Standard Test Methods for
Identification of Fibers in Textiles[1]

This standard is issued under the fixed designation D 276; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

Methods D 276 – 62 T, Identification of Fibers in Textiles were discontinued in 1969 because the responsible subcommittee failed to recommend adoption as standard after several years publication as a tentative. The subcommittee action was based on the members' knowledge that the standard did not include several fiber types introduced to the textile trade after the methods were published, and that the techniques required to identify these fibers were lacking in the text, so that the text had become out of date. Reinstatement as a standard using the previously assigned designation was requested since the listed procedures were reliable and the text was considered to be the best available, though not all-inclusive. Extensive editorial changes were made in various sections in 1972, and the methods were reinstated as D 276 – 72.

The methods have been revised completely, emphasizing infrared spectroscopic techniques for identifying man-made fiber types. Methods for determining several physical properties and solubility data useful for confirming infrared spectral identifications have been included. The longitudinal and cross-section photographs of the various fibers have been omitted since they are published elsewhere and the usefulness for identification is limited. Extensive editorial changes have been made throughout the text.

AATCC Test Method 20 was first published in 1947 and has been revised or reaffirmed on a regular basis since that time. The most current version is AATCC "Test Method 20–1999"[2].

1. Scope

1.1 These test methods cover the identification of the following textile fibers used commercially in the United States:

Acetate (secondary)	Nylon
Acrylic	Nytril
Anidex	Olefin
Aramid	Polycarbonate
Asbestos	Polyester
Cotton	Ramie
Cuprammonium rayon	Rayon (viscose)
Flax	Saran
Fluorocarbon	Silk
Glass	Spandex
Hemp	Triacetate
Jute	Vinal
Lycocell	Vinyon
Modacrylic	Wool
Novoloid	

1.2 Man-made fibers are listed in 1.1 under the generic names approved by the Federal Trade Commission and listed in Terminology D 123, Annex A1 (except for fluorocarbon and polycarbonate). Many of the generic classes of man-made fibers are produced by several manufacturers and sold under various trademark names as follows (Note 1):

Acetate	Acele®, Aviscon®, Celanese®, Chromspun®, Estron®
Acrylic	Acrilan®, Courtelle®, Creslan®, Dralon®, Orlon®, Zefran®
Anidex	Anim/8®
Aramid	Arenka®, Conex®, Kevlar®, Nomex®, Twaron®
Cuprammonium	Bemberg®
Fluorocarbon	Teflon®
Glass	Fiberglas®, Garan®, Modiglass®, PPG®, Ultrastrand®
Lyocell	Tencel®
Modacrylic	Dynel®, Kanecaron®, Monsanto SEF®, Verel®
Novoloid	Kynol®
Polyamide (Nylon) 6	Caprolan®, Enka®, Perlon®, Zefran®, Enkalon®
Polyamide (Nylon) 6, 6	Antron®, Blue C®, Cantrece®, Celanese Phillips®, Enka®Nylon
Polyamide (Nylon) (other)	Rilsan®(nylon 11), Qiana®, StanylEnka®,(Nylon 4,6)
Nytril	Darvan®
Olefin	Durel®, Herculon®, Marvess®, Polycrest®
Polyester	Avlin®, Beaunit®, Blue C®, Dacron®, Encron®, Fortrel®, Kodel®, Quintess®, Spectran®, Trevira®, Vyoron®, Zephran®, Diolen®, Vectran®
Rayon	Avril®, Avisco®, Dynacor®, Enka®, Fiber 700®, Fibro®, Nupron®, Rayflex®, Suprenka®, Tyrex®, Tyron®, Cord-enka®
Saran	Enjay®, Saran®
Spandex	Glospun®, Lycra®, Numa®, Unel®

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[1] These test methods are under the jurisdiction of ASTM Committee D13 on Textiles and are the direct responsibility of Subcommittee D13.51 on Chemical Conditioning and Performance.

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

| Triacetate | Arnel® |
| Vinyon | Avisco®, Clevyl®, Rhovyl®, Thermovyl®, Volpex® |

NOTE 1—The list of trademarks in 1.2 does not include all brands produced in the United States or abroad and imported for sale in the United States. The list does not include examples of fibers from two (or more) generic classes of polymers spun into a single filament. Additional information on fiber types and trademarks is given in References (1, 2, and 3).[3]

1.3 Most manufacturers offer a variety of fiber types of a specific generic class. Differences in tenacity, linear density, bulkiness, or the presence of inert delustrants normally do not interfere with analytic tests, but chemical modifications (for such purposes as increased dyeability with certain dyestuffs) may affect the infrared spectra and some of the physical properties, particularly the melting point. Many generic classes of fibers are sold with a variety of cross-section shapes designed for specific purposes. These differences will be evident upon microscopical examination of the fiber and may interfere with the measurements of refractive indices and birefringence.

1.4 Microscopical examination is indispensable for positive identification of the several types of cellulosic and animal fibers, because the infrared spectra and solubilities will not distinguish between species. Procedures for microscopic identification are published in AATCC Method 20 and in References (4-12).

1.5 Analyses by infrared spectroscopy and solubility relationships are the preferred methods for identifying man-made fibers. The analysis scheme based on solubility is very reliable. The infrared technique is a useful adjunct to the solubility test method. The other methods, especially microscopical examination are generally not suitable for positive identification of most man-made fibers and are useful primarily to support solubility and infrared spectra identifications.

1.6 This includes the following sections:

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* See Note 3.

2. Referenced Documents

2.1 *ASTM Standards:*

D 123 Terminology Relating to Textiles[4]
D 629 Test Methods for Quantitative Analysis of Textiles[4]
D 789 Test Methods for Determination of Relative Viscosity, Melting, Point, and Moisture Content of Polyamide (PA)[5]
D 792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement[5]
D 941 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Lipkin Bicapillary Pycnometer[6]
D 1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer[6]
D 1505 Test Method for Density of Plastics by the Density-Gradient Technique[5]
D 1776 Practice for Conditioning Textiles for Testing[4]
E 131 Terminology Relating to Molecular Spectroscopy[7]
E 175 Terminology of Microscopy[8]
2.2 *AATCC Method:*
Test Method 20 for Identification of Fibers in Textiles[2]

3. Terminology

3.1 *Definitions:*

3.1.1 *birefringence (double refraction), n*— a property of anisotropic materials which manifests itself as a splitting of a light ray into components having different vibration directions which are transmitted at different velocities.

3.1.1.1 *Discussion*—The vibration directions of the components are the principal axes of the material and the corresponding indices of refraction are its principal (maximum or minimum) refractive indices. Numerically, birefringence is the difference between the maximum and minimum refractive indices.

3.1.2 *density*—mass per unit volume.

3.1.2.1 *Discussion*—Due to the volume of included air, the apparent density of fibers and yarns will differ from the densities of the materials of which the fibers and yarns are composed. Test results for fiber density will also vary depending on the test method used. Density is commonly expressed as grams per cubic centimetre (g/cm^3), but the preferred term in the International System of units is kilograms per cubic metre (kg/m^3). Multiply g/cm^3 by 1000 to obtain kg/m^3 and multiply lb/ft^3 by 16.018 to obtain kg/m^3.

3.1.3 *fiber birefringence, n*—the algebraic difference of the index of refraction of the fiber for plane polarized light vibrating parallel to the longitudinal axis of the fiber and the index of refraction for light vibrating perpendicular to the long axis.

3.1.3.1 *Discussion*—Fiber birefringence may be either positive or negative, and is not necessarily referred to the principal optical axes of the material.

3.1.4 *fiber density, n*—mass per unit volume of the solid matter of which a fiber is composed, measured under specified conditions.

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[3] The boldface numbers in parentheses refer to the list of references at the end of this method.

[4] *Annual Book of ASTM Standards*, Vol 07.01.
[5] *Annual Book of ASTM Standards*, Vol 08.01.
[6] *Annual Book of ASTM Standards*, Vol 05.01.
[7] *Annual Book of ASTM Standards*, Vol 03.06.
[8] *Annual Book of ASTM Standards*, Vol 14.04.

3.1.4.1 *Discussion*—Unless otherwise indicated, fiber density is understood to be measured by immersion (buoyancy) techniques, at 21 ± 1°C, excluding effects due to included air and swelling or dissolving of the fiber by the immersion fluid.

3.1.5 *refraction*, *n*—the deflection from a straight path undergone by a light ray in passing obliquely from one medium (as air) into another (as glass) in which its velocity is different.

3.1.6 *refractive index (index of refraction)*, *n*—the ratio of the velocity of radiation (as light) in the first of two media to its velocity in the second as it passes from one into the other.

3.1.6.1 *Discussion*—When refractive index is referred to as a property of a substance, the first medium is understood to be vacuum. The index of refraction is equal to the ratio of the sine of the angle of the incident ray to the sine of the angle of the refracted ray (angles measured from the normal to the common boundary). In general the refractive index of a substance varies with the frequency of the radiation **(13)**.

3.2 For definitions of other terms used in these test methods refer to Terminology D 123 for textiles, Terminology E 131 for terms relating to infrared spectroscopy, and Terminology E 175 for terms relating to microscopy.

4. Summary of Test Method

4.1 The fiber generic type is identified from its solubility in various reagents, using a solubility decision scheme (Fig. 1).

4.2 Alternatively, infrared spectra of fibers from textile materials to be identified are obtained using a FTIR (Fourier Transform Infrared) or a double-beam spectrophotometer. Identification of the fiber generic class is made by analysis of the fiber spectrum using a decision chart (Fig. 2).
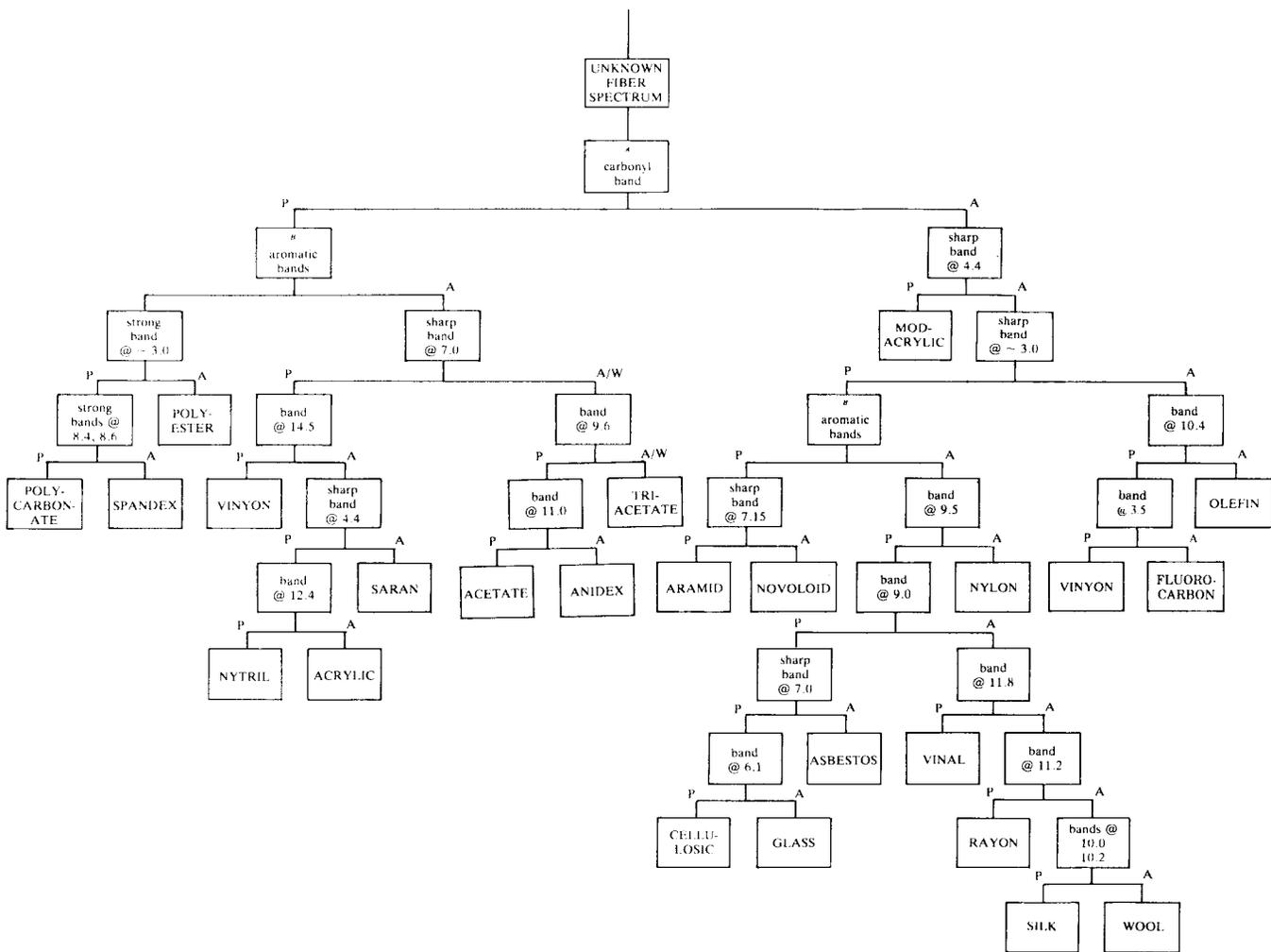

M	= melts
S	= soluble
I	= insoluble
ppt	= precipitate
RT	= room temperature

[A] Acidify with excess HCl, add lead acetate dropwise.
[B] Rinse with water, allow to dry in room air.
[C] Some modarylic fibers cannot be distinguished from acrylic fibers in this solubility scheme.

FIG. 1 Scheme for Identification of Fibers According to Solubility

UNKNOWN FIBER

acetone 10 min @ RT

cyclohexane 2 min @ boil — 5% NaOH 2 min @ boil

CHLOR-INATED PVC | acetic acid 2 min @ boil

acidify HCl / lead acetate | SARAN | rinse/dry formic acid 2 min @ RT

ACETATE | MOD-ACRYLIC

no ppt | ppt

SILK | WOOL | methylene chloride 2 min @ RT | DMF 2 min @ RT

TRIACETATE | acetic acid 2 min @ boil | methylene chloride 2 min @ RT | DMF 2 min @ 70°C

NYLON | VINAL | NYTRIL | VINYON | 70% ammonium thiocyanate 2 min @ boil | m-cresol 2 min 100°C

ACRYLIC | SPANDEX | chloroform 2 min @ RT | trichloro-ethylene 1 min @ boil

POLY-CARBONATE | POLYESTER | OLEFIN | 75% H₂SO₄ 2 min @ RT

CELLULOSE | ANIDEX ARAMID ASBESTOS FLUOROCARBON GLASS NOVOLOID

Bands located according to wavelength in m.
P = present   A = absent   A/W = absent or weak
[A] located at ~5.75
[B] located at 6.23, 6.30 & 6.70

FIG. 2 Scheme for Analysis of Infrared Spectra

4.3 For plant (native cellulose) and animal hair fibers microscopical examination of longitudinal and cross-sections is used to distinguish species.

4.4 Additional physical properties of the fiber, such as density, melting point, regain, refractive indices, and birefringence are determined and are useful for confirming the identification (see Table 1).

5. Significance and Use

5.1 These test methods are a generally reliable means of identifying the generic types of fibers present in a sample of textile material of unknown composition. The methods are generally not useful for distinguishing fibers of the same generic class from different manufacturers or for distinguishing different fiber types of the same generic class from one producer.

5.2 Many fibers are chemically modified by their producers in various ways so as to alter their properties. It is possible for such modifications to interfere seriously with the analyses used in these test methods. Considerable experience and diligence of the analyst may be necessary to resolve satisfactorily these difficulties.

5.3 Dyes, lubricants, and delustrants are not present normally in amounts large enough to interfere with the analyses.

5.4 These test methods are not recommended for acceptance testing of commercial shipments because of the qualitative

nature of the results and because of the limitations previously noted.

NOTE 2—For statements on precision and bias of the standard quantitative test methods for determining physical properties for confirmation of fiber identification refer to the cited test method. The precision and bias of the nonstandard quantitative test methods described are strongly influenced by the skill of the operator. The limited use of the test methods for qualitative identification cannot justify the effort that would be necessary to determine the precision and bias of the techniques.

6. Sampling, Selection, Preparation, and Number of Specimens

6.1 The quantity of material per specimen and the number of specimens required differ according to the types of analyses that are to be conducted. It is possible to make an identification using a sample of less than 10 mg of each type of fiber present.

6.2 In order to identify the components of a textile material reliably, it is essential that an adequate sample of each type of fiber present be isolated physically, and vice-versa. It is not possible, in general, to identify the components of a mixture by analysis of the mixture's infrared spectrum and, in fact, false conclusions may be drawn if such a procedure is attempted. Comparison of the spectra of unknown materials to a proper set of reference spectra of various fiber types (see 7.1) can be useful for avoiding these problems.

6.3 An essential first step in isolation and identification of

TABLE 1 Typical Values of Physical Properties Useful for Identifying Fibers

Fiber	Melting[A] Point °C	Refractive Index[B]		Birefringence[B] ϵ - ω	Density, mg/mm³
		Parallel to Fiber Axis, ϵ	Perpendicular to Fiber Axis, ω		
Acetate	260	1.479	1.477	0.002	1.32
Acrylic	dnm	1.524	1.520	0.004[C]	1.17
Anidex	s 190	[D]	[D]	[E]	1.22
Aramid					
Nomex®	371	1.790	1.662	0.128	1.37
Kevlar®	425	2.322	1.637	0.685	1.42
Asbestos	. . .	1.5–1.57	1.49	0.01–0.08	2.1–2.8
Cellulosic					
Flax	dnm	1.596	1.528	0.068	1.54
Cotton	dnm	1.580	1.533	0.047	1.54
Fluorocarbon	288	1.37	. . .	. . .	2.1
Glass	s 570	1.547	1.547	0.000	2.47–2.57
Modacrylic	dnm	1.536	1.531	0.005	1.28–1.37
Novoloid	dnm	1.650	1.648	0.002	1.29
Nylon					
nylon 6	219	1.568	1.515	0.053	1.14
nylon 6,6	254	1.582	1.519	0.063	1.14
Qiana®	275	1.554	1.510	0.044	1.03
nylon 4	265	1.550	. . .	. . .	1.25
nylon 11	185	1.55	1.51	0.04	1.04
Nytril	176	1.484	1.476	0.008	1.20
Olefin					
polyethylene	135	1.556	1.512	0.044	0.93
polyethylene	170	1.530	1.496	0.034	0.90
Polycarbonate	294	1.626	1.566	0.060	1.21
Polyester					
2 GT[E]	256	1.710	1.535	0.175[F]	1.38
4 GT[G]	227	1.690	1.524	0.166	1.32
CHDM-T[H]	283	1.632	1.534	0.098	1.24
oxybenzoate[I]	225	1.662	1.568	0.094	1.34
Rayon					
cuprammonium	dnm	1.548	1.527	0.021	1.53
viscose	dnm	1.547	1.521	0.026	1.52
Saran	170	1.603	1.611	-0.008	1.62–1.75
Silk	dnm	1.591	1.538	0.053	1.35
Spandex	230	1.5	. . .	. . .	1.2
Triacetate	288	1.472	1.471	0.001	1.30
Vinal	dnm	1.543	1.513	0.030	1.30
Vinyon (PVC)	dnm	1.541	1.536	0.005[D]	1.40
Wool	dnm	1.556	1.547	0.009	1.31

[A]dnm indicates the fiber does not melt, s indicates softening point.

[B] The listed values are for specific fibers which warrant the highly precise values given. For identification purposes these values should be regarded as indicating only the relative values of the properties.

[C]Varies, always weak, sometimes negative.

[D]The fiber is opaque.

[E]Ethylene glycol type.

[F]Staple and fully oriented filament yarns (FOY), partially oriented (POY) and undrawn yarns may have much lower values of birefringence and refractive index.

[G]1,4-butanediol type.

[H]1,4-cyclohexanedimethanol type.

[I] p-ethylene oxybenzoate type.

fibers is visual examination and physical separation of all visually different types of fibers in the material. In order to accomplish this, it is necessary to consider the following:

6.3.1 A single yarn may be composed of more than one type of fiber (a blend of polyester and cotton staples, for instance). In such cases it may be impractical to separate the fibers mechanically. A selective solvent (refer to Table 1 of Test Methods D 629) can be very useful in these cases, if one can be found. The density gradient column may also be used for separation.

6.3.2 A plied yarn may be made with one type of fiber in one ply and a different type in another ply.

6.3.3 Warp and filling yarns may be of different types and not every yarn in the warp (or filling) is necessarily made from the same type of fiber.

7. Reference Standards

7.1 Successful identification of fibers used in textile products depends on experience and familiarity with the fibers. An alternative test for identification of an unknown fiber is by comparison with properly identified fibers used as reference standards. It is desirable to have available authentic samples of the fibers to be identified, and it is essential to have those that are unfamiliar. A library of reference fiber infrared spectra obtained using the same techniques and instrument used for the unknown fiber is essential.

8. Purity of Reagents

8.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical

Reagents of the American Chemical Society, where such specifications are available.[9] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

FIBER IDENTIFICATION BY MICROSCOPIAL EXAMINATION

9. Scope

9.1 As previously mentioned this test method is useful for identification of various cellulosic and animal fibers and to distinguish man-made fibers form the cellulosic and animal fibers. Examine and observe the fiber characteristics as directed in the AATCC test method 20.

10. Precision and Bias

10.1 No information is presented about either the precision or bias of Test Methods D 276 for microscopical examination since the test result is nonquantitative.

FIBER IDENTIFICATION USING SOLUBILITY RELATIONSHIPS

11. Scope

11.1 This test method covers the identification of fibers by determining their solubility or insolubility in various reagents and comparing these data to the known solubilities of the several generic classes of fibers. Other techniques (such as, microscopical examination or comparison of physical properties) are used to confirm the identification or to distinguish between those fiber classes (anidex, aramid, asbestos, fluorocarbon, glass, and novoloid) which are not dissolved by any of the reagents used in this scheme.

12. Interferences

12.1 If the refractive indices of a fiber and a non-solvent liquid nearly coincide, the fiber may be virtually invisible when immersed in the liquid, even though it is not dissolved. In practice, fibers are generally sufficiently opaque that this is not a serious problem, but it should be guarded against, especially when using *m*-cresol, which has a refractive index (1.54) very near to that of most fibers.

12.2 Most polymer solutions are saturated at low concentrations, and it is essential that only small specimens and fresh solvent be used.

12.3 Not all nylon fibers are dissolved by dilute formic acid. At least 98 % pure formic acid is necessary for reliable identification.

13. Reagents

13.1 *Acetic Acid*, glacial (CH_3COOH).

13.2 *Acetone* (CH_3COCH_3).

13.3 *Ammonium Thiocyanate Solution* (NH_4SCN) (70 %).

13.4 *Chloroform* ($CHCl_3$).

13.5 *m-Cresol* ($CH_3C_6H_4OH$).

13.6 *Cyclohexane* (C_6H_{12}).

13.7 *nn'-Dimethylformamide* ($HCON(CH_3)_2$) (DMF).

13.8 *Formic Acid* ($HCOOH$) (98 %).

13.9 *Hydrochloric Acid* (HCL). Mix 1 + 1 by volume with distilled water.

13.10 *Lead Acetate Solution* ($Pb(CH_3COO)_2$) (2.0 %).

13.11 *Methylene Chloride* (CH_2Cl_2).

13.12 *Sodium Hydroxide Solution* (NaOH) (5.0 %).

13.13 *Sulfuric Acid Solution* (H_2SO_4) (75 %).

13.14 *Trichloroethylene* ($CHCl:CCl_2$).

14. Procedure

14.1 Place several of the unknown fibers in the solvent at the indicated temperature. Wait for the specified period and check to see whether the fibers have been dissolved. The fibers should be watched during the test to determine whether there might be a mixture of fiber types, some of which are soluble and some insoluble.

NOTE 3—**Precaution:** In addition to other precautions, do not mix reagents or add reagents to a fiber solution except as specifically indicated in the scheme in Fig. 1. These procedures should be carried out in a hood and the laboratory adequately ventilated.

NOTE 4—With the exception of the test to distinguish silk and wool and the melting of Saran, all of the decision points of the scheme (Fig. 1) are based on whether or not the solid fiber is dissolved by the reagent. Accordingly, if the fiber is dissolved at any point in the analysis, perform subsequent tests on a fresh specimen. Insoluble specimens may be removed from the solvent, rinsed in distilled water, and used for subsequent analyses, if required by limited sample size.

15. Report

15.1 State that the specimens were identified as directed in Test Methods D 276, Solubility Procedure. Describe the material(s) or product(s) sampled and the method of sampling used.

15.2 Report the following information:

15.2.1 Generic class of the fiber specimen, and

15.2.2 Which procedures were used to confirm the tentative identification of the specimen, and the numerical values obtained in physical property tests.

16. Precision and Bias

16.1 No information is presented about either the precision or bias of Test Methods D 276 for measuring solubility relationships since the test result is nonquantitative.

FIBER IDENTIFICATION BY INFRARED SPECTROSCOPY

17. Scope

17.1 This test method covers identification of fibers by interpretation of an absorption spectrum from infrared spectrophotometric analysis of the homogenous specimen obtained by one of three techniques: potassium bromide (KBr) disk, film, or internal reflection spectroscopy.

NOTE 5—The internal reflection spectroscopy technique is more difficult to use satisfactorily than the KBr disk or film techniques and it is not recommended for use except by an operator experienced in the technique.

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

18. Apparatus, Reagents, and Materials

18.1 *Infrared Spectrophotometer*, FTIR or double-beam instrument, having a wavelength range from 2.5 to 15.0 :m.

18.2 *KBr Disk Technique*:

18.2.1 *Hydraulic Press*, with a range from 0 to 34.5 MPa (0 to 5000 psi).

18.2.2 *Ball Mill, Vibratory*, 1-g capacity.

18.2.3 *Die*, for pressing potassium bromide (KBr) disks as recommended by the instrument manufacturer.

18.2.4 *Potassium Bromide* (KBr), spectro-grade.

18.3 *Film Technique*:

18.3.1 *Rock Salt Plate* for solution-cast films.

18.3.2 *TFE-Fluorocarbon Sheets and Heated Platens* for melt-cast films.

18.3.3 *Acetone* (CH_3COCH_3).

18.3.4 *Butyrolacetone* ($C_4H_6O_2$).

18.3.5 *Methylene Chloride* (CH_2Cl_2).

19. Preparation of Specimens

19.1 *Preparation of KBr Disk*—Cut a homogeneous fiber specimen (1 to 3 mg) into pieces less than 1 mm long with scissors. Mix with about 200 mg of powdered KBr and grind in the ball mill for 1 to 2 min. Transfer the ground mixture to a KBr-disk die and press at approximately 14 MPa (2000 psi) pressure for 2 to 5 min.

19.2 *Preparation of Films*—Consult Table 2 for fibers that melt readily or are soluble in suitable solvents. Cast films from solution on a rock salt plate using a glass stirring rod and permit the solvent to evaporate. Cast melt films by pressing fibers between TFE-fluorocarbon sheets between heated platens on a laboratory hydraulic press. As a general guide, the films should be cast thin enough to be nearly transparent.

20. Procedure

20.1 Verify the calibration of the spectrophotometer according to the manufacturer's instructions. Place the specimen in the sample beam of the instrument and record the spectrum while scanning from 2.5 to 15 :m.

21. Interpretation of Results

21.1 The infrared spectrum from a fiber specimen will have several absorption bands for peaks. For identification, the positions of the peaks according to wavelength or wave number should match those of a known reference spectrum (Figs. 3-26). As an aid in matching spectra, consult the decision chart (Fig. 2) and follow the scheme through to one of the end points, according to the presence or absence of the indicated bands.

21.2 It is desirable to confirm the identification by other methods, such as those described in Sections 14-35. Refer to the tabulation of fiber physical properties in Table 1 for this purpose.

21.3 If the infrared spectrum indicates either a native cellulose or a wool fiber, it is desirable to resort to microscopic examination (see AATCC Test Method 20) to determine species.

22. Report

22.1 State that the specimens were identified as directed in Test Methods D 276, Infrared Spectroscopy Procedure. Describe the material(s) or product(s) sampled and the method of sampling used.

22.2 Report the following information:

22.2.1 The generic class of the fiber specimen,

22.2.2 The identification and location of the standard reference spectrum to which the spectrum of the unknown fiber was compared; and the location (wavelength), relative strength (strong, medium, weak), and character (sharp, moderate, broad) of any absorption peaks which are not common to the two spectra, and

22.2.3 Which procedures were used to confirm the tentative identification of the specimen, and the numerical values obtained in physical property tests.

23. Precision and Bias

23.1 No information is presented about either the precision or bias of Test Methods D 276 for infrared identification technique since the test result is nonquantitative.

PHYSICAL PROPERTIES TO CONFIRM IDENTIFICATION

FIBER DENSITY

24. Scope

24.1 Fiber density is measured by density-gradient column method. Determine density by the density-gradient column, pycnometer, and a technique based on Archimedes' principle as directed in the AATCC Test Method 20.

25. Pycnometer Method

25.1 Determine density by the pycnometer method as directed in Method B of Test Methods D 792.

TABLE 2 Suggested Method of Obtaining Film Satisfactory for IR Spectroscopy

NOTE 1—The listed techniques are known to be reliable. This listing is not meant to exclude other fiber types or to restrict the choice of alternative solvents.

| Fiber Generic Type	Cast Film From
Acetate	acetone solution
Modacrylic	acetone or butyrolactone solution
Nylon	melt
Nytril	acetone or butyrolactone solution
Olefin	melt
Polyester	melt
Saran	melt
Triacetate	methylene chloride solution
Vinal	methylene chloride solution
Vinyon	methylene chloride solution

FIG. 3 Infrared Absorption Spectrum of a Typical Acetate Fiber
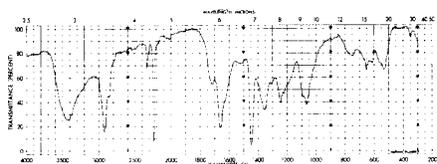
Acrilan®, Creslan®, Orlon®, Zefran®
The spectra of these fibers may differ somewhat due to small compositional differences.

FIG. 4 Infrared Absorption Spectrum of a Typical Acrylic Fiber
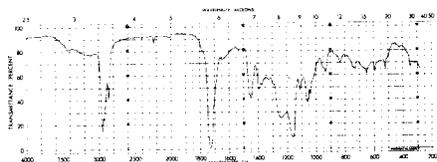
Anim/8®

FIG. 5 Infrared Absorption Spectrum of a Typical Anidex Fiber

26. Method Based on Archimedes' Principle

26.1 *Scope*—Density measurements based on Archimedes' principle are described in Method A of Test Methods D 792. The procedure described here is applicable only to fibers that can be obtained in the form of continuous filaments.

26.2 *Summary of Method*—A small specimen is weighed in air and when immersed in a liquid of lower density than the fiber (and which thoroughly wets the material). By Archimedes' principle the difference in weight (the buoyant force) is equal to the weight of the liquid displaced; that is, the product of the volume of the fiber specimen and the density of the liquid. Several measurements of immersed weight are made over a period of time, until the weight becomes constant or changes very slowly. The immersed weight is plotted as a function of time, and the apparent density obtained by extrapolating to zero time.

26.3 *Apparatus*:

26.3.1 *Analytical Balance*, with a capacity of at least 1 g and sensitivity of ±0.1 mg.

26.3.2 *Wire Hook*, fashioned from a 150-mm (6-in) length of No. 34 Nichrome wire.

NOTE 6—The wire size is critical because of surface phenomena; coarser wire has greater surface area and finer wire flexes, causing a greater effective surface.

26.3.3 *Platform Bridge*, to place over the balance pan.

26.3.4 *Stopwatch*, or other suitable timer.

26.3.5 *Beaker*, 250-mL capacity.

26.4 *Reagents and Materials*:

26.4.1 *Refrigerant-TF*, for removing fiber lubricants and finishes.

26.4.2 *Displacement Liquids* (*n*-heptane is recommended for most fibers except olefins, for which methanol is the liquid of choice). The specific gravity of each batch must be determined by a pycnometric technique such as Test Method D 941 or Test Method D 1217.

26.4.2.1 *n-Heptane*, at least 99 % mol purity. Approximate density 684 kg/m³ (0.684 g/cm³).

26.4.2.2 *Methanol*—Approximate density 791 kg/m³ (0.791 g/cm³).

26.5 *Preparation of Specimens*:

26.5.1 Make a small skein (about 25 mm in diameter) of 0.5 to 1.0 g of fiber, being careful not to stretch the fiber. Securely tie the ends of the skein and cut off the free ends.

26.5.2 Thoroughly rinse the specimen in refrigerant-TF to remove lubricant, and allow to dry in room air.

26.5.3 Condition the specimens for testing in the standard atmosphere for testing textiles, as directed in Practice D 1776.

26.6 *Procedure*:

26.6.1 Determine the weight of the conditioned specimen, *D*, to the nearest 0.1 mg.

26.6.2 Place the bridge over the balance pan and place the beaker of displacing fluid on the bridge. Hang the wire hook with its flee end immersed in 200 mL of displacing liquid (Fig. 26) and weigh. Record the weight of the wire hook, *H*, to the nearest 0.1 mg.

26.6.3 Remove the hook and beaker from the balance and place the specimen on the hook. Simultaneously start the time and immerse the specimen in the liquid. Using a stirring rod, vigorously agitate the sample to remove occluded air. Frequently, several minutes of vigorous effort are required to remove all of the air.

26.6.4 Replace the beaker on the bridge and hang the wire hook as shown in Fig. 26. Take care that the specimen is immersed completely and that it does not touch the sides of the beaker.

26.6.5 Record the time and immersed weight of the sample and hook, *W + H*, to the nearest 0.1 mg at approximately 1-min intervals until 10 min total immersed time has elapsed. Therefore record the time and weight at about 2-min intervals until at least 20 min total time has elapsed.

26.7 *Calculations*:

26.7.1 For each recorded time, calculate the apparent density of the fiber using Eq 1:

$$P_f = \cfrac{D}{D + H - (W + H)} \cdot P_l \qquad (1)$$

where:
P_f = apparent fiber density, kg/m3,
P_l = density of the liquid, kg/m3,
D = conditioned weight of the specimen, g,
H = immersed weight of the wire hook, g, and
$(W + H)$ = immersed weight of the hook and specimen, g.

26.7.2 Make a plot of apparent density versus immersion time. Extrapolate the straight-line portion back to zero time, if possible. Fiber density is the extrapolated apparent density at zero time.

26.8 *Report*:

26.8.1 State that the specimens were tested as directed in Test Methods D 276, Method Based on Archimedes' Principle. Describe the material(s) or product(s) sampled and the method of sampling used.

26.8.2 Report the following information:

26.8.2.1 Fiber density, to three significant figures, and

26.8.2.2 The displacing liquid used, its density to four significant figures, and the method used to determine its density.
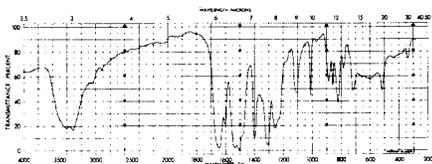
(a) Kevlar®
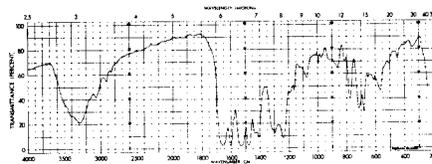
(b) Nomex®

**FIG. 6 Infrared Absorption Spectra of Typical Aramid Fibers**
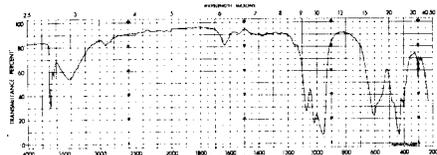
FIG. 7 Infrared Absorption Spectrum of a Typical Asbestos Fiber

Dynel®, Kanecaron®, Monsanto SEF®, Verel®

The spectra of these fibers may differ somewhat due to compositional differences.

FIG. 11 Infrared Absorption Spectrum of a Typical Modacrylic Fiber
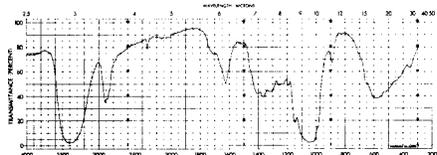
Cotton, Flax, Jute, Ramie

FIG. 8 Infrared Absorption Spectrum of a Typical Native Cellulosic Fiber

Kynol®

FIG. 12 Infrared Absorption Spectrum of a Typical Novoloid Fiber
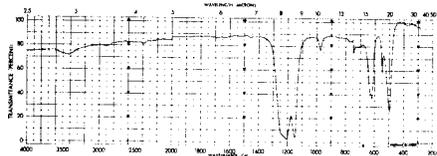
Teflon®

FIG. 9 Infrared Absorption Spectrum of a Typical Fluorocarbon Fiber
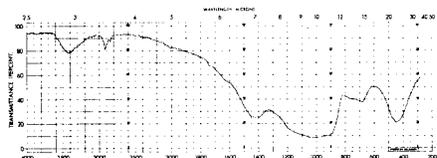
FIG. 10 Infrared Absorption Spectrum of a Typical Glass Fiber

27. Precision and Bias

27.1 *Precision*—The precision of this test method for determining fiber density is not known. An interlaboratory study has been organized. The analysis of data will be completed and the precision statement will be included in the test method by the end of the year 2000. Single-determination density estimates by one operator for one sample of fiber from measurements in two different displacing liquids (one a mild solvent for the fiber, the other a mild swelling agent) agreed to within 0.0004 mg/mm^3, but differed from a third estimate in a liquid known to have poor wetting characteristics for the fiber by 0.002 mg/mm^3. It is believed that the components of variance of this method are such that fiber densities are reproducible to within ±0.01 mg/mm^3 by all operators and laboratories, but there are

no data to support this assumption.

27.2 *Bias*—No justifiable statement can be made on the bias of this method for determining fiber density, since the true value cannot be determined by an accepted referee method.

FIBER MELTING POINT

28. Scope

28.1 This test method allows determining the temperature at which the material begins to lose its shape or form and becomes molten or liquefies. Allowing material to reach its melting point results in permanent fiber change.

29. Apparatus, Reagents, and Materials

29.1 *Melting Point Apparatus*:

29.1.1 *Fisher-Johns Melting Point Apparatus*[10], or

29.1.2 *Differential Scanning Calorimeter* (DSC).

29.2 *Materials Needed for Fisher-Johns Apparatus*:

29.2.1 *18-mm Micro Cover Glasses*, round No. 2.

29.2.2 *Test Reagent Set*[11]—A set of test reagents, precision 0.5°C, or reagent grade chemicals for checking the calibrated thermometers.

29.3 *Materials Needed for DSC*:

29.3.1 *Aluminum Pans and Lids.*

---

[10] The Fisher-Johns Melting Point Apparatus, available from Fisher Scientific Corp., 717 Forbes St., Pittsburgh, PA 15219, has been found satisfactory for this test method.

[11] These substances may be obtained from Eastman Kodak Co., Rochester, NY.

(a) nylon 6

(b) nylon 6,6

(c) nylon 11

(d) Qiana®
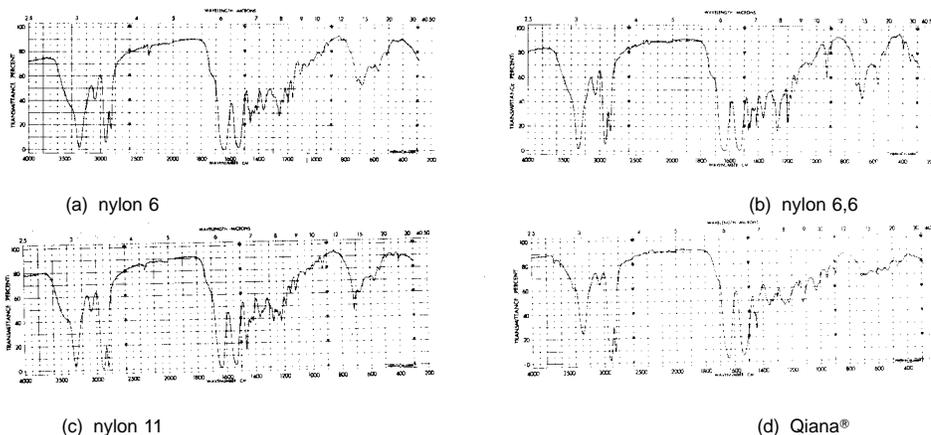
FIG. 13 Infrared Absorption Spectra of Typical Nylon Fibers
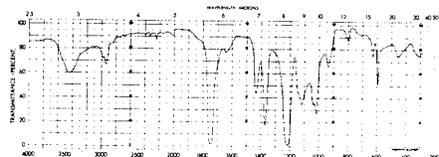

FIG. 14 Infrared Absorption Spectrum of a Typical Nytril Fiber

29.3.2 *Indium, Zinc, and Tin Standards.*

29.3.3 *Sample Crimper.*

30. Calibration

30.1 *Calibration of Fisher-Johns Apparatus*—The calibration of each thermometer should be verified periodically as recommended by the hot stage manufacturer or with reagent grade chemicals, using the same rate of temperature rise chosen for the tester. Recommended chemicals include l-menthol melting at 42 to 43°C, hydroxyquinoline at 75 to 76°C, acetanilide at 113 to 114°C, succinic acid at 189 to 190°C, and phenolphthalein at 261 to 262°C (**10**).

30.2 *Calibration of DSC*—The temperature and heat calibration should be performed using the indium, zinc, and tin standards for the working temperature range.

31. Procedure

31.1 Determine the approximate melting point of the fiber by reference to Table 2 or by a trial run.

31.2 *Procedure for Fisher-Johns Apparatus*:

31.2.1 Place the specimen between two cover glasses, lay the entire unit on the hot stage, and press gently but firmly into place. Rapidly heat the stage to about 15°C below the expected melting point, and then lower the heating rate to about 3 or 4°C/min.

31.2.2 Press lightly on the upper cover glass with a spatula or pick needle and observe the specimen with the aid of the lens.

31.2.3 Record the temperature to within 1°C when the specimen is seen to melt rapidly and spread between the glasses or when the final traces of solid fiber structure disappear in cases where the fiber melts slowly over a wide range of temperature. If the melting range is greater than 1°C, report the melting range.

31.2.4 Cool the stage to about 50°C below the observed melting point and repeat the measurement on a new specimen.

31.3 *Procedure for DSC*:

31.3.1 *Sample Preparation*—Cut the samples into very small pieces with scissors and put approximately 5 to 7 mg specimens into the aluminum pan. Make a small hole on the lid with a needle and close the pan with the help of a sample crimper. Make another sample, which will be used as the "reference" sample by closing an empty pan with lid.

31.3.2 Put the sample and the reference in the designated heating blocks (in accordance with the instrument manufacturer's manual) inside the heater chamber. Close the chamber, and start heating at the rate of 10°C/min to approximately 50°C above the melting point of the fiber being tested in accordance with Table 2.

31.3.3 Record the temperature. Cool the chamber, and repeat the measurement on a new specimen.

32. Report

32.1 State that the specimens were tested as directed in Test Methods D 276. Describe the material(s) or product(s) sampled and the method of sampling used. Report the melting point as the average of the two determinations to the nearest °C.

33. Precision and Bias

33.1 *Precision*—No data are available on which to judge the precision of this test method for determining fiber melting point. An interlaboratory study has been organized. The analysis of data will be completed and the precision statement will be included in the test method by the end of the year 2000. For the purpose of fiber identification, results should not be considered suspect unless the duplicate observations of one operator differ by more than 2°C.

33.2 *Bias*—No justifiable statement can be made on the bias of this method for determining fiber melting point, since the true melting point cannot be established by an accepted referee method.

REFRACTIVE INDICES AND BIREFRINGENCE

34. Scope

34.1 Refractive indices and birefringence are measured by

TABLE 15
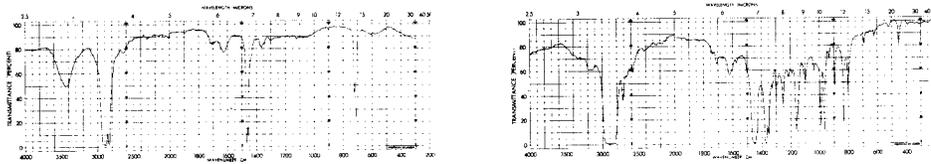
(a) polyethylene

(b) polypropylene

**FIG. 15 Infrared Absorption Spectra of Typical Olefin Fibers**
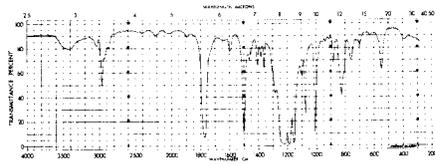
FIG. 16 Infrared Absorption Spectrum of a Typical Polycarbonate Fiber

Difference of Refractive Indices test method. Determine the refractive indices for plane-polarized light parallel to and perpendicular to the fiber length within 0.001, in accordance with AATCC Test Method 20.

34.2 Calculate the birefringence using Eq:

$$\Delta n = \epsilon - \omega \qquad (2)$$

where:
Δn = birefringence,
ϵ = refractive index parallel to fiber axis, and
ω = refractive index perpendicular to fiber axis.

35. Precision and Bias

35.1 *Precision*—The components of variance of this test method for determining fiber birefringence have not been determined. An interlaboratory study has been organized. The analysis of data will be completed and the precision statement will be included in the test method by the end of the year 2000. The coefficient of variation of measured values for single fibers from one sample is about 10 %, under ideal conditions. The method is often very difficult, and many of the phenomena observed when viewing fibers with polarized light are subject to misinterpretation. The extinction bands are sometimes very broad, so that the compensating retardation corresponding to maximum extinction cannot be determined precisely. Significant differences between values reported by different observers are known to occur.

35.2 *Bias*—Subject to the comments in 35.1, the compensator test method for determining fiber birefringence is not biased relative to the difference of refractive indices, which is a reference method.
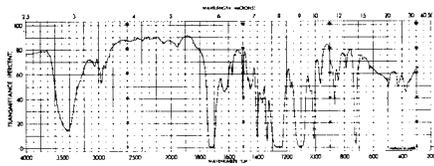
(a) 2 GT
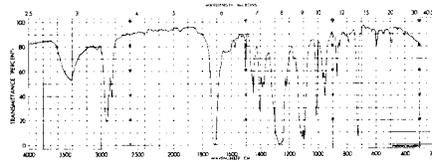
(b) CHDM-T
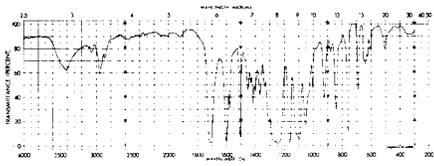
(c) oxybenzoate
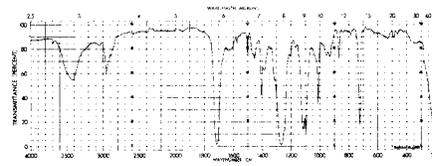
(d) 4 GT

(a) 2 GT (ethylene glycol) type: Avlin®, Dacron®, Encron®, Fortrel®, Kodel®, Spectran®, Trevira®,
(b) CHDM-T (1,4)-cyclohexanedimethanol) type: Kodel®
(c) p-ethylene oxybenzoate type: A-Tell®
(d) 4 GT (1,4-butanediol) type: Fortrel®

FIG. 17 Infrared Absorption Spectra of Typical Polyester Fibers
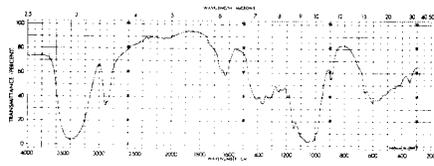
viscose, cuprammonium (Bemberg®)

FIG. 18 Infrared Absorption Spectrum of a Typical Rayon Fiber
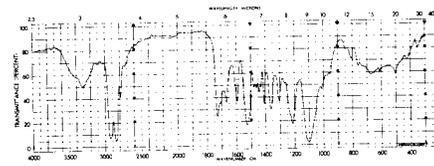
Lycra®

FIG. 21 Infrared Absorption Spectrum of a Typical Spandex Fiber
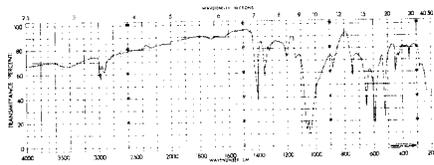
FIG. 19 Infrared Absorption Spectrum of a Typical Saran Fiber
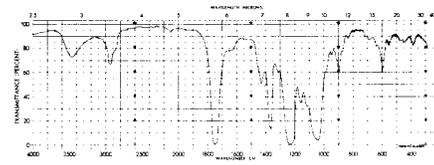
Arnel®

FIG. 22 Infrared Absorption Spectrum of a Typical Triacetate Fiber
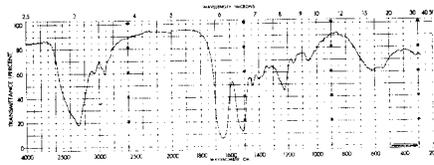
FIG. 20 Infrared Absorption Spectrum of a Typical Silk Fiber
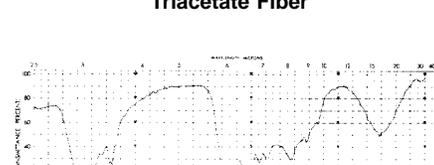
FIG. 23 Infrared Absorption Spectrum of a Typical Vinal Fiber
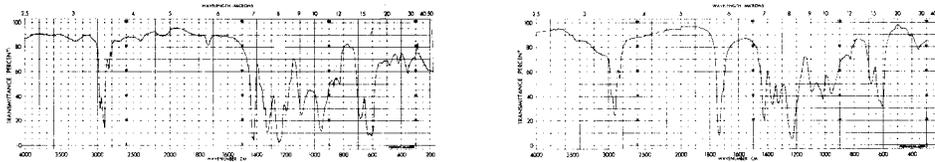
(a) Rhovyl®

(b) Vinyon

FIG. 24 Infrared Absorption Spectra of Typical Vinyon Fibers
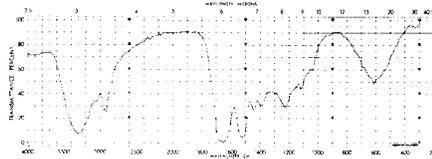
FIG. 25 Infrared Absorption Spectrum of a Typical Wool Fiber
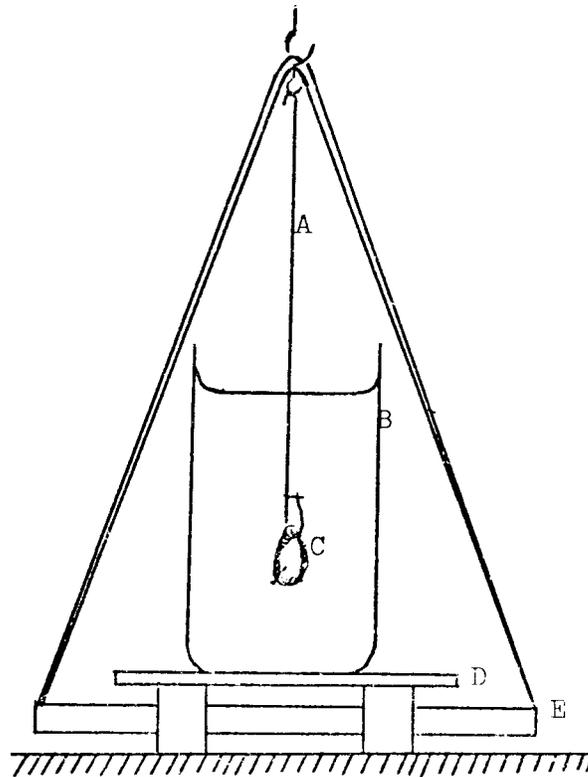
A) No. 34 Nichrome wire hook.   D) Platform bridge.
B) 250-ml beaker.   E) Balance pan.
C) Sample skein.

**FIG. 26 Arrangement for Fiber Density Measurement using
Archimedes' Method**

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(**13**) Hartshorne, N. H., and Stuart, A., *Crystals and the Polarizing Microscope*, Edward Arnold, Ltd., London, England, 1960.