, and related fibers are analyzed quantitatively by counting them after identification as directed in 27.1-27.5. The method is not suitable for use with fibers having no characteristic longitudinal markings for identification.

25. Apparatus

25.1 *Microscope and Accessories*, as specified in Test Method D 2130.

26. Sampling

26.1 *Fabrics*—A swatch of a fabric measuring at least 50 by 50 mm should be available. Count the number of yarns in both the warp and filling directions and select from each direction at random a number of yarns proportional to the fabric count. The combined number of warp and filling yarns should total at least 20 for fabrics containing mixtures of vegetable fibers and should total about 10 for fabric containing mixtures of hair fibers.

26.2 *Yarns*—Cut a sample of yarn at least 2 m in length at reasonable intervals into not less than twenty 50-mm sections for yarns containing mixtures of vegetable fibers and not less than ten 50-mm sections for yarns containing mixtures of hair fibers. If less than 2 m of yarn are available, cut the entire length into sections. For yarns containing animal fibers, prepare samples as directed in Test Method D 2130, 6.2.

26.3 *Vegetable Fibers*—Cut approximately 25 mm of each yarn or yarn section into lengths of 0.5 to 1 mm. The shorter the lengths, the easier it is to prepare a homogeneous fiber suspension. Collect the cut fibers on a paper of contrasting color and transfer to a 75 or 125-mL Erlenmeyer flask, or cut the fibers directly into the flask. Add sufficient water, stopper the flask, and shake the contents to obtain a uniform and fairly dense fiber suspension. Bringing the water to a boil or the addition of a few glass pellets facilitates the separation of the fibers.

26.4 *Hair Fibers*—From each yarn or yarn section, cut lengths of about 6 mm.

27. Procedure

27.1 *Preparation of Slide*:

27.1.1 *Vegetable Fibers*—With a Carborundum glass-marking pencil, draw two parallel lines about 25 mm apart across a glass slide. Using a wide-mouth pipet, draw 0.5 to 1 mL of the well-shaken suspension and place it between the two reference lines on the slide. The amount of liquid drawn from the flask is dependent upon the density of the suspension. Just sufficient liquid should be placed on the slide so that after evaporation a thin uniform film of fibers remains. After all moisture has evaporated from the slide, stain the fibers with Herzberg strain (Note 23) and cover with a cover glass. This method can not be used for dyed fibers unless the fibers are first stripped of the dyes.

NOTE 23—To prepare Herzberg stain, add Solution A to Solution B, stir, allow to stand overnight, then decant the clear liquid from the sediment, and add a leaf of iodine. Keep the reagent in a dark-colored glass bottle. For colors produced by the Herzberg stain (zinc-chloriodide reagent) on various textile fibers, see AATCC Test Method 20a.

Solution A:	
Zinc chloride	50 g
Water	25 mL
Solution B:	
Potassium iodide	5.5 g
Iodine	0.25 g
Water	12.5 mL

27.1.2 *Hair Fibers*—Mount each length (26.4) on a separate glass slide, using immersion oil as the mounting medium. Take care to separate and parallel the fibers before they are covered with a cover glass.

27.2 *Counting Fibers*:

27.2.1 *Vegetable Fibers*—Use a microscope equipped with a moveable stage and a cross-hair ocular, and having a magnification of 200 to 250×. Begin to count near either the upper or lower corners of the field and as the slide is moved slowly across the field in the horizontal direction, identify and count all fibers passing through the center of the cross-hairs. After each scan across the field, move the slide 1 to 2 mm vertically and identify and count the fibers as the field is again traversed. Repeat this procedure until the whole slide has been covered. In a similar manner, count the fibers by moving the slide vertically and spacing each scan 1 to 2 mm horizontally. The spacing after each scan is dependent upon the number of fiber sections on the slide. The combined horizontal and vertical counts should total at least 1000 fibers. If a fiber passes the cross-line more than once, record each passing.

NOTE 24—Linen fibers may be present in the fabric or yarn in the form of fiber bundles. Usually most of these bundles are reduced to single fibers during the preparation of the fiber suspension. If, however, the linen fibers appear on the slide in the form of a bundle, make an attempt to count each of the individual fibers in the bundle.

27.2.2 *Hair Fibers*—Identify and count all of the fibers in the yarn sections and classify them as wool, camel hair, mohair, etc., according to their microscopic characteristics. The specimen shall be large enough so that at least 1000 fibers can be counted.

27.3 *Determination of Fiber Diameter*— Measure the average diameter of 100 fibers for each type of fiber present in the mixture (Note 25). Use the fibers taken from the original yarns and not from the fiber suspensions. As the average diameters of vegetable fibers may vary over fairly wide limits, the average diameters of the fibers in the fabric being analyzed should be determined if accurate results are to be obtained. If maximum accuracy is not required, the composition may be calculated by using the data in Table 3.

NOTE 25—For more specific directions, refer to Test Method D 2130. In the case of fibers such as cotton, which are not circular or nearly circular in cross-section, the fiber diameter should be determined by the measurement of the average cross-section as directed in discontinued Method D 1444–77. This procedure is given in Annex A1.

TABLE 3 Density and Moisture Regain of Common Fiber Types

Fiber	Density, Mg/m^3	Moisture regain, %^{A}
Acetate	1.32	5.0
Acrylic	1.17	1.8
Aramid:		
Nomex	1.37	6.5
Kevlar	1.42	3.5
Cellulosic:		
Flax	1.50	8.75
Cotton	1.54	7.4
Hemp	1.48	12.0
Jute	1.48	13.75
Ramie	1.51	7.8
Modacrylic	1.28–1.37	0.5–3.0
Polyamides:		
Nylon 6	1.14	4.0
Nylon 6-6	1.14	4.2
Olefin:		
Polypropylene	0.90	0.1
Polyester:		
2GT	1.38	0.4
CHDM-T	1.24	0.2
Rayon	1.52	13.3
Silk	1.35	9.4
Spandex	1.20	1.3
Triacetate	1.30	3.5
Wool	1.31	13.0

^{A}The moisture regain values listed are typical measured values and are not necessarily commerical moisture regain values. For commerical moisture regain values refer to Table D 1909.

27.4 *Calculation*—Multiply the mean of the squares of the diameters of the fibers of each type by the specific gravity (see Table 5), and calculate the weight percentage from these figures as shown in the following example:

Example 1—A microscopical analysis shows the proportion of cotton, linen, and viscose rayon to be as follows:

	Cotton	Linen	Viscose Rayon
Number of fibers counted, N	521	458	235
Mean of squares of diameters of fibers, D^2	324.0	256.0	466.6
Specific gravity, S	1.55	1.50	1.53
$N \times D^2 \times S$	261 600	175 900	167 700
Percentage of fiber in sample	43	29	28

NOTE 26—*Fineness of Textile Fibers*—A chart and tables showing the comparative fineness together with average width ranges for various textile fibers are given in Table 4 and Table 3 as additional information for use in the identification of fibers.

TABLE 4 Fineness Ranges and Fiber Diameters of Various Textile Fibers^A

U.S. Wool Classification				
Wool Grades			Wool Top Grades	
Grade	Range for Average Fiber Diameter, µm^B	Standard Deviation, µm Maximum	Grade	Average Fiber Diameter Range, µm^C
Finer than 80s	under 17.70	3.59	Finer than 80s	under 18.10
80s	17.70 to 19.14	4.09	80s	18.10–19.59
70s	19.15 to 20.59	4.59	70s	19.60–21.09
64s	20.60 to 22.04	5.19	64s	21.10–22.59
62s	22.05 to 23.49	5.89	62s	22.60–24.09
60s	23.50 to 24.94	6.49	60s	24.10–25.59
58s	24.95 to 26.39	7.09	58s	25.60–27.09
56s	26.40 to 27.84	7.59	56s	27.10–28.59
54s	27.85 to 29.29	8.19	54s	28.60–30.09
50s	29.30 to 30.99	8.69	50s	30.10–31.79
48s	31.00 to 32.69	9.09	48s	31.80–33.49

TABLE 4 *Continued*

46s	32.70 to 34.39	9.59	46s	33.50–35.19
44s	34.40 to 36.19	10.09	44s	35.20–37.09
40s	36.20 to 38.09	10.69	40s	37.10–38.99
36s	38.10 to 40.20	11.19	36s	39.00–41.29
Coarser than 36s	over 40.20	...	Coarser than 36s	over 41.29

U.S. Mohair Classification

Mohair Grades			Mohair Top Grades	
Grade	Average Fiber Diameter Range, µm[D]	Standard Deviation, max, µm	Grade	Fineness Range, µm[E]
Finer than 40s	under 23.01	7.2	Finer than 40s	under 23.55
40s	23.01–25.00	7.6	40s	23.55–25.54
36s	25.01–27.00	8.0	36s	25.55–27.54
32s	27.01–29.00	8.4	32s	27.55–29.54
30s	29.01–31.00	8.8	30s	29.55–31.54
28s	31.01–33.00	9.2	28s	31.55–33.54
26s	33.01–35.00	9.6	26s	33.55–35.54
24s	35.01–37.00	10.0	24s	35.55–37.54
22s	37.01–39.00	10.5	22s	37.55–39.54
20s	39.01–41.00	11.0	20s	39.55–41.54
18s	41.01–43.00	11.5	18s	41.55–43.54
Coarser than 18s	over 43.01		Coarser than 18s	over 43.54

Hair Fibers and Silk

Miscellaneous Hair Fibers (1)		Silk (1)	
Fiber	Average Fineness, µm	Fiber	Average Fineness, µm
Vicuna	13.0 to 14.0	Cultivated silk	10.0 to 13.0
Cashmere	14.5 to 19.0	Tussah silk	28.5
Camel hair	17.0 to 23.0		
Alpaca	26.0 to 28.0		
Llama	20.0 to 27.0		

Vegetable Fiber (1)		Glass Fiber (2)			
Fiber	Average Fineness, µm	Filament Diameter Designation	Theoretical Diameter, [F] µm	Staple Fiber Diameter Designation	Average Diameter, µm
Cotton	16.0 to 21.0	D	5.3	E	7.1
Flax (linen)	15.0 to 17.0	E	7.4	G	9.7
Jute	15.0 to 20.0	G	9.0	J	11.4
Hemp	18.0 to 23.0				
Kapok	21.0 to 30.0				
Ramie	25.0 to 30.0				

Viscose Rayon, Acetate, Nylon, Vinyon, Polyester

	Theoretical Fiber Diameter, µm				
Filament Denier	Viscose Rayon	Acetate and Vinyon	Nylon	Polyester	Olefin
1	9.6	10.3	11.1	10.1	12.5
2	13.6	14.5	15.7	14.3	17.7
3	16.7	17.8	19.3	17.5	21.7
4	19.3	20.6	22.3	20.2	25.0
5	21.6	23.0	24.9	22.6	28.0
6	23.6	25.2	27.3	24.8	30.7
7	25.5	27.3	29.5	26.8	33.1
8	27.3	29.1	31.5	28.6	35.4
9	28.9	30.9	33.4	30.4	37.5
10	30.5	32.6	35.2	32.0	39.6
12	33.4	35.7	38.6	35.1	43.3
14	36.1	38.5	41.7	37.9	46.8
16	38.6	41.2	44.5	40.5	50.1
18	40.9	43.7	47.3	42.9	53.1
20	43.1	46.1	49.9	45.3	56.0

[A] *Source of Data:*
(1) Werner von Bergen and W. Krauss, "Textile Fiber Atlas," Textile Book Publishers, Inc., New York, NY (1949).
(2) Owens-Corning Fiberglass Corp.

[B] Specification D 419 (Replaced by Specification D 3991).

[C] Specification D 472 (Replaced by Specification D 3992).

[D] Specification D 3109 (Replaced by Specification D 3991).

[E] Specification D 1381 (Replaced by Specification D 3992).

[F] Calculated from the denier and specific gravity and based on a theoretical circular cross-section of the fiber.

27.5 *Report*:

27.5.1 State that the specimens were tested as directed in the microscopical analysis procedure of Test Methods D 629. Describe the material(s) or product(s) sampled and the method of sampling used. Report the percentage of each fiber in the sample.

27.6 *Precision and Bias*—See 13.8.1 and 13.8.2.

27.6.1 *Precision*—The precision of the procedure for microscopical analysis of textiles in Test Methods D 629 is being established.

27.6.2 *Bias*—The procedures in Test Methods D 629 for measuring the fiber content of textiles by microscopical analysis has no known bias because the value of the fiber content of textiles by microscopical analysis can be defined only in terms of a test method.

28. Indexing Terms

28.1 This standard is indexed under the following terms: animal fibers (except wool); bast, leaf, and fruit fibers; chemical properties (textiles); cotton; extractable matter; man-made fibers, analysis; moisture content; moisture regain; microscopical analysis; quantitative analysis; silk, textile fibers, and wool.

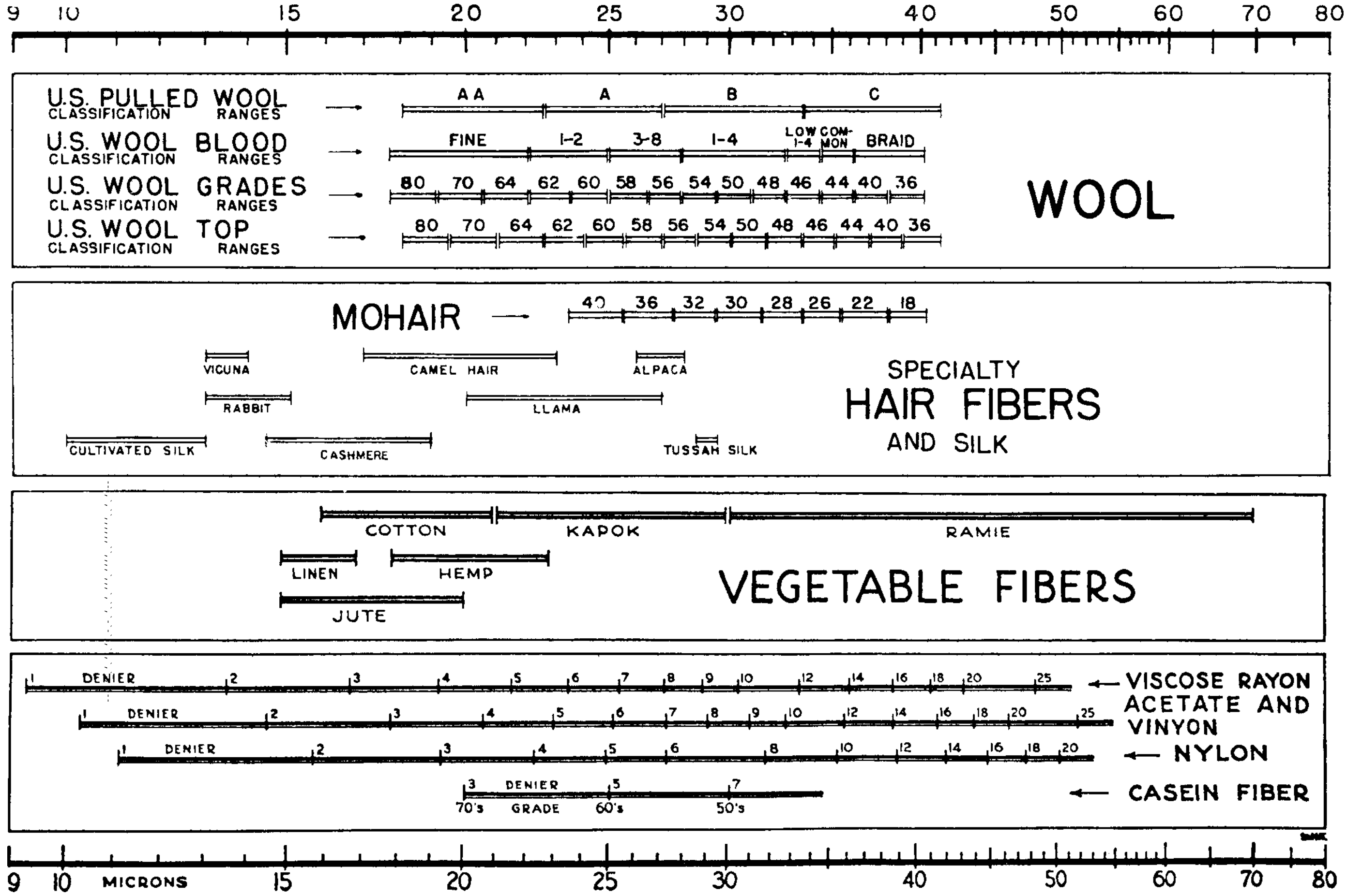


FIG. 1 Comparative Scale for Fineness of Various Textile Fibers

ANNEX

(Mandatory Information)

A1. PROCEDURE FOR DETERMINING THE CROSS-SECTIONAL AREAS OF COTTON FIBERS

A1.1 *Specimen Preparation:*

A1.1.1 Pull a 400-mg sample apart longitudinally and superimpose the two portions so that their ends are even. Repeat this process until the fibers are untangled, thoroughly mixed, and approximately parallel. By careful longitudinal division, separate from the sample a bundle of fibers weighing approximately 40 mg. Comb the bundle with a 50 mm wide fiber comb having 13-mm long needles and spaced 48 needles to the inch until the fibers are straight and parallel.

A1.1.2 Insert the bundle of combed fibers into the slot of a hand microtome. With sharp scissors, cut off both ends of the bundle as close as possible to the plate. Apply a drop of clear lacquer to one cut face of the tuft remaining in the slot, wait until it shows through on the other side, and then apply a drop of lacquer on the second side. The lacquer must be colorless, optically clear, and quick-drying. Let the lacquer dry thoroughly (about 15 min).

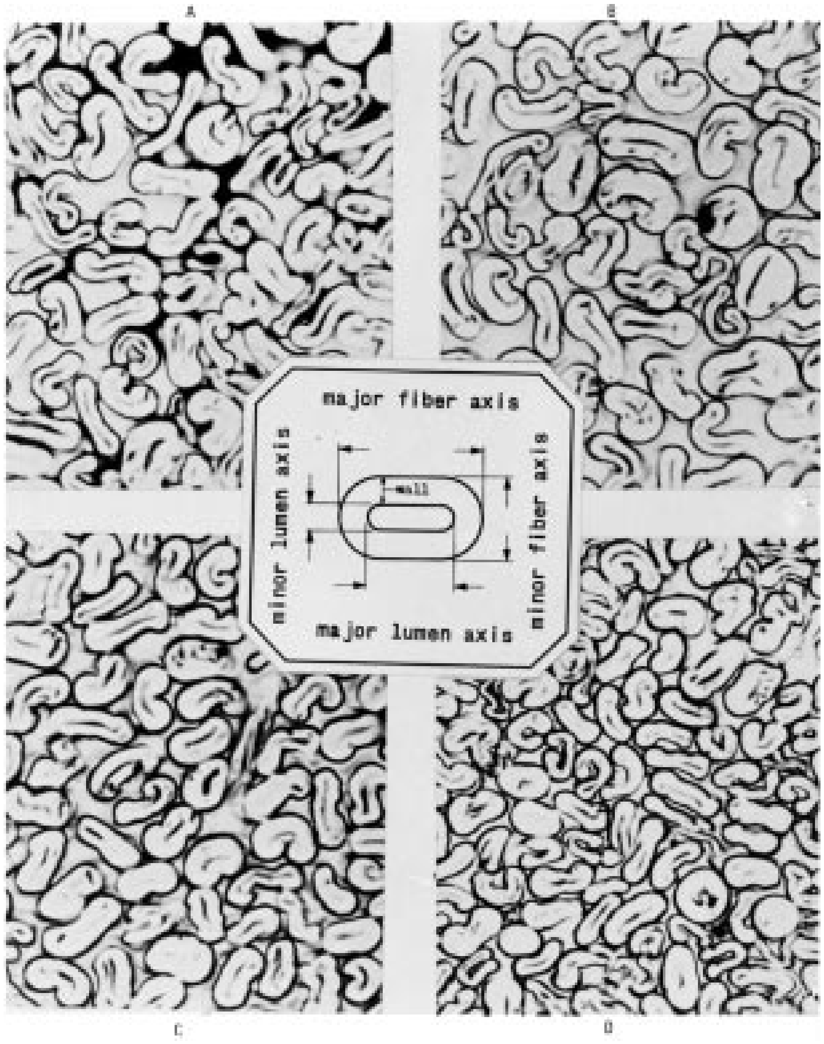
A1.1.3 Under a binocular microscope (magnification approximately 45X), using a sharp safety razor blade, cut off both ends of the tuft flush with the microtome plate. Attach an auxiliary plunger to the plate.

A1.1.4 Under the binocular microscope propel the embedded tuft through the slot until it protrudes 4 to 7 µm above the surface of the plate. Brush a coat of lacquer over the protruding end and let it dry about 5 min or until it is firm and hard. Slide off lacquer and fiber together with a single stroke of a sharp razor blade. Mount the section in a drop of mineral oil on a microscope slide and place over it a cover glass.

A1.1.5 Examine the section at a magnification of approximately 250X. If it is not uniformly thin and satisfactory in appearance, continue to cut sections, coating the face of the fiber block with clear, colorless lacquer after each successive slice. Allow lacquer to dry. When a satisfactory section had been made, photograph the specimen at a magnification of at least 250X. Make a length standard or reference mark on the negative and make an enlargement with an over-all magnification of 1000X. Draw a pair of lines intersecting at right angles near the center of the print. Starting at the intersection, select the nearest 25 fibers in each quadrant, omitting those that are damaged, distorted, or blurred. If more than 10 % of the fiber cross sections must be omitted, prepare and photograph a new cross section.

A1.2 *Procedure:*

A1.2.1 Measure the major and minor axes (see Fig. A1.1) of the 100 fiber cross sections selected. Use a transparent millimetre scale and read it to the nearest 0.5 mm. When the major axis is a straight line, read its total length directly from the scale as it lies on the axis. When the major axis is curved, mark it off into segments of such length as to define essentially straight portions of the total axis. With the scale, measure each segment as though it were a straight line. The sum of the lengths of the segments is thelength of the major axis. Calculate the average fiber diameter by the following equation:



A = Stoneville
B = Rowden
C = Hopi Acala
D = Sea Island

FIG. A1.1 Representative Cross Sections of Different Varieties of Cotton (magnification approx 625x)

$$\text{Average fiber diameter, } \bar{u} = (s + \bar{t})/2 \qquad (A1.1)$$

where:

$\bar{s}$ = average fiber major axis, $\bar{s} = \Sigma\, s/n$, and

$\bar{t}$ = average fiber minor axis, $\bar{t} = \Sigma\, t/n$.

where:

s = fiber major axis, µm

t = fiber minor axis, µm, and

n = number of observations (100 recommended).

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