Test Method for Fiber Analysis: Quantitative

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	Aramid (see 17.17)
Linen	meta-aramid
Ramie	para-aramid
Silk	Modacrylic
Wool	Nylon (see 17.1)
	Olefin
	Polybenzimidazole (see 17.17)
	Polyester
	Rayon
	Spandex
	Triexta

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.

2.1.1 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.

2.1.2 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.

2.2 Fiber composition is generally expressed in the laboratory 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.

When used in commerce for the representation of the nominal fiber content of end use items such as garments, moisture regain is typically added back to bone dry numbers generated. ASTM D1909, Standard Table of Commercial Moisture Regains for Textile Fibers, can be used for this purpose.

2.3 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.

2.4 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.

2.5 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 AATCC M11.

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 hexane and ethyl alcohol inside an adequately ventilated laboratory hood. CAUTION: Hexane and ethyl alcohol are 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 Hexane, ethyl alcohol and acetone are flammable liquids and should be stored in the laboratory only in small quantities 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; see web site: www.osha.gov for latest version). In addition, the American Conference of Govern-

mental Industrial Hygienists (AC-GIH) 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 17.2).

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, 200-250 magnification.

5.13 Projection microscope, capable of 500 magnification.

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.

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

5.16 Flask cover (see 17.16).

5.17 Wet grinder/polisher equipped with 10 in platen for use with 10-in. abrasive discs.

5.18 Adhesive-backed abrasive discs, 10-in. (grit: 120, 240, 320, 400, 600, 800, 1200).

5.19 1-gallon vacuum chamber with pump capable of maintaining vacuum pressure of at least 25 in-Hg.

5.20 2-piece castable mounting cups, 1.5 in.

5.21 Rigid mounting card: non-absorbent yarn sample mounting card for use with epoxy resin mounting method. See Fig. 1 for dimensions suitable for 1.5-in. sample cups.

6. Reagents

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

6.2 Hexane (C_6H_{14}).

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.

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

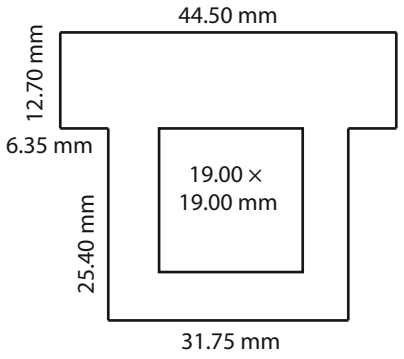

Fig. 1—Rigid Mounting Card (all tolerances are ±0.38 mm).

6.15 Isopropanol (C_3H_7)HO (70%)

6.16 N,N-Dimethylacetamide $CH_3C(O)N(CH_3)_2$

6.17 Methanol (CH_3OH), reagent grade.

6.18 Sodium Hydroxide (NaOH), pellet, 95% reagent grade.

6.19 Xylenes ($C_6H_4(CH_3)_2$), mixed, 95% reagent grade.

6.20 Lithium Chloride (LiCl), crystal reagent grade.

6.21 Epoxy release agent for use with castable mounting materials.

6.22 Epoxy resin suitable for creating castable mounts.

6.23 Epoxy hardener compatible with the selected epoxy resin.

6.24 Cyclohexanone $(CH_2)_5CO$.

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 17.4).

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:

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

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 Hexane Treatment (for removal of oils, fats, waxes, etc.). Extract the dried specimen with hexane 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 17.15.

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 17.15.

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.

9.2.1 Calculate the nonfibrous content of the specimen as follows:

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

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:

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

where:

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

9.2.3 Additional techniques for the extraction and analysis of textile finishes can be found in AATCC TM94, Test Method for Finishes in Textiles: Identification.

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:

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

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 17.5).

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 17.6) 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.

Table I—Chemical Methods for Analysis of Fiber Mixtures

	Wool/ Hair***	Spandex	Silk	Rayon, Lyocell	Polyester, Triexta	Poly-amide-imide	PLA	Para-aramid	Olefin	Nylon	Mod-acrylic	Meta-aramid	Mela-mine	Cotton, Hemp, Linen, Ramie	Acrylic
Acetate	1 4 12 (5)	1	1 12 (5)	1 12	1 12 (9)	1 12	1 12	1 12	1 (10)	1 12 (2)	N/A	1 12	1 12	1 12	1 12
Acrylic	7 8 (5)	(12)	7 8 (3) (5)	7 8 (3)	7 8 (9)	7 8	7 8	7 8	7 8 (10) (12)	7 8 (2) (3) (6)	(1)	7 8	7 8	7 8	
Cotton, Hemp, Linen, Ramie, Jute, Sisal	4 (5)	(7) (8) (12)	(5)	(3)	4 (9)	4	4	4	4 (10) (12)	(2) (6)	4 (1)	4 (11)	4		
Melamine	(5)	(7) (8) (12)	(3) (4) (5)	(3) (4)	(9)	—	—	—	(10) (12)	(2) (6)	(1)	(11)			
Meta-aramid	(5)	(7) (8) (12)	(3) (4) (5)	(3) (4)	(9)	11	—	11	(10) (12)	(2) (6)	(1)				
Modacrylic	(5)	1*	1* (3) (4) (5)	1* (3) (4)	1* (9)	1*	1*	1*	1* (10) (12)	1* (2) (6)					
Nylon	2** 3 6 (5)	2** 3 6 (7) (8) (12)	(5)	2** 6	2** 3 6 (9)	2** 3 6	2** 3 6	2** 3 6	2** 3 6 (10) (12)						
Olefin	10 12 (5)	10 (7) (8)	10 12 (3) (4) (5)	10 12 (4)	10 12 (9)	10 12	10 12	10 12							
Para-aramid	(5)	(7) (8) (12)	(3) (4) (5)	(3) (4)	(9)	—	—								
PLA	(5)	(7) (8) (12)	(3) (4) (5)	(3) (4)	(9)	—									
Polyamide-imide	(5)	(7) (8) (12)	(3) (4) (5)	(3) (4)	(9)										
Polyester, Triexta	(5)	9 (7) (8) (12)	(3) (4) (5)	9 (3) (4)											
Rayon, Lyocell	4 (5)	(7) (8) (12)	(5)												
Silk	3 4	(7) (8) (12)													
Spandex	7 8 12														

Notes:
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.
NA = Chemical Methods not applicable to separate two different fiber types of the same generic class. Use the microscopy sections of AATCC TM20A.
— = Method Under Development.
Section 11.2 contains details of table use.

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
8	N,N-dimethylacetamide	section 12.8
9	alkaline methanol	section 12.9
10	xylenes	section 12.10
11	4% lithium chloride in N,N-dimethylacetamide	section 12.11
12	Cyclohexanone	section 12.12

*Not suitable for all modacrylic fibers
**Not suitable for all nylon fibers
***Hair fibers referenced in AATCC TM20A are synonymous with fibers with scales on surface in Table I of AATCC TM20

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 17.6) 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 17.7).

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 17.6) 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.