AATCC takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this test method. Users of this test method are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This test method is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reaffirmed or withdrawn. Your comments are invited either for revision of this test method or for additional methods and should be addressed to the AATCC Technical Center. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing, you should make your views known to Amy G. Hammonds, Laboratory Director, at the address shown below.

Fiber Analysis: Quantitative

Developed in 1957 by AATCC Committee RA24; revised 1958, 1959, 1975, 1995, 2000, 2004, 2005; reaffirmed 1971, 1978, 1981, 1989; editorially revised 1980, 1982 (new title), 1985, 2002. Related to ISO 1833.

1. Purpose and Scope

1.1 This method presents individual procedures for the quantitative determination of moisture content, nonfibrous content and fiber composition of textiles.

1.2 The procedures for the determination of fiber composition include mechanical, chemical and microscopical methods. They are applicable to blends of the following generic classes:

Natural Fibers	Man-Made Fibers
Cotton	Acetate
Hair	Acrylic
Hemp	Modacrylic
Linen	Nylon (see 17.1)
Ramie	Olefin
Silk	Polyester
Wool	Rayon
	Spandex

2. Uses and Limitations

2.1 The procedure given for the removal of nonfibrous materials will remove most, but not all, of these components. Each treatment is applicable only to certain categories of these substances and no general scheme can be given that is all inclusive. Some of the newer finishes may present special problems and the analyst will have to deal with these cases as they arise. Thermosetting resins and crosslinking latices are not only difficult to remove but in some cases cannot be wholly removed without destroying the fiber. When it is necessary to modify a procedure, or use a new one, one should make sure that the fibrous portion of the specimen under test is not attacked. Fiber composition is generally expressed either on the oven-dry weight of the textile as received or on the oven-dry weight of the clean fiber after nonfibrous materials are first removed from the textile before the fiber analysis is carried out, or if the treatments described in Section 9 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.

2.2 The procedure for determining fiber composition by mechanical separation is applicable to those textiles wherein the different fibers making up its composition are segregated in separate yarns, or plies, in the textile product.

2.3 The chemical procedures for fiber composition described herein are applicable to most of the current, commercial production fibers within each generic class listed. Known exceptions are noted in Table II. However, there may be instances in which a method may not be fully adequate for a newly developed fiber falling within one of the listed generic classes and for re-used and/or physically or chemically modified fibers. Caution should be exercised when applying these methods to such cases.

2.4 The microscopical procedures for fiber composition are applicable to all fibers and their accuracy depends to a considerable extent upon the ability of the analyst to identify the individual fibers present. However, owing to the tedious nature of this technique, its use is generally limited to those mixtures which cannot be separated mechanically or chemically; e.g., mixtures of hair and wool and mixtures of cotton, linen, hemp and/or ramie.

3. Terminology

3.1 **clean-fiber content,** n.—the amount of fiber after removal of nonfibrous content.

3.2 **fiber,** n.—*in textiles*, a generic term for any one of the various types of matter that form the basic elements of a textile and which are generally characterized by flexibility, fineness and high ratio of length to thickness.

3.3 **moisture content,** n.—that part of the total mass of a material that is absorbed or adsorbed water, compared to the total mass.

3.4 **nonfibrous content,** n.—products such as fiber finishes, yarn lubricants, slasher sizing, fabric softeners, starches, china-clay, soaps, waxes, oils and resins which are applied to fiber, yarn, fabric or apparel.

3.5 Additional terms used in this test method can be found in standard chemical dictionaries, in dictionaries of common terms or in *A Glossary of AATCC Standard Terminology* (located elsewhere in this TECHNICAL MANUAL).

4. Safety Precautions

NOTE: These safety precautions are for information purposes only. The precautions are ancillary to the testing procedures and are not intended to be all inclusive. It is the user's responsibility to use safe and proper techniques in handling materials in this test method. Manufacturers MUST be consulted for specific details such as material safety data sheets and other manufacturer's recommendations. All OSHA standards and rules must also be consulted and followed.

4.1 Good laboratory practices should be followed. Wear safety glasses in all laboratory areas.

4.2 All chemicals should be handled with care.

4.3 Perform the soxhlet extractions in Section 9, Nonfibrous Material—Clean Fiber Content, using Fluorocarbon 113 (such as Freon TF) or hydrochlorofluorocarbon (such as Genesolv 2000) and ethyl alcohol inside an adequately ventilated laboratory hood. CAUTION: Ethyl alcohol is highly flammable.

4.4 Perform Chemical Analysis Procedure No. 1 (Table II, 100% acetone) inside a ventilated laboratory hood. CAUTION: Acetone is highly flammable.

4.5 Ethyl alcohol and acetone are flammable liquids and should be stored in the laboratory only in small containers away from heat, open flame and sparks.

4.6 In preparing, dispensing, and handling hydrochloric acid (20%), sulfuric acids (59.5% and 70%), and formic acid (90%) used in Chemical Analysis Procedure Methods No. 2, 3, 4, and 6 (Table II), use chemical goggles or face shield, impervious gloves and an impervious apron. Concentrated acids should be handled only in an adequately ventilated laboratory hood. CAUTION: Always add acid to water.

4.7 In preparing ammonium hydroxide (8:92) for use in Chemical Analysis Procedure Method No. 4 (Table II, 70% sulfuric acid), use chemical goggles or face shield, impervious gloves and an impervious apron. Dispense, mix and handle ammonium hydroxide only in an adequately ventilated laboratory hood.

4.8 An eyewash/safety shower should be located nearby and a self-contained breathing apparatus should be readily available for emergency use.

4.9 Exposure to chemicals used in this procedure must be controlled at or below levels set by governmental authorities (e.g., Occupational Safety and Health Administration's [OSHA] permissible exposure limits [PEL] as found in 29 CFR 1910.1000 of January 1, 1989). In addition, the American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Values (TLVs) comprised of time weighted averages (TLV-TWA), short term exposure limits (TLV-STEL) and ceiling limits (TLV-C)

are recommended as a general guide for air contaminant exposure which should be met (see 17.17).

5. Apparatus

5.1 Analytical balance, capable of weighing to 0.1 mg.

5.2 Oven, maintained at 105-110°C.

5.3 Desiccator, containing anhydrous silica gel, calcium sulfate (such as Drierite) or its equivalent.

5.4 Soxhlet extractor, 200 mL capacity.

5.5 Constant temperature bath, adjustable, capable of controlling temperature to ± 1°C.

5.6 Weighing bottle, 100 mL capacity, glass, with ground glass cover. (Alternate: aluminum weighing can; same size, tight cover.)

5.7 Erlenmeyer flask, 250 mL capacity, ground glass stopper.

5.8 Beaker, borosilicate heat resistant glass, 250 mL capacity.

5.9 Filtering crucible, fritted glass, coarse porosity, 30 mL.

5.10 Suction flask, with adapter, to hold filtering crucible.

5.11 Weighing bottle, large enough to hold filtering crucible.

5.12 Microscope, equipped with a moveable stage and a cross-hair ocular, 200-250X magnification.

5.13 Projection microscope, capable of 500X magnification (see 17.12).

5.14 Fiber cutter: A device comprised of two razor blades, a threaded pin and an assemblage that will hold the blades rigidly in position. The device is operated by applying pressure vertically downward. It cuts fibers approximately 250 µm in length (see 17.3).

5.15 Wedge scale: Strips of heavy paper or Bristol board imprinted with a wedge for use at 500X magnification (see 17.4).

6. Reagents

6.1 Ethyl alcohol (95%), pure or denatured.

6.2 Fluorocarbon 113 (such as Freon TF) or hydrochlorofluorocarbon (such as Genesolve 2000).

6.3 Hydrochloric acid (HCl), 0.1*N*.

6.4 Enzyme solubilizing preparation.

6.5 Acetone (CH_3COCH_3), reagent grade.

6.6 Hydrochloric acid (HCl) (20%). Dilute HCl, sp gr 1.19, with water until the specific gravity of the solution is 1.10 at 20°C.

6.7 Sulfuric acid (H_2SO_4) (59.5%). Add H_2SO_4, sp gr 1.84, slowly to water. After the solution has cooled to 20°C, adjust the density to a value between 1.4902 and 1.4956 g/mL.

6.8 Sulfuric acid (H_2SO_4) (70%). Add H_2SO_4, sp gr 1.84, slowly to water. After the solution has cooled to 20 ± 1°C, adjust the density to a value between 1.5989 and 1.6221 g/mL (see 17.18).

6.9 Sulfuric acid (H_2SO_4) (1:19). Slowly stir 1 volume of H_2SO_4, sp gr 1.84, into 19 volumes of water.

6.10 Sodium hypochlorite (NaOCl). Prepare a solution of NaOCl, 5.25% available chlorine. Sodium hypochlorite based household bleach (nominally 5.25%) has been found to be acceptable.

6.11 Sodium bisulfite ($NaHSO_3$) (1%). Freshly prepared.

6.12 Formic acid (HCOOH) (90%), sp gr of 1.202 at 20°C.

6.13 Ammonium hydroxide (NH_4OH) (8:92). Mix 8 volumes of NH_4OH, sp gr 0.90, with 92 volumes of water.

6.14 Herzberg stain. Add the previously prepared solution A to solution B; allow to stand overnight; decant the clear liquid into a dark colored glass bottle and add a leaf of iodine.

Solution A		**Solution B**	
Zinc Chloride	50 g	Potassium Iodide	5.5 g
Water	25 mL	Iodine	0.25 g
		Water	12.5 mL

7. Sampling

7.1 It is not possible to give specific instructions for taking a laboratory test sample from all types of textile materials to which these methods may be applicable; but a few general recommendations will be given.

7.1.1 The sample should be as representative as possible of the lot of material from which it was taken.

7.1.2 If a reasonably large lot is available, and if it is possible to do so, samplings should be taken from different, widely separated areas or parts of the lot.

7.1.3 In the case of fabrics where there is a definite repetition in the pattern, the sample should include all yarns in a complete pattern (see 17.6).

7.1.4 In the case of yarns, not less than a 2-meter length should be taken.

Test Methods

8. Moisture Content

8.1 Procedure. Place not less than 1 g of the textile to be tested in a previously tared weighing bottle and immediately replace the cover. Weigh to the nearest 0.1 mg using the analytical balance and record the weight. Place the uncovered weighing bottle containing the specimen in an oven maintained at 105-110°C for 1.5 h. At the end of the time period, remove the bottle from the oven, immediately replace the cover and put it in the desiccator. When the bottle and contents have cooled to room temperature, remove them from the desiccator and reweigh. Repeat the heating and reweighing process for periods of 30 min until the weight is constant to within ± 0.001 g and record the constant weight.

8.2 Calculations.

8.2.1 Calculate the moisture content of the specimen as follows:

$$M = \frac{A - B}{A - T} \times 100$$

where:

M = moisture content, percent.
A = weight of sample before drying + bottle.
B = weight of sample after drying + bottle.
T = tare weight of weighing bottle.

9. Nonfibrous Material—Clean Fiber Content

9.1 Procedure. Take a specimen of not less than 5 g, dry it to constant weight in an oven at 105-110°C (see 8.1), record the oven-dry weight to the nearest 0.1 mg using an analytical balance and then subject it to one, or more, of the following treatments, as appropriate. When specific type of nonfibrous content is known, only that specific treatment, or treatments, need be performed; otherwise, all treatments must be applied.

9.1.1 Hydrochlorofluorocarbon Treatment (for removal of oils, fats, waxes, certain thermoplastic resins, etc.). Extract the dried specimen with hydrochlorofluorocarbon in a soxhlet extractor, siphoning over a minimum of six times. Air dry, and then dry at 105-110°C to constant weight. For an alternative to soxhlet extractor, see 17.19.

9.1.2 Alcohol Treatment (for removal of soaps, cationic finishes, etc.). Extract the dried specimen with ethyl alcohol in a soxhlet extractor, siphoning over a minimum of six times. Air dry, and then dry at 105-110°C to constant weight. For an alternative to soxhlet extractor, see 17.19.

9.1.3 Aqueous Treatment (for removal of water soluble materials). Immerse the dried specimen for 30 min in water at 50°C using a 100:1 liquid to fabric ratio. Stir occasionally or use a mechanical shaker. Rinse 3 times in fresh portions of water and dry at 105-110°C to constant weight.

9.1.4 Enzyme Treatment (for removal of starch, etc.). Immerse the dried specimen in aqueous solution of the enzyme preparation following the manufacturer's recommendations as to concentration, liquid to fabric ratio and temperature and time of immersion. Rinse thoroughly with hot water and dry at 105-110°C to constant weight.

9.1.5 Acid Treatment (for removal of amino resins). Immerse the dried specimen in 100 times its weight of 0.1*N* HCl at 80°C for 25 min, stirring occasionally. Rinse thoroughly with hot water and dry at 105-110°C to constant weight.

9.2 Calculations.

Table I—Chemical Methods for Analysis of Fiber Mixtures

	Wool	Silk	Rayon	Polyester	Olefin	Nylon	Mod-acrylic	Hair	Cotton, Hemp, Linen, Ramie	Acrylic	Spandex
Acetate	1 4 (5)	1 (5)	1	1 4	1	1 (2)		1 (5)	1	1	
Acrylic	(5)	(3) (5)	(3)			(2) (3) (6)	(1)	(5)			
Cotton, Hemp, Linen, Ramie	4 (5)	(3) (5)	(3)	4		(2) (3) (6)	(1)	(5)			(7)
Hair			5	5	5	(2) 5 6	(1) 5				
Modacrylic	1 (5)	1 (3) (5)	1 (3)	1	1	1 (2) (3) (6)					
Nylon	2 3 (5) 6	(5)	2 6	2 3 6	2 6						2**
Olefin	(5)	(5)									
Polyester	(5)	(3) (4) (5)	(3) (4)								
Rayon	3 4 (5)	(5)									
Silk	3 4										

1 *100% acetone: section 12.1
2 20% hydrochloric acid: section 12.2
3 59.5% sulfuric acid: section 12.3
4 70% sulfuric acid: section 12.4
5 sodium hypochlorite: section 12.5
6 90% formic acid: section 12.6
7 dimethylformamide: section 12.7

*Not suitable for all modacrylic fibers
**Not suitable for all nylon fibers

Section 11.2 contains details of table use.

9.2.1 Calculate the nonfibrous content of the specimen as follows:

$$N = \frac{C - D}{C} \times 100$$

where:
N = nonfibrous materials, percent.
C = dry weight, specimen, before treatment.
D = dry weight, specimen, after treatment.

9.2.2 Calculate the clean fiber content of the specimen as follows:

$$F = \frac{D}{C} \times 100$$

where:
F = clean fiber content, percent; other terms as in 9.2.1

10. Mechanical Separation

10.1 Procedure. Remove the nonfibrous materials using the appropriate treatment (see 9.1). Separate the component yarns by mechanical dissection; combine those yarns, or plies, having the same fiber composition and determine the oven-dry weight of each generic type present.

10.2 Calculation. Calculate the content of each generic fiber as follows:

$$X_i = \frac{W_i}{E} \times 100$$

where:
X_i = content of fiber i, percent.
W_i = oven-dry weight of fiber i, after separation.
E = weight of clean, oven-dry specimen taken for analysis.

11. Chemical Analysis—General

11.1 Specimen Preparation. Before analyses are undertaken, the laboratory test sample should be disintegrated, homogenized and a portion of the homogenate taken for the chemical treatment(s). In the case of a fabric, one should unravel it into its individual yarns, cut the yarns into lengths not greater than 3 mm, thoroughly mix the cut pieces and then take a representative portion for the specific determination. An alternate procedure, suitable in many cases, is to grind the sample using a Wiley Mill, homogenize the ground fibers by slurrying them in a water suspension in a Waring Blender and taking the representative portion from the dried homogenate for the specific determination. Yarns are treated the same way but omitting the unnecessary steps.

11.2 Method Application. A tabulation of appropriate chemical treatments for binary fiber mixtures is given in Table I. To use this table one enters at the left side on the line listing one of the components of the binary mixture and moves to the box under the column listing the other component and the number therein is the method, or methods, that are applicable for that specific combination. The unbracketed methods are those that dissolve the fiber at the left side of the diagram while the bracketed ones dissolve the fiber at the top of the diagram. Mixtures of more than two components may be analyzed by proper application of a sequence of the individual methods. Table II presents the relative solubilities of the various fibers in all the reagents and, from this, one can select the proper methods and their sequence for the analysis of multifiber mixtures (see 17.7).

Table II—Solubilities of Fibers in Reagents Used in the Chemical Methods

	Chemical Method					
	No. 1 100% CH_3COCH_3	No. 2 20% HCl	No. 3 59.5% H_2SO_4	No. 4 70% H_2SO_4	No. 5 NaOCl	No. 6 90% HCOOH
Acetate	S	I	S	S	I	S
Acrylic	I	I	I	I*	I	I
Cotton	I	I	SS	S	I	I
Hair	I	I	I	I	S	I
Hemp	I	I	SS	S	I	I
Linen	I	I	SS	S	I	I
Modacrylic	S or I*	I	I	I	I	I
Nylon	I	S	S	S	I	S
Olefin	I	I	I	I	I	I
Polyester	I	I	I	I	I	I
Ramie	I	I	SS	S	I	I
Rayon	I	I	S	S	I	I
Silk	I	PS	S	S	S	PS
Wool	I	I	I	I	S	I

*Depending on type

KEY TO SYMBOLS: S = SOLUBLE
PS = PARTIALLY SOLUBLE (Method not applicable)
SS = SLIGHTLY SOLUBLE (Useable but correction factor required)
I = INSOLUBLE

Section 11.2 contains details of table use.

12. Chemical Analysis Procedures

12.1 Method No. 1, 100% Acetone: Weigh accurately a 0.5-1.5 g portion of the clean, dry, prepared specimen and record the weight to the nearest 0.1 mg. Transfer into a 250 mL Erlenmeyer flask. Add 100 times its weight of acetone and agitate vigorously for 15 min keeping the temperature at 40-50°C. Decant the liquid from the undissolved residue, add a fresh portion of acetone and agitate for a few more minutes. Repeat the decanting and agitation process one more time and then filter the undissolved residue by suction through a dried weighed, fritted-glass, filtering crucible. Dry the crucible and residue in air and then in an oven at 105-110°C to constant weight. Record the weight of the dried residue to the nearest 0.1 mg.

12.2 Method No. 2, 20% Hydrochloric acid: Weigh accurately a 0.5-1.5 g portion of the clean, dry, prepared specimen and record the weight to the nearest 0.1 mg. Transfer into a 250 mL Erlenmeyer flask. Add 50-150 mL of 20% hydrochloric acid (100 mL reagent/g of sample); shake vigorously and let stand for 5 min at 15-25°C. Shake again and let stand for 15 min. Shake for a third time (see 17.8) and filter the mixture through a dried weighed fritted-glass crucible. Wash into the crucible any residue left in the flask using a little more 20% hydrochloric acid. Apply suction to drain the excess liquor from the filter residue. Wash the residue in the crucible with about 40 mL of 20% hydrochloric acid and then with water until the filtrate is neutral to litmus. Disconnect the suction and add to the crucible about 25 mL of ammonium hydroxide (8:92) allowing the fiber residue to soak for 10 min before applying suction to drain it. Wash the residue with about 250 mL of water, allowing it to soak in the water for about 15 min. After the final washing, apply suction to remove rinse water, and dry the crucible and residue in an oven at 105-110°C to constant weight. Record the dry weight to the nearest 0.1 mg.

12.3 Method No. 3, 59.5% Sulfuric acid: Weigh accurately a 0.5-1.5 g portion of the clean, dry, prepared specimen and record the weight to the nearest 0.1 mg. Transfer into a 250 mL Erlenmeyer flask. Add 50-150 mL of 59.5% sulfuric acid (100 mL reagent/g of sample) and shake vigorously for 1 min. Let stand for 15 min at a temperature of 15-25°C. Shake again and let stand for another 15 min, shake for a third time (see 17.8) and then filter the mixture through a dried weighed fritted-glass crucible. Wash into the crucible any residue left in the flask using three 10 mL aliquots of 59.5% sulfuric acid. Apply suction to drain the excess liquor from the fiber residue after the addition of each aliquot. Wash the residue in the crucible with 50 mL of sulfuric acid (1:19), then with water until the filtrate is neutral to litmus. Disconnect the suction and add to the crucible about 25 mL of ammonium hydroxide (8:92), allowing the fiber residue to soak for 10 min before applying suction to drain it. Wash the residue with about 150 mL of water, allowing it to soak in the water for about 15 min. After the final washing, apply suction to remove the rinse water and dry the crucible and fiber residue in an oven at 105-110°C to constant weight. Record the weight of the dried residue to the nearest 0.1 mg (see 17.9).

12.4 Method No. 4, 70% Sulfuric acid: Weigh accurately a 0.5-1.5 g portion of the clean, dry, prepared specimen and record the weight to the nearest 0.1 mg. Transfer into a 250 mL Erlenmeyer flask. Add 50-150 mL of 70% sulfuric acid (100 mL reagent/g of sample) and shake vigorously for 1 min. Let stand for 15 min at a temperature of 15-25°C. Shake again and let stand for another 15 min; shake for a third time (see 17.8) and then filter the mixture through a fritted-glass crucible which has been oven-dried, cooled in a desiccator and weighed to 0.1 mg. Wash into the crucible any residue left in the flask using three 10 mL aliquots of 70% sulfuric acid. Apply suction to drain the excess liquor from the fiber residue after the addition of each aliquot. Wash the residue in the crucible with 50 mL of sulfuric acid (1:19), then with water until the filtrate is neutral to litmus. Disconnect the suction and add to the crucible about 25 mL of ammonium hydroxide (8:92); allow the fiber residue to soak for 10 min before applying suction to drain it. Wash the residue with about 150 mL of water, allowing it to soak in the water for about 15 min. After the final washing, apply suction to remove excess water and dry the crucible and fiber residue in an oven at 105-110°C to constant weight. Record the weight of the dry residue to the nearest 0.1 mg.

12.5 Method No. 5, Sodium hypochlorite: Weigh accurately a 0.5-1.5 g portion of the clean, dry, prepared specimen and record the weight to the nearest 0.1 mg. Transfer into a 250 mL Erlenmeyer flask. Add 50-150 mL of sodium hypochlorite reagent (100 mL reagent/g of sample). Stir the specimen in this solution for 20 min making sure the temperature is maintained at 25 ± 1°C (use constant temperature bath) (see 17.10) and then filter through a dried weighed, fritted-glass crucible. Wash thoroughly with sodium bisulfite (1%) followed by water and remove the excess water by suction. After the final washing, apply suction to remove excess water and dry in an oven at 105-110°C to constant weight. Record the weight of the dried residue to the nearest 0.1 mg.

12.6 Method No. 6, 90% Formic acid: Weigh accurately a 0.5-1.5 g portion of the clean, dry, prepared specimen and record the weight to the nearest 0.1 mg. Transfer into a 250 mL Erlenmeyer flask. Add 50-150 mL of 90% formic acid (100 mL reagent/g of sample) and shake frequently over a period of 15 min (see 17.11). Decant the supernatant liquid into a dried, weighed, fritted-glass crucible, add another equal portion of 90% formic acid to the flask and agitate for an additional 15 min. Filter the contents of the

flask through the crucible, rinse with two 50 mL portions of 90% formic acid and drain with the aid of suction. Wash the residue with 50 mL of water and then allow it to soak in 25 mL of ammonium hydroxide (8:92) for about 10 min. Wash the residue thoroughly with water until the filtrate is neutral to litmus. Drain the residue with the aid of suction and dry in an oven at 105-110°C to constant weight. Record the weight of the dried residue to the nearest 0.1 mg.

12.7 Method No. 7, Dimethylformamide: Weigh accurately a 0.5-1.5 g portion of the clean, dried, prepared specimen and record the weight to the nearest 0.1 mg. Transfer to a 250 mL Erlenmeyer flask. Add 50-150 mL of dimethylformamide reagent (100 mL reagent/g of sample). Agitate for 20 min keeping the temperature at 98 ± 1°C. Decant the liquid from the undissolved residue, add a fresh portion of dimethylformamide and agitate for a few more minutes. Repeat the decanting and agitation process one more time and then filter the undissolved residue by suction through a dried, weighed fritted glass filtering crucible. Dry the crucible and residue in air and then in an oven at 105-110°C to constant weight. Record the weight of the dried residue to the nearest 0.1 mg.

12.8 Calculations: Calculate the content of each generic fiber type as determined by any one of the above applicable chemical methods using one of the following equations:

12.8.1 If the fiber is *dissolved* by the test reagent:

$$X_i = \frac{G - H_i}{G} \times 100$$

12.8.2 If the fiber is insoluble in the test reagent:

$$X_i = \frac{H_i}{G} \times 100$$

where:

X_i = content of fiber *i*, percent.
G = weight of clean, dry, prepared specimen
H_i = weight of dried residue after treatment

13. Microscopical Analysis, General

13.1 The following procedure may be used for the quantitative analysis of textiles containing two or more fiber types which cannot be separated readily by mechanical or chemical methods. The procedures rely on the ability of a technician to identify and count, by means of a microscope, the relative number of fibers of each type in a prepared specimen. Such a count will result in a percent blend by *number* of fibers. In order to convert this result to a percent by weight, the size of the fibers being counted and their respective densities must be included in the calculation.

13.2 Microscope slides may be prepared to scan longitudinal or cross-section views of a fiber sample. The fiber images may be viewed either through a microscope or as projected onto a horizontal plane. While either viewing method may be used for identification and counting of fibers, the projection method is specifically used for measuring fiber diameters using a wedge scale (see 14.3.2).

13.3 Methods which may be used to identify fibers during the fiber counting procedures are discussed in AATCC Test Method 20. They include the following:

	AATCC 20
Herzberg stain (zinc chloro-iodide)	Section 9.9.1
Acid phloroglucinol reagent	Section 9.9.2
Longitudinal appearance	Tables I and II
Cross-section appearance	Tables I and II and Appendix I

It is recommended that reference tests be made on known fibers rather than placing total reliance on photographic reproductions and word descriptions of colors.

14. Microscopical Analysis Procedures

14.1 Preparation of Slides.

14.1.1 Longitudinal Sections of Vegetable Fibers (cotton, flax, ramie, etc.): A swatch of fabric measuring at least 5 × 5 cm should be available. Count the number of yarns in both the warp and filling and select from each direction at random a number of yarns that is proportional to the fabric count. The combined number of warp and filling yarns should total at least 20 (see 17.12). If the sample is in yarn form, take at least a two-meter length and, from it, cut not less than twenty 5-cm sections at random. Cut approximately 2.5 cm of each yarn, or yarn section, into lengths of 0.5-1 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 125 mL Erlenmeyer flask. Add sufficient water so that after stoppering the flask and shaking the contents, a uniform and fairly dense fiber suspension is obtained. Quick boiling or the addition of a few glass pellets facilitates the separation of the fibers. Using a glass-marking pencil, draw two parallel lines about 1 in. apart across a glass slide. With a wide-mouth pipette, draw 0.5-1 mL of the well shaken suspension and place it between the two reference lines on the slide. The amount of liquid taken 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 of the moisture has evaporated from the slide, stain the fibers with Herzberg stain and cover with a cover glass.

14.1.2 Longitudinal Sections of Wool, Hair and other round Fibers: Select a representative swatch, or yarn sections, as in 14.1.1. With a fabric swatch, remove the outermost yarns in both directions so that the warp and filling yarns are protruding approximately 1 cm. Lay the sample flat on a table and, using a fiber cutter, force the blades vertically downward into the warp fringe. Repeat the operation with the filling fringe. Remove the device with the top plate up, release the tension on the cutting blades and remove them together by their ends between the thumb and forefinger. Carefully separate the blades so that the cut fiber sections will adhere to the edge of one or both blades. Place a few drops of mineral oil on a clean slide and, with a dissecting needle, scrape the fiber sections onto the oil. Thoroughly disperse the fibers in the oil with the dissecting needle and cover with a cover glass. For yarn samples, align the sections side-by-side and perform the above operation starting with the use of the fiber cutter.

14.1.3 Cross-Sections of Fibers: Select representative yarns, or yarn sections, from the sample and render them to staple by removing twist and drawing out fibers. Align the fibers parallel to each other so as to form a well blended tuft. Prepare a slide following the instructions of AATCC Test Method 20, Section 9.3.

14.2 Fiber Counting.

14.2.1 Fibers Viewed Through Microscope: Place the slide prepared as in 14.1 on the moveable stage on a microscope equipped with a crosshair ocular and having a magnification of 200-250X. Begin to count near either the upper or lower corner 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 crosshairs. After each trip across the field, move the slide 1-2 mm vertically and identify and count the fibers as the field is again traversed. Repeat this procedure until the whole slide has been covered. The spacing between each traverse is dependent upon the number of fiber sections on the slide. If a fiber passes the crosshair more than once, record each passing (see 17.13). In a similar manner, count the fibers by moving the slide vertically. The combined horizontal and vertical counts should total at least 1000 fibers.

14.2.2 Projected Images of Fibers: Calibrate the microprojector so that it will give a magnification of 500X in the plane of the projected image. To do this, place a stage micrometer (having units of 0.01 mm) on the stage of the micro-

projector with its scale toward the objective and place a large sheet of white, nonglare paper in the projection plane. Lower or raise the microscope until an interval of 0.20 mm on the stage micrometer will measure 100 mm when sharply focused in the center of the paper. Place the slide prepared as in 14.1 on the stage of the microprojector with the cover glass toward the objective. Draw a 10-cm diameter circle in the center of the white paper in the projection plane. All measurements and counts are to be made within this circle. Begin to count near either the upper or lower corner of the field and proceed exactly as described in 14.2.1 (see 17.14).

14.2.3 Video Imaging of Fibers: Using a video monitor with a crosshair placed in the center of the screen coupled to a video camera attached to a correctly adjusted transmitted light microscope scan the slide as outlined in 14.2.1.

14.3 Fiber Measurement.

14.3.1 Fibers with Noncircular Cross-Sections:

14.3.1.1 Paper Tracing Method: Prepare a slide as described in 14.1.3. Place this slide on the stage of the microprojector and project the image onto a sheet of graph paper having one millimeter squares. Trace the image of the fibers on the graph paper using a sharpened pencil, taking care not to retrace fibers previously traced. If there are not sufficient fibers on the slide to provide 100 of each type, prepare another slide as described above using another tuft of fibers. Continue to trace and to count on the new slide until 100 fibers of each type have been tallied. By counting squares, and parts of squares, determine the cross-sectional area of each individual fiber of each type. Calculate the mean cross-sectional area for each fiber type present by summing the individual values recorded for that type and dividing the sum by the total number of fibers of that type measured. Final values should be in mm^2.

14.3.1.2 Digital Tracing Method: Prepare a slide as described in 14.1.3. Place this slide on the stage and adjust until a clear well formed image appears on the video monitor. Use of a 50X objective with C mount video camera coupling has been found acceptable for most fiber types. Using correctly calibrated image analysis software and a digital image capture board digitally capture the cross-sectional image. Using a mouse or stylus trace around the captured cross-sectional images. Use the image analysis software to store the resultant cross-sectional areas. If there are not sufficient fibers on the slide to provide 100 of each type, prepare another slide as described above using another tuft of fibers. Continue to trace and count on the new slide until 100 fibers of each type have been tallied. Calculate the mean cross-sectional area automatically using the statistical functions of the digital image analysis software. Final values should be in μm^2.

14.3.2 Fibers with Circular Cross-Sections: Prepare a slide as described in 14.1.2. Be sure to make measurements on the same day that the slide was prepared. Position the slide in the microprojector or microscope so that all areas of the slide can be reached.

14.3.2.1 Measurement with Wedge Scale and Microprojector: Bring each individual fiber being measured into sharp focus on the wedge scale. Adjust the position of the scale until the fiber image is projected on the wedge with a fine line and mark on the wedge the point that corresponds to the width of the fiber midway of its length. Traverse the slide and measure successive fibers of each type following a planned course. Measure fibers only when their midpoint falls within the 10-cm circle central located in the field. Exclude from measurement those fibers that cross another fiber at the point of measurement and those that are shorter than 150 µm. A minimum of 100 fibers of each type present should be measured. Calculate the mean cross-sectional area of each fiber type. Final values should be in μm^2 (see 17.15). Considerable variation can occur in the average diameter of hair and wool fibers. Thus, for accurate results on any specific sample, the fiber diameters must be measured. If, however, maximum accuracy is not required, the diameters given in Tables III and IV may be used.

14.3.2.2 Measurement with Digital Image Analysis Software: Using a correctly calibrated image analysis system measure the diameter of the individual fibers in question.

14.4 Calculations. Calculate the content of each fiber as percent by weight using equation 1 if 14.3.1 was used to determine average area of fiber cross-section images, and equation 2 if section 14.3.2 was used to determine fiber diameters (see 17.16).

$$X_i = \frac{N_i \times A_i \times S_i}{\Sigma(N \times A \times S)} \quad (1)$$

$$X_i = \frac{N_i \times D_i^2\ \pi/4 \times S_i}{\Sigma(N \times D_i^2\ \pi/4 \times S)} \quad (2)$$

where:

X_i is content of fiber i, percent (by weight)
N_i is relative number of fibers of type i
A_i is average area of fiber images of fiber i
D_i^2 is the mean of diameter squared of fibers of type i
$D_i^2\ \pi/4$ is the mean cross-sectional area of round cross-section fibers of type i
S_i is specific gravity of type i fiber

Table III—Comparative Scale for Fineness of Various Textile Fibers in Micrometers (µm)

	IWTO Super Fine Wools Grades		
'X' Value	**Average Fineness µ**	**'X' Value**	**Average Fineness µ**
Super 80's	19.5 ± 0.25	Super 150's	16.0 ± 0.25
Super 90's	19.0 ± 0.25	Super 160's	15.5 ± 0.25
Super 100's	18.5 ± 0.25	Super 170's	15.0 ± 0.25
Super 110's	18.0 ± 0.25	Super 180's	14.5 ± 0.25
Super 120's	17.5 ± 0.25	Super 190's	14.0 ± 0.25
Super 130's	17.0 ± 0.25	Super 200's	13.5 ± 0.25
Super 140's	16.5 ± 0.25	Super 210's	13.0 ± 0.25

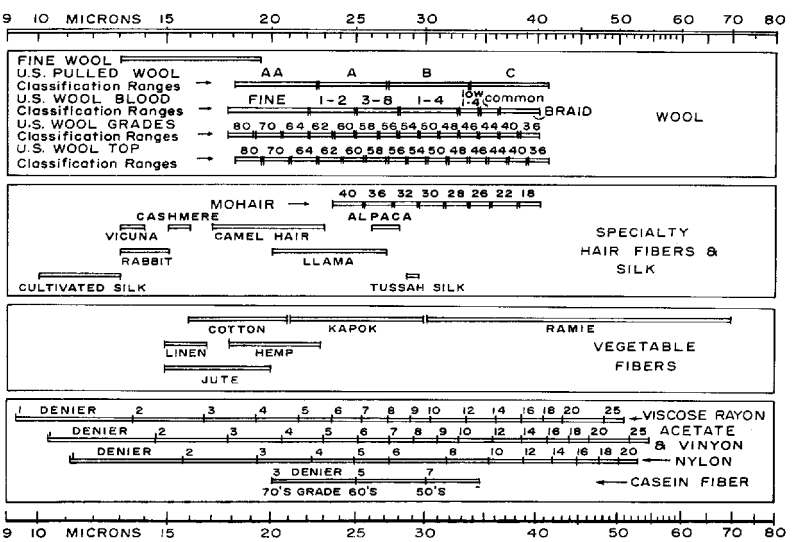

Table IV—Fineness Ranges and Fiber Diameters of Various Textile Fibers[a] in Micrometers (μm)

U.S. Wool Classification

Wool Grades			Wool Top Grades		Pulled Wool
Numerical System	Average Diameter	Blood System[c]	Numerical System	Average Diameter[d]	Grades
80s	17.7-19.1	Fine	80s	18.1-19.5	AA
70s	19.2-20.5	Fine	70s	19.6-21.0	AA
64s	20.6-22.0	Fine	64s	21.1-22.5	AA
62s	22.1-23.4	½	62s	22.6-24.0	A
60s	23.5-24.9	½	60s	24.1-25.5	A
58s	25.0-26.4	⅜	58s	25.6-27.0	A
56s	26.5-27.8	⅜	56s	27.1-28.5	B
54s	27.9-29.3	¼	54s	28.6-30.0	B
50s	29.4-30.9	¼	50s	30.1-31.7	B
48s	31.0-32.6	¼	48s	31.8-33.4	B
46s	32.7-34.3	Low ¼	46s	33.5-35.1	C
44s	34.4-36.1	Common	44s	35.2-37.0	C
40s	36.2-38.0	Braid	40s	37.1-38.9	C
36s	38.1-40.2	Braid	36s	39.0-41.2	C

Hair Fibers and Silk

Mohair (1)		Miscellaneous Hair Fibers (1)		Silk (1)	
Grade	Fineness Range	Fiber	Average Fineness	Fiber	Average Fineness
40s	23.55-25.54	Vicuna	13.0-14.0	Cultivated silk	10.0-13.0
36s	25.55-27.54	Cashmere	14.0-19.0	Tussah silk	28.5
32s	27.55-29.54	Camel hair	17.0-23.0		
30s	29.55-31.54	Alpaca	26.0-28.0		
28s	31.55-33.54	Llama	20.0-27.0		
26s	33.55-35.54				
22s	35.55-38.04				
18s	38.05-40.54				

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

Theoretical Fiber Diameter[d]

Rayon (3), Acetate (3), Nylon (4), and Vinyon (3)				Casein Fiber (5)		
Filament Denier	Viscose Rayon	Acetate and Vinyon	Nylon	Grade	Denier	Fiber Diameter
1	9.6	10.3	11.1	70s	3	20
2	13.6	14.5	15.7	60s	5	25
3	16.7	17.8	19.3	50s	7	30
4	19.3	20.6	22.3			
5	21.6	23.0	24.9			
6	23.6	25.2	27.3			
7	25.5	27.3	29.5			
8	27.3	29.1	31.5			
9	28.9	30.9	33.4			
10	30.5	32.6	35.2			
12	33.4	35.7	38.5			
14	36.1	38.5	41.7			
16	38.6	41.2	44.5			
18	40.9	43.7	47.3			
20	43.1	46.1	49.9			

[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 Fiberglas Corp.
(3) American Viscose Corp.
(4) E. I. du Pont de Nemours and Co.
(5) Aralac Incorporated.

[c] Trade application.

[d] U.S. Standards, Federal Register, January 13, 1954; Specifications for Fineness of Wool Tops and Assignment of Grade (ASTM Designation: D 3992).

$\Sigma(N_i \times A_i \times S_i)$ is sum of the respective N × A × S products for each fiber type in the blend

$\Sigma(N \times D_i^2\ \pi/4 \times S)$ is sum of the respective $N \times D_i^2\ \pi/4 \times S$ products for each fiber type in the blend

See Table V for specific gravity values.

15. Report

15.1 Report the percentage fiber content by weight of the sample analyzed. State if nonfibrous content has been removed or if results are based on other than oven-dry weights.

16. Precision and Bias

16.1 A chemical separation interlaboratory test was conducted using a PET/Wool intimate blend fabric with a nominal fiber content of 55% PET/45% Wool according to the fabric manufacturer, with results as noted in Tables VI and VII.

16.1.1 Between Laboratories

Standard Deviation = $\sqrt{0.6305}$ = 0.7940% polyester
Precision: ± t.975(6df)×S = 2.45 × 0.7940% = ± 1.9454% polyester

16.1.2 Between Operators within Laboratories.

Standard deviation = $\sqrt{0.0655}$ = 0.2559% polyester
Precision: ± t.975(6df)×S = 2.45 × 0.2559% = ± 0.627% polyester

16.2 *Interpretation*. The above statistics apply to the PET/Wool fabric tested which may represent a best case scenario for the determination of fiber content by chemical separation. Additional studies are underway in conjunction with Committee RA102, Statistics Advisory, involving different blend levels.

Table V

Fiber	Specific Gravity
Acetate	1.31
Acrylic	1.16-1.22
Cotton	1.55
Hair	1.32
Hemp	1.48
Linen	1.50
Modacrylic	1.28-1.38
Nylon	1.14
Olefin	0.93
Polyester	1.23-1.40
Ramie	1.51
Rayon	1.52
Silk	1.25
Spandex	1.0-1.2
Wool	1.31

In the case of a fiber with a range of values, knowledge of the specific type in the generic class may permit the selection of a precise specific gravity; or the density of a fiber may be determined using the procedure described in AATCC Test Method 20, Section 9.6.

Table VI—Nested Factorial Design (Polyester %)

Laboratory	A		B		C		D		E	
Operator	1	2	3	4	5	6	7	8	9	10
	58.00	57.57	58.60	58.00	57.95	58.27	58.35	59.88	58.30	57.78
	58.09	57.65	58.00	57.70						
	58.04	57.60								
Totals	174.13	172.82	116.60	115.70	57.95	58.27	58.35	59.88	58.30	57.78

Table VII—ANOVA

	Degrees of Freedom (df)	Sum of Squares	Mean Square	F Ratio
Between Laboratories	4	2.5221	0.6305	2.161
Between Operators within Laboratories	5	0.3275	0.0655	0.224
Residual (or error)	6	1.7501	0.2917	—
Totals	15	4.5997		

16.3 *Bias*. The quantitative fiber analysis can only be defined in terms of a test method. There is no independent method for determining the true value. As a means of estimating this property, the method has no known bias.

17. Notes

17.1 Applicable only to Nylon 6 and Nylon 6,6.

17.2 A projection microscope may be obtained from Kenneth A. Dawson Co., 106 Concord Ave., Belmont MA 02178; tel: 800/362-6795; or Unitron Instrument Co., 66 Needham St., Newton Highland MA 02161.

17.3 A Swiss Fiber Cutting Device is available from Harold Wolf, Clifton NJ. An FRL Fiber Cutter is available from Albany International Research Co., 777 West St., Mansfield MA 02048-9114; tel: 508/339-7300. Razor blades for the FRL Fiber Cutter are available from American Safety Razor Co., Industrial Products Div., Razor Blade Lane, Verona VA 24482-0979; tel: 800/336-4061; fax: 703/248-7122.

17.4 Wedge Scales may be obtained from E. J. Powers Press, 201 South St., Boston MA 02111; and Essex Ruling and Printing Co., Lawrence MA 01840.

17.5 Since sodium hypochlorite solutions lose strength on standing, it is recommended that they be standardized frequently. The following is a suitable method for determining the available chlorine content of such solutions: Dilute a 10 mL aliquot of the solution to be tested to 250 mL with water in a volumetric flask. Pipette 25 mL from the volumetric flask to an Erlenmeyer flask; add 3-5 mL of a 10% solution of potassium iodide (KI) and then add 2-3 mL acetic acid (CH_3COOH). Mix well and titrate with 0.1*N* sodium thiosulfate ($Na_2S_2O_3$) until the yellow color of the iodine is nearly destroyed. Add 5 mL of a starch indicator solution and titrate until the blue color entirely disappears. Calculate the percentage of available chlorine by weight as follows:

$$\text{Available Chlorine, \%} = 3.5A/B$$

where:

A = mL of 0.1N sodium thiosulfate used
B = g of 10 mL aliquot taken

17.6 Exception: when the pattern is smaller than 15 × 15 cm, a sufficient number of complete patterns should be taken to be equivalent to not less than 225 cm^2.

17.7 Whenever there is any doubt about the effectiveness of a given method in dissolving a specific fiber, or whenever there is an application of a method to a new type of fiber, one should always examine the residue in the filtering crucible after weighing. This precaution should always be taken when a fiber blend consists of (1) one predominate fiber with one (or more) minor components; or (2) one very minor fiber with one (or more) major components.

17.8 If a mechanical shaking machine is available, the flask may be shaken on it continuously for 30 min.

17.9 Cotton is not completely insoluble in H_2SO_4 (59.5%). Furthermore, a small amount of rayon remains undissolved in this solvent. In the analysis of cotton/rayon blends, interlaboratory tests indicate that to allow for the above bias, the composition of the specimen should be calculated as follows:

$$\text{Corrected cotton, \%} = \frac{100\ aJ}{F} - 1.6$$

where:

for raw cotton, a is 1.062
for bleached cotton, a is 1.046
J is the oven-dry residue weight
F is the oven-dry weight of clean fiber before treatment.

Corrected Regenerated Cellulose Rayon,

% = 100 – corrected cotton percent.

17.10 Extreme care must be taken to control both time of exposure to the reagent and the temperature of treatment. If either is insufficient, the desired fiber or fibers, will not be completely dissolved. If either becomes excessive, it will cause attack of other fibers.

17.11 A mechanical shaking machine may be used for this purpose.

17.12 For fancy woven fabrics, use all the yarns in one or more complete patterns or a representative fraction, if the pattern is large.

17.13 Linen fibers may be present in the fabric or yarn in the form of fiber bundles. Most bundles are reduced to single fibers during the preparation of the fiber suspension. If, however, some bundles do appear on the slide, an attempt should be made to count each of the individual fibers in the bundle.

17.14 A regular microscope may be used for the fiber counting and, if a suitable calibrating device is available, it may also be used for measuring fiber diameters.

17.15 For further information on marking cell numbers on a wedge scale, and for examples of calculations of how to determine the average fiber diameter using cell numbers, see ASTM D 2130, Method of Test for Diameter of Wool and Other Animal Fibers by Microprojection.

17.16 One is cautioned not to mix units; e.g., if some fiber diameters have been determined in µm, then all diameters must be in µm. For ease of calculation, the terms D_i^2 and D^2 may be used in place of $D_i^2\ \pi/4$ and $D^2\ \pi/4$ respectively. If this is done one cannot use both cross-sectional areas and squared diameters in the same equation.

17.17 Available from Publications Office, ACGIH, Kemper Woods Center, 1330 Kemper Meadow Dr., Cincinnati OH 45240; tel: 513/742-2020.

17.18 Sulfuric acid (70%) is available from Fisher Scientific Co. at its various locations.

17.19 Any extractor capable of heating the sample in the solvent up to 150°C while pressurizing up to 2000 may be used. Solvent in use must have an auto ignition of higher than 200°C. The accelerated solvent extractor (ASE) manufactured by Dionex Corp. has been found to be an acceptable alternative to Soxhlet extractor.

18. References

18.1 ASTM D 276, Standard Test Methods for Identification of Fibers in Textiles.

18.2 ASTM D 629, Standard Test Methods for Quantitative Analysis of Textiles.

18.3 ASTM D 1776, Standard Practice for Conditioning and Testing Textiles.

18.4 ASTM D 1909, Standard Table of Commercial Moisture Regains for Textile Fibers.

18.5 ASTM D 2130, Method of Test for Diameter of Wool and Other Animal Fibers by Microprojection.

18.6 ASTM D 4920, Standard Terminology Relating to Moisture in Textiles.

18.7 ASTM Methods are available from ASTM, 100 Barr Harbor Dr., West Conshohocken PA 19428; tel: 610/832-9500; fax: 610/832-9555.