

Test Method for Fiber Analysis: Qualitative

1. Purpose and Scope

1.1 This test method describes physical, chemical and microscopical techniques for identifying textile fibers found in textile products and sold in the United States. Fibers may be examined in raw fiber form or taken from yarn or fabric.

1.2 These test methods may be used to identify generic fiber types as defined by the Textile Fibers Products Identification Act and subsequent rules and regulations of the Federal Trade Commission or ISO 2076, Textiles man-made fibres-generic names. Quantitative methods for determining percentages in blends of fibers are covered by AATCC TM20A, Test Method for Fiber Analysis: Quantitative.

1.3 The test methods apply to fibers which are shown below grouped by generic classifications:

Natural Fibers

Cellulose (Vegetable)

cotton
hemp
jute
linen
ramie
sisal (agave)
manila hemp (abaca)

Keratin (Animal)

alpaca
camel
cashmere
horse
llama
mohair
rabbit
vicuna
wool
yak

Fibroin (Animal)

silk
Bombyx (cultivated)
tussah (wild)

Mineral

asbestos

Man-Made Fibers

acetate
secondary triacetate
acrylic
anidex
aramid
meta aramid
para aramid
azlon
glass
metallic
modacrylic
novoloid
nylon
6
6,6
11
nytril
olefin
lastol
polyethylene
polypropylene
polybenzimidazole
polyester
elastrelle
rayon
cuprammonium
lyocell
viscose
rubber
saran
spandex
triexta
vinyl
vinyon

amination is particularly useful in characterizing the natural fibers. It must be used with caution on man-made fibers since they are frequently produced in a number of modifications which alter the longitudinal or cross-sectional appearance. In addition, man-made fibers may contain some or no delusterant or other additive particles. Filaments of a given type may vary in size or cross-sectional shape. Individual filaments may have two or more component sections of the same or different generic types.

2.1.2 Even natural fibers show a fairly wide variation in typical cross-section. No specific specimen will look exactly like the pictures published. A sufficient number of fibers should be examined to cover the range of appearance in any specimen.

2.2 Successful identification of fibers depends upon experience and familiarity with the fibers. The identification of an unknown fiber is best made by comparison with properly identified fibers used as reference standards. For this reason it is desirable to have available at least one representative fiber sample from each generic class of fibers, which can be used for comparative identification.

2.3 This test method provides means for identifying the generic classification of the common fiber types. In special cases, as when dealing with fibers not described in this method or attempting to distinguish between products of different suppliers of the same generic types, one must consult standard texts on fiber identification or technical bulletins issued by suppliers of man-made fibers. See references Section 13.

3. Terminology

3.1 For definitions of technical terms, refer to the AATCC M11 in this *Manual of International Test Methods and Procedures*.

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 In preparing, dispensing and handling the reagents outlined in Section 6, 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.

4.4 All poisonous and flammable reagents should be mixed and handled only in an adequately ventilated laboratory hood. CAUTION: Acetone and ethyl alcohol are highly flammable and should be stored in the laboratory only in small containers away from heat, open flame and sparks.

4.5 An eyewash/safety shower should be located nearby and an organic vapor respirator should be readily available for emergency use.

4.6 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 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 12.1).

5. Apparatus (see 12.2)

5.1 Compound microscope with matched objectives and eye pieces to achieve magnifications of 100-500× and equipped with a polarizer and analyzer.

5.2 Glass slides and cover glasses.

5.3 Dissection needles.

5.4 Scissors (small) and tweezers (fine).

5.5 Cross-sectioning device of one type such as listed below.

5.5.1 Stainless steel plate, 2.54 × 7.62 × 0.0254 cm (1 × 3 × 0.01 in.) drilled with 0.09 cm (0.04 in.) diameter holes. Soft copper magnet wire AWG #34, 0.016 cm (0.0063 in.) diameter.

5.5.2 Microtome, hand.

5.6 Razor blades, sharp, thin, single edge or double edge with holder.

5.7 Density gradient tube, glass approximately 2.5 cm (1 in.) in diameter

and 45 cm (18 in.) long with a sealed bottom and a 24/40 standard taper joint glass cap closure at top to avoid moisture pickup or evaporation of solvents. Tiny glass spheres of calibrated density may be used as density standards.

5.8 Melting point apparatus consisting of a heated block, temperature measuring device such as a thermometer and means for controlling the rate of heating and viewing the specimen at low magnification. The instrument should have a range of 100-300°C or more, and an accuracy of ± 1 degree over the entire range.

5.9 Micro-FTIR Instrument.

5.10 Differential Scanning Calorimeter.

6. Reagents and Materials (see 12.2)

6.1 Mounting Reagents.

6.1.1 Mineral oil, U.S.P. or other immersion fluid.

6.1.2 Collodion, solution of nitrocellulose (4 g/100 mL) in 1:3 alcohol/ethyl ether.

6.2 Bleaching Reagent.

6.2.1 Hydrosulfite-caustic solution. Dissolve 2 g sodium hydrosulfite and 2 g sodium hydroxide in 100 mL of water.

6.3 Staining Reagents.

6.3.1 Zinc chloro-iodide reagent. Dissolve 20 g of zinc chloride in 10 mL of water. Add 2.1 g of potassium iodide and 0.1 g of iodine dissolved in 5 mL of water. Add a leaf of iodine.

6.3.2 Acid phloroglucinol reagent. Dissolve 2 g of phloroglucinol in 100 mL of water. Use with equal volume of concentrated hydrochloric acid.

6.4 Refractive Index Immersion Liquids.

6.4.1 Hexadecane (cetane) C. P. Grade, RI = 1.434.

6.4.2 Alpha chloronaphthalene, RI = 1.633. Poisonous. Avoid inhaling vapors.

6.4.3 Blends of the above. Assume that the refractive index varies linearly by volume of ingredients. For example, to make a liquid of RI = 1.550 mix 42 parts by volume of hexadecane with 58 parts by volume of alpha chloronaphthalene.

6.5 Fiber Solvents.

6.5.1 Acetic acid, glacial. Corrosive. Do not get in eyes or on skin.

6.5.2 Acetone, reagent grade. (CAUTION: Highly flammable.)

6.5.3 Sodium hypochlorite solution, 5%. Home laundry bleach is satisfactory.

6.5.4 Hydrochloric acid, concentrated reagent, 20%. Dilute 50 mL of concentrated hydrochloric acid, 38%, to 95 mL with distilled water.

6.5.5 Formic acid, 85%. Corrosive. Do not get in eyes or on skin.

6.5.6 1,4-Dioxane.

6.5.7 *m*-Xylene.

6.5.8 Cyclohexanone.

6.5.9 Dimethylformamide. (CAUTION: If spilled on skin, wash off immediately.)

6.5.10 Sulfuric acid solution, $59.5 \pm 0.25\%$ by weight, density 1.4929 ± 0.0027 g per mL at 20°C. Weigh into a beaker 59.5 g of concentrated sulfuric acid (sp gr 1.84). Weigh into a 250 mL Pyrex Erlenmeyer flask 40.5 g of distilled water. Cautiously add *the acid to the water* swirling and cooling in ice water or under a tap. *Wear goggles!* The solution becomes very hot and may boil and spatter if not cooled during mixing. After the solution has cooled to 20°C (68°F) adjust the density to a value between 1.4902 and 1.4956 g per mL.

6.5.11 Sulfuric acid solution, $(70 \pm 1\%)$ by weight, density 1.6105 ± 0.0116 g per mL at 20°C. Weigh 70 g of concentrated sulfuric acid (sp gr 1.84) and 30 g of water and mix with the same precautions as in 6.5.10. After the solution has cooled to 20°C (68°F) adjust the density to a value between 1.5989 and 1.6221 g per mL.

6.5.12 *m*-Cresol, reagent grade. Poisonous. Use in ventilated hood.

6.5.13 Hydrofluoric acid, 49% reagent grade. Very dangerous. Use goggles and hood under suction. Do not inhale vapors or allow to contact skin.

7. Sampling

7.1 To obtain a representative sample for identification it is necessary to consider the following:

7.1.1 If the sample is loose fiber or yarn, it may contain one fiber only or it may be an intimate blend of two or more fibers.

7.1.2 A yarn sample may be a ply or wrapped core blend of two or more yarns. These yarns in turn may be the same or different and may themselves contain a blend of fibers.

7.1.3 Woven or knitted fabrics may be constructed of yarns from one fiber type or from yarns made with multiple fiber types. Further, fabrics may be made with yarns that have different individual fiber types or multiple fiber types in the length or width directions of the fabric construction, that may require separate analysis.

7.1.4 Different fiber types may be dyed to the same color. Contrarywise, the same fiber type may appear in different colors in the finished article by mixing stock dyed or yarn dyed materials or using fibers with modified dyeing characteristics.

7.1.5 It is essential that the specimens selected represent the entire sample of raw fiber, yarn or fabric under examination.

8. Specimen Preparation

8.1 In many cases the identity of an unknown fiber can be established without pretreatment.

8.2 When the presence of starch, wax, oil or other coating obscures the appearance

of the fiber, try heating in warm to hot distilled water to remove the foreign matter. If this fails, try extracting with organic solvents, 0.5% hydrochloric acid or 0.5% sodium hydroxide. Some fibers such as nylon are damaged by acid and some such as azlon, silk and wool, are damaged by caustic treatment (see 9.7).

8.3 To separate vegetable fiber bundles, treat with 0.5% sodium hydroxide solution, rinse well with water and dry.

8.4 To strip dye from colored fibers, especially the cellulosic fibers, heat for 30 min at 50°C with hydrosulfite caustic solution (see 6.2.1).

9. Procedure

9.1 The identification of fibers is usually carried out by subjecting specimens to a variety of selected tests until enough information is obtained to make a satisfactory judgment as to the generic class or specific type. The selection of tests and order in which they are performed can change depending on the knowledge already available and results of the preliminary tests.

9.2 Visual and Microscopical Examination.

9.2.1 Examine the sample of material submitted for identification. Note form (loose fibers, yarn, fabric, etc.), color, fiber length and fineness, uniformity of appearance and probable end-use. If the sample is a fabric, separate out yarns by unraveling or cutting. If the fabric is woven, separate warp and filling yarns. If yarns differ in color, luster, size or other apparent ways, make a physical separation of these yarns for separate identification.

9.2.2 The fibers may be identified using either light or scanning electron microscopy. If using transmitted light microscopy, place a small quantity of the fibers on a glass slide, tease fibers apart, mount with a drop of mineral oil or other immersion fluid, cover with a cover slip and examine under the microscope.

9.2.3 Observe the fiber characteristics as seen in traverse section and classify into four general classes.

9.2.4 Fibers with surface scales. These are the animal hair fibers listed in Purpose and Scope (see 1.3). All of the natural protein fibers listed except silk are included in this group. Continue detailed microscopical examination including cross-section examination (see 9.3). Compare with the characteristics in Table I, photographs in the appendix of this method, and known specimens of hair fibers (see 12.3). Cashmere is the fine (de-haired) undercoat fibers produced by a Cashmere goat (*Capra hircus laniger*). The fiber is generally non-medullated and has a mean maximum diameter of 19 microns. The coefficient of variation

around the mean shall not exceed 24%. There can be no more than 3% (by weight) of cashmere fibers over 30 microns. Additional confirmation of this class may be made by burning test (see 9.5); density (see 9.6); and solubility (see 9.7).

9.2.5 Fibers with Cross Markings. These are the vegetable fibers other than cotton listed in 1.3. Continue detailed microscopical examination including cross-section examination (see 9.3). Compare with characteristics as given in Table II, photographs and known specimens of vegetable fibers. To distinguish linen and ramie from hemp, observe the direction of rotation on drying (see 9.8). If the fibers are light in color, stain with zinc chloroiodide reagent and with acid phloroglucinol as directed in 9.9. Additional confirmation of this class may be made by tests described in 9.4, 9.5, 9.6 and 9.7.

9.2.6 Convoluted Fibers. This class includes cotton and tussah silk. The two are easily distinguished by cross-section (see 9.2), burning test (see 9.5) and solubility (see 9.7) or micro-FTIR (see Appendix II). If fibers are light in color, they may be distinguished by staining with zinc chloroiodide reagent (see 9.9).

9.2.7 Other Fibers. This class includes all of the man-made fibers, Bombyx silk and asbestos. The latter two can be identified by microscopical appearance including cross-section (see 9.3). Burning tests (see 9.5) and solubility tests (see 9.7) are especially significant for asbestos and are useful in confirming the presence of silk.

The man-made fibers are best identified by micro-FTIR, solubility, melting point, refractive indices and other optical characteristics and density: properties which relate to chemical nature rather than physical shape. Cross-sections may vary and staining tests can be misleading since modifications of a generic type may dye differently. Used with discrimination and in combination with other tests, cross-sections and dye tests may sometimes be helpful in narrowing down the number of possibilities. Metallic fibers have a distinctive shiny appearance, but a shiny residue may not indicate a metallic fiber. Using a transmitted light microscope at 5×, 10× and 20× magnification powers will not reveal diffraction, interference and gloss effects that can deceptively appear to be metallic in nature. Chemical (colorimetric) and/or instrumental (Atomic Absorption Spectrophotometric, Inductively Coupled Plasma Spectrometry and/or Energy Dispersive X-ray or X-ray Fluorescence) analyses should be performed to confirm their presence.

Examples of x-ray and elemental analysis are provided in the *AATCC Fiber Identification Supplement* (see 12.4).

9.3 Microscopical Cross-Section Examination.

Table I—Characteristics of Fibers with Scales on Surface

Note: The characteristic designated by a capital letter X in this table is that which is especially significant.

Microscopical Appearance	Alpaca	Camel	Cashmere ^{d,e}	Horse	Llama	Mohair	Vicuna	Wool ^c	Yak
Longitudinal:									
Epidermis:									
Pronounced	—	—	—	—	—	—	—	X	—
Faint	x	x	X	x	x	X	x	—	x
Coronal ^a	—	x	x	X	—	—	x	x	x
Imbricate ^b	x	x	—	—	x	x	—	x	x
Smooth edge	—	x	x	x	—	x	x	x	—
Serrated edge	X	—	—	x	X	—	—	—	x
Medulla:									
Occurrence:									
Usually present	x	—	—	x	X	—	—	—	—
Seldom present	—	x	X	—	—	x	x	x	—
Never present	—	—	—	—	—	—	—	—	x
Type:									
Fragmental	x	x	—	—	x	—	x	x	—
Interrupted	x	—	—	—	x	x	x	x	—
Continuous	x	—	—	x	x	x	—	—	—
Size (ratio to fiber diameter):									
Under ¼	—	—	—	—	—	x	x	x	—
¼ to ½	x	—	—	x	x	x	—	x	—
Over ½	—	—	—	X	—	—	—	—	—
Pigment:									
Diffuse	—	—	x	—	—	—	—	—	—
Streaky	x	X	x	—	x	—	x	—	x
Granular	—	x	—	x	—	—	—	—	x
None	—	—	—	—	—	X	—	X	—
Cross-Section:									
Contour:									
Round to oval	—	x	x	x	—	x	x	x	x
Oval to elongated	x	—	—	—	x	—	—	x	—
Kidney	x	—	—	—	x	—	—	—	—
Medulla contour:									
Round to oval	—	—	—	x	—	x	x	x	—
Oval to elongated	X	—	—	—	X	—	—	—	—
Kidney to dumbbell	X	—	—	—	X	—	—	—	—
Pigment distribution:									
Uniform	x	X	—	—	x	—	x	—	x
Centric	—	—	X	—	—	—	—	—	—
Eccentric	—	—	—	X	—	—	—	—	—
Fineness (in µm)									
Average	26 to 28	18	15 to 19.0	—	26 to 28	—	13 to 14	—	18 to 22
Range	10 to 50	9 to 40	5 to 30	—	10 to 40	10 to 90	6 to 25	10 to 70	8 to 50
Number of scales per 100 µm									
	—	—	6 to 7	—	—	Under 5.5	—	Over 5.5	Over 7

^a Coronal means crownlike, and refers to scales in which the visible scale edge completely encircles the fiber.

^b Imbricate means overlapping, and refers to scales in which the visible scale edges overlap like shingles on a roof and cover only a part of the fiber circumference.

^c The term wool is used here to represent clothing wool and not carpet wool.

^d The longitudinal/epidermal appearance of cashmere fiber while generally fainter than that of sheep's wool is not as faint as some specialty fibers such as camel and alpaca.

^e The average diameter for cashmere fibers was broadened in 2000 based on a review of published research concerning Asian *capra hircus*.

Table II—Appearance of Fibers with Cross Markings or Swellings

Longitudinal Section:	Linen	Hemp	Ramie
Ratio, lumen of fiber diameter	under ⅓	usually over ⅓	over ⅓
Cell ends	pointed	blunt or forked	blunt
Cross-Section:			
Contour	sharp polygon	rounded polygon	elongated polygon
Lumen	round or oval	irregular	irregular

9.3.1 Obtain a parallel bundle of fibers or yarn. Thread a loop of copper wire through one hole in the stainless steel plate. Catch the bundle or yarn in the loop and pull it through the hole. Use suf-

ficient fibers to pack the hole full. If necessary, use some extra, readily identifiable other fiber to fill up the hole.

9.3.2 With a sharp razor blade make a smooth cut on both sides of the plate.

9.3.3 Examine the section in air, or covered by a mounting fluid like mineral oil under a cover slip. Use transmitted light and useful magnification of 200–500×. Compare with photographs in Appendix or cross-sections of known fibers.

9.3.4 If the Hardy type microtome is used, follow instructions with the instrument. Insert a bundle of fibers or yarn into the slot. Slide the tongue into the slot to compress the bundle. Adjust the quantity of fibers for a tight package. Cut off both sides. Apply a drop of collodion to one cut face, wait until it shows through on the other side and then apply a drop to the other side. Let dry thoroughly. Slice off excess collodion and fiber on both sides of the plate with a sharp razor blade.

9.3.5 Attach the auxiliary plunger, and with the screw advance the embedded tuft through the slot until it protrudes 20–40 µm above the plate. Add a drop of collodion and let dry about 5 min or until firm. Slice off collodion and embedded fiber section with a sharp razor blade held at about 45° angle with the slot. Cut with a single stroke.

9.3.6 Remove the section, transfer to a glass slide, mount with a drop of mineral oil or other immersion liquid, add a cover slip and examine. Continue to cut sections as described in 9.3.5 until good clear cross-sections are obtained. Examine and compare to known cross-sections.

9.4 Refractive Index.

9.4.1 Place a small sample of the fiber on a glass slide. Immerse in a drop of a chloronaphthalene and cetane mixture (or equivalent) of refractive index 1.55.

9.4.2 Insert the polarizer into the substage of the microscope to provide light polarized in the 6–12 o'clock direction. Align the long dimension of a fiber in the same direction. Close the diaphragm of the substage condenser so as to provide axial illumination.

9.4.3 Focus carefully on the outlines of the fiber. With the fine adjustment, raise the focus to just above the top of the fiber. If the fiber is roughly a cylinder, it will act like a lens. If the refractive index of the fiber is *higher* than the immersion liquid, the fiber will act like a positive lens. A bright line of light will move into the middle of the fiber as the focus is raised. If the refractive index of the fiber is *lower* than the immersion liquid, the light will flare out as the focus is raised and the middle of the fiber will become darker. Repeat until certain of the direction.

9.4.4 The test can be seen best with round fibers. On flat ribbons it may be easier to see movement of a bright line—the Becke line—at the outlines of the fiber. Movement is in the same direction, toward the medium of higher refractive index as the focus is raised.

9.4.5 Rotate the specimen 90° and repeat the test.

9.4.6 Rotate the specimen 45°. Insert the analyzer in the body tube or eyepiece to provide crossed polars. Observe if the fiber appears very bright (strong birefringence), dim (weak birefringence) or dark (no birefringence).

9.4.7 Refer to Table IV for refractive index of fibers in the long and cross direction and an estimate of birefringence. Consider that the retardation depends on the thickness of the fiber specimen as well as the extent of drawing of a given specimen. Compare with known fibers of similar diameter for birefringence.

9.4.8 Select other immersion liquids and repeat 9.4.1 through 9.4.5. As the refractive index of the liquid approaches that of the fiber, the outlines of the fiber become less distinct. Match the liquid to the fiber within 0.01 refractive index.

9.5 Burning Test.

9.5.1 Take a small tuft of fibers (held in tweezers) and place close to the side of a small flame. Note if the fibers melt or shrink from the flame.

9.5.2 Move the fibers into the flame. Note whether the fibers will burn when held in the flame. Remove from the flame very slowly and carefully. Note whether

the fibers continue to burn outside of the flame. Make sure that the fiber was ignited before making this latter observation.

9.5.3 Blow out the flame, if still burning, and smell the smoke. Note the odor and examine the color and nature of any ash residue.

9.5.4 Compare the behavior observed with that listed in Table III and that of known reference fibers. Flame retardant modifications of some fibers—cotton, rayon, acetate and modacrylic, for example—are available. Burning is retarded; odor on burning and ash may be changed. Colored fibers—especially those colored by pigments—will retain color in the colored residue.

9.5.5 Odors of some burning fibers are quite characteristic. The animal fibers and man-made protein fibers (azlon) have the odor of burning hair or feathers. The vegetable fibers and regenerated cellulose (rayon) smell like burning paper. The smell of burning rubber is a familiar odor. Other man-made fibers—such as acrylic, nylon and spandex—have characteristic odors which can be recognized with experience.

9.6 Density.

9.6.1 Prepare a density gradient col-

Table III—Reaction to Flame

	Melts Near Flame	Shrinks From Flame	Burns in Flame	Continues to Burn	Appearance of Ash
Natural Fibers					
silk	yes	yes	yes	slowly	soft black bead
wool	yes	yes	yes	slowly	irregular black
cellulose	no	no	yes	yes	light grayish
asbestos	no	no	no	no	may blacken
Man-Made Fibers					
acrylic	yes	yes	yes	yes	hard black
acetate					irregular
azlon					shaped
nytril					bead
polybenzimidazole	no	no	no	no	may blacken
polyester	yes	yes	yes	yes	hard black
					round bead
nylon	yes	yes	yes	yes	hard gray
					round bead
olefin	yes	yes	yes	yes	hard tan bead
vinal					
modacrylic	yes	yes	yes	no	hard black
saran					irregular bead
vinylon					
metallic	yes	yes	no	no	metal bead
glass	yes	slowly	no	no	hard clear
					bead
rubber	yes	yes	yes	no	irregular mass
spandex	yes	no	yes	yes	fluffy black
					or gray
anidex	yes	no	yes	yes	brittle black
					irregular bead
rayon	no	no	yes	yes	none
aramid	no	yes	yes	no	hard black
					bead
novoloid	no	no	brief	no	carbon
trixta	yes	yes	yes	yes	hard black
					round bead

Table IV—Physical Properties of Fibers

	Refractive Index		Birefringence	Density g/cc	Melting Point Degree C
	Long	Cross			
Natural Fibers					
asbestos (chrysotile)	1.50-1.55	1.49	strong	2.1-2.8	over 350
cellulose	1.58-1.60	1.52-1.53	strong	1.51	none
silk	1.59	1.54	strong	1.32-1.34	none
wool and other hair	1.55-1.56	1.55	weak	1.15-1.30	none
Man-Made Fibers					
acetate, secondary	1.47-1.48	1.47-1.48	weak	1.32	260
acetate, tri.	1.47-1.48	1.47-1.48	weak	1.30	288
acrylic	1.50-1.52	1.50-1.52	weak, negative	1.12-1.19	none
anidex		opaque	—	1.22	softens at 190
aramid	—	—	strong	1.38	chars at 400
azlon	1.53-1.54	1.53-1.54	none	1.30	none
glass	1.55	1.55	none	2.4-2.6	850
metallic		opaque	—	varies	over 300
modacrylic	1.54	1.53	weak	1.30 or 1.36	188* or 120
novoloid		1.5-1.7	none	1.25	none
nylon	1.57	1.51	strong	1.12-1.15	213-225
nylon 6,6	1.58	1.52	strong	1.12-1.15	256-265
nytril	1.48	1.48	nil	1.20	218
polybenzimidazole	>1.70	>1.70	moderate	1.4	none
polyester	1.71-1.73 or 1.63	1.53-1.54	intense	1.38 or 1.23	250-260 or 282
polyethylene	1.56	1.51	strong	0.90-0.92	135
polypropylene	1.56	1.51	strong	0.90-0.92	170
rayon	1.54-1.56	1.51-1.53	strong	1.51	none
rubber		opaque	—	0.96-1.06	none
saran	1.61	1.61	weak, negative	1.70	168
spandex		opaque	—	1.20-1.21	230
triexta	1.57	—	—	1.33	226-233
vinal	1.55	1.52	strong	1.26-1.30	—
vinylon	1.53-1.54	1.53	weak, negative	1.34-1.37	230 or 400

*Not sharp. Sticking point is 176°C.

umn as follows. Clamp the density gradient glass tube in a firm vertical stand. Pour into the tube 25 mL of tetrachloroethylene. Now prepare mixtures of xylene and tetrachloroethylene by volume in descending order of percent tetrachloroethylene: 90/10, 80/20, 70/30, 60/40, 50/50, 40/60, 30/70, 20/80 and 10/90. Pour 25 mL of each in order carefully down the side of the gradient tube. Finally, put 25 mL of xylene on top.

9.6.2 Take short fragments of dyed reference fibers and tie in a knot snipping off loose ends. Boil for about 2 min in xylene solvent to remove moisture and air. Place in the column. After about a half hour they should come to rest at the level representing their density. Calibrated glass spheres may be used to determine the actual density at various levels.

9.6.3 Prepare the unknown fiber in like manner, place in the gradient column and note at what level it floats. Densities of fibers are listed in Table IV.

9.7 Solubility Test.

9.7.1 For tests at room temperature (20°C) place a small sample of the fibers in a watch crystal, test tube or 50 mL beaker and cover with the test solvent (see Table V). Use about 1 mL of solvent per

10 mg of fiber.

9.7.2 If the test is conducted at the boiling point of the solvent, first bring the solvent to a boil in a beaker on an electric hot plate in a ventilated hood. Adjust the hot plate temperature to maintain slow boiling and keep watch so that the solvent does not boil dry. Drop fiber sample into the boiling solvent.

9.7.3 If the test is conducted at some intermediate temperature, heat a beaker of water on a hot plate and adjust the temperature with a thermometer. Place the fiber sample in the test solvent in a test tube and immerse in the heated water bath.

9.7.4 Note if the fiber dissolves completely, softens to a plastic mass or remains insoluble. Compare with data on fiber solubility in Table V.

9.7.5 Solubility can also be used as a test to determine the presence of a metal component in a fiber. On dissolving in meta-cresol a shiny residue is evidence of a metal component.

9.8 Drying Twist Test.

9.8.1 Separate out a few parallel fibers. Dip in water and squeeze off excess. Tap the end of the bundle to make separate fibers flare out. Hold over a hot plate in

warm air to dry the fibers. Hold the fibers so that the free ends point toward the observer. Note the direction of twist as the fibers dry. Linen and ramie twist in a clockwise direction; hemp and jute twist in a counter clockwise direction.

9.9 Stain Tests.

9.9.1 Place a few fibers on a microscopical slide. Apply a drop of zinc chloro-iodide reagent and cover with a cover slip taking care to avoid bubbles. Examine the fibers for staining. Hemp, ramie and cotton stain violet; linen stains brownish violet; jute stains brown. Many other fibers stain shades of yellow brown including silk.

9.9.2 Warm a few fresh fibers on a microscopical slide with a drop of acid phloroglucinol reagent. Woody fibers such as unbleached jute stain a deep magenta because of the presence of lignin.

9.10 Melting Point.

9.10.1 Melting Point Apparatus.

9.10.2 Place a few fibers on the clean heating block. Cover with a cover slip. Turn the heat on and set the heating rate for fast heating. Observe the thermometer and the sample. When the temperature reaches 100°C, reduce the rate of heating. (If previous tests already indicate the fiber, the rate can be set at about 10°C per min until 10-20°C below the expected melting point.) Near the melting point the rate should be reduced to about 2°C per min.

9.10.3 Observe the fibers for evidence of softening. At the melting point, liquid begins to form which wets the cover slip. Fibers eventually merge and form a liquid mass. As the test is in progress it is sometimes helpful to press gently on the cover slip with tweezers to see if the fibers flatten under pressure. If this melting point is passed at a high heating rate, repeat the test with a fresh sample.

9.10.4 Compare the melting point found with those listed in Table IV.

9.10.5 Microscope Mounted Apparatus.

9.10.6 Place a few fibers on one of the small glass slides provided and cover with a cover slip. Place the slide on the stage of the microscope with the fibers over the central hole of the stage. Put the baffle and cover plate in place.

9.10.7 Insert the polarizing accessories in the optical path of the microscope to provide crossed polars. The fibers should be visible if aligned diagonally to the direction of polarization. If fibers are not visible under these lighting conditions, remove the polarizing accessories and observe in ordinary transmitted light.

9.10.8 Set a high heating rate with the voltage regulator until the temperature reaches 100°C. Reduce the rate as the expected melting point is approached as directed in 9.10.2.

9.10.9 Observe the fibers. As melting takes place there is a decrease in birefrin-

Table V—Solubility of Fibers

	acetic acid	acetone	sodium hypochlorite	hydrochloric acid	formic acid	1,4 dioxane	m-xylene	cyclohexanone	dimethyl formamide	sulfuric acid	sulfuric acid	m-cresol	hydrofluoric acid
Concentration (%)	100	100	5	20	85	100	100	100	100	59.5	70	100	50
Temperature (°C)	20	20	20	20	20	101	139	156	90	20	38	139	50
Time (minutes)	5	5	20	10	5	5	5	5	10	20	20	5	20
acetate	S	S	I	I	S	S	I	S	S	S	S	S	I
acrylic	I	I	I	I	I	I	I	I	S	I	I	P	I
anidex	I	I	I	I	I	I	I	I	I	I	I	I	I
aramid	I	I	I	I	I	I	I	I	I	I	I	I	I
azlon	I	I	S	I	I	I	I	I	I	I	S	I	I
cotton & linen	I	I	I	I	I	I	I	I	I	I	I	I	I
glass	I	I	I	I	I	I	I	I	I	I	I	I	S
modacrylic	I	SE	I	I	I	SP	I	S	SP*	I	I	P	I
novoloid	I	I	I	I	I	I	I	I	I	I	I	I	†
nylon	I	I	I	S	S	I	I	I	S	S	S	S	I
nytril	I	I	I	I	I	I	I	S	S	I	I	SP	I
olefin	I	I	I	I	I	I	S	S	I	I	I	I	I
polybenzimidazole	I	I	I	I	I	I	I	I	I	I	I	I	I
polyester	I	I	I	I	I	I	I	I	I	I	I	I	I
rayon	I	I	I	I	I	I	I	I	I	S	S	I	I
saran	I	I	I	I	I	S	S	S	S	I	I	I	I
silk	I	I	S	I	I	I	I	I	I	S	S	I	I
spandex	I	I	I	I	I	I	I	S	S	SP	SP	SP	I
teflon	I	I	I	I	I	I	I	I	I	I	I	I	I
triexta	I	I	I	I	I	I	I	I	I	I	I	S	I
vinyl	I	I	I	S	S	I	I	I	I	S	S	I	I
vinylon	I	S	I	I	I	S	S	S	S	I	I	S	I
wool	I	I	S	I	I	I	I	I	I	I	I	I	I

S = Soluble

I = Insoluble

P = Forms plastic mass

SP = Soluble or forms plastic mass

SE = Soluble except for one modacrylic fiber characterized by low flammability and liquid inclusions visible in cross-section.

* = Soluble at 20°C without plastic mass.

† = Novoloid turns red

gence; the fibers become dark. Read as melting point the temperature at which the fibers become completely dark. If crossed polars are not used, observe melting as described in 9.10.2.

9.10.10 Compare the melting point found with those listed in Table IV.

9.11 Micro-FTIR.

9.11.1 Compare FTIR spectra with FTIR spectra in Appendix II or other library sources (see Appendix II—Figs. 1-9).

10. Report

10.1 Report the fiber type and, if more than one type is present, where each is located. For example, the report could be “woven fabric with nylon 6,6 warp and cotton/rayon filling.”

11. Precision and Bias

11.1 A precision and bias statement is not applicable because data are not generated by this method.

12. Notes

12.1 Available from Publications Office, ACGIH, Kemper Woods Center, 1330 Kemper Meadow Dr., Cincinnati OH 45240; tel: +1.513.742.2020; web site: www.acgih.org.

12.2 For potential equipment information pertaining to this test method, please visit the online *AATCC Buyer's Guide* at www.aatcc.org/bg. AATCC provides the possibility of listing equipment and materials sold by its Corporate members, but AATCC does not qualify, or in any way approve, endorse or certify that any of the listed equipment or materials meets the requirements in its test methods.

12.3 Reference samples, along with interlab data, of cashmere and wool, both separate and combined, are available from AATCC, P.O. Box 12215, Research Triangle Park NC 27709; tel: +1.919.549.8141; fax: +1.919.549.8933; e-mail: ordering@aatcc.org; web site: www.aatcc.org.

12.4 Available from AATCC, P.O. Box 12215, Research Triangle Park NC 27709; tel: +1.919.549.8141; fax: +1.919.549.8933; e-mail: ordering@aatcc.org; web site: www.aatcc.org.

13. References

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13.4 Wildman A. B., *The Microscopy of Animal Textile Fibers*, Wool Industries Research Association, Torridon, Leeds, England, 1954.

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13.11 IWTO Draft Test Method 58-97, “Quantitative Analysis of Blends of Wool with Specialty Fibers by Scanning Electron Microscope.” Describes method for distin-