Designation: D276 – 12

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

This standard is issued under the fixed designation D276; 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.

This standard has been approved for use by agencies of the U.S. Department of Defense.

INTRODUCTION

Methods D276 – 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and that the techniques required to identify these fibers were lacking in the text, so that the text had become out of date.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 D276 – 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 20–2011"[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 D123, 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	Kevlar®, Nomex®, Technora®, TeijinConex®, 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®

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

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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.

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®, Cordenka®
Saran	Enjay®, Saran®
Spandex	Glospun®, Lycra®, Numa®, Unel®
Triacetate	Arnel®
Vinyon	Avisco®, Clevyl®, Rhovyl®, Thermovyl®, Volpex®

NOTE 1—The list of trademarks in 1.2 contains only examples and 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 Refs (**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 These test methods include 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:*[4]

D123 Terminology Relating to Textiles
D629 Test Methods for Quantitative Analysis of Textiles
D792 Test Methods for Density and Specific Gravity (Relative Density) of Plastics by Displacement
D941 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Lipkin Bicapillary Pycnometer (Withdrawn 1993)[5]
D1217 Test Method for Density and Relative Density (Specific Gravity) of Liquids by Bingham Pycnometer
D1776 Practice for Conditioning and Testing Textiles
E131 Terminology Relating to Molecular Spectroscopy
E175 Terminology of Microscopy

2.2 *AATCC Method:*[2]

Test Method 20 Fiber Analysis: Qualitative
Test Method 20A Fiber Analysis: Quantitative

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

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

[4] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[5] The last approved version of this historical standard is referenced on www.astm.org.

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.

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 D123 for textiles, Terminology E131 for terms relating to infrared spectroscopy, and Terminology E175 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).

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.

5.5 Qualitative and quantitative fiber identification is actively pursued by Committee RA24 (Fiber Identification) of AATCC and presented in AATCC Test Method 20 and Test Method 20A. Since precision and bias development is also part of the AATCC test methods, both AATCC and ASTM D13 have agreed that new development will take place in RA24. However, because there is valuable information still present in the ASTM standards, Test Methods D276 and D629 will be maintained as active standards by ASTM.

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

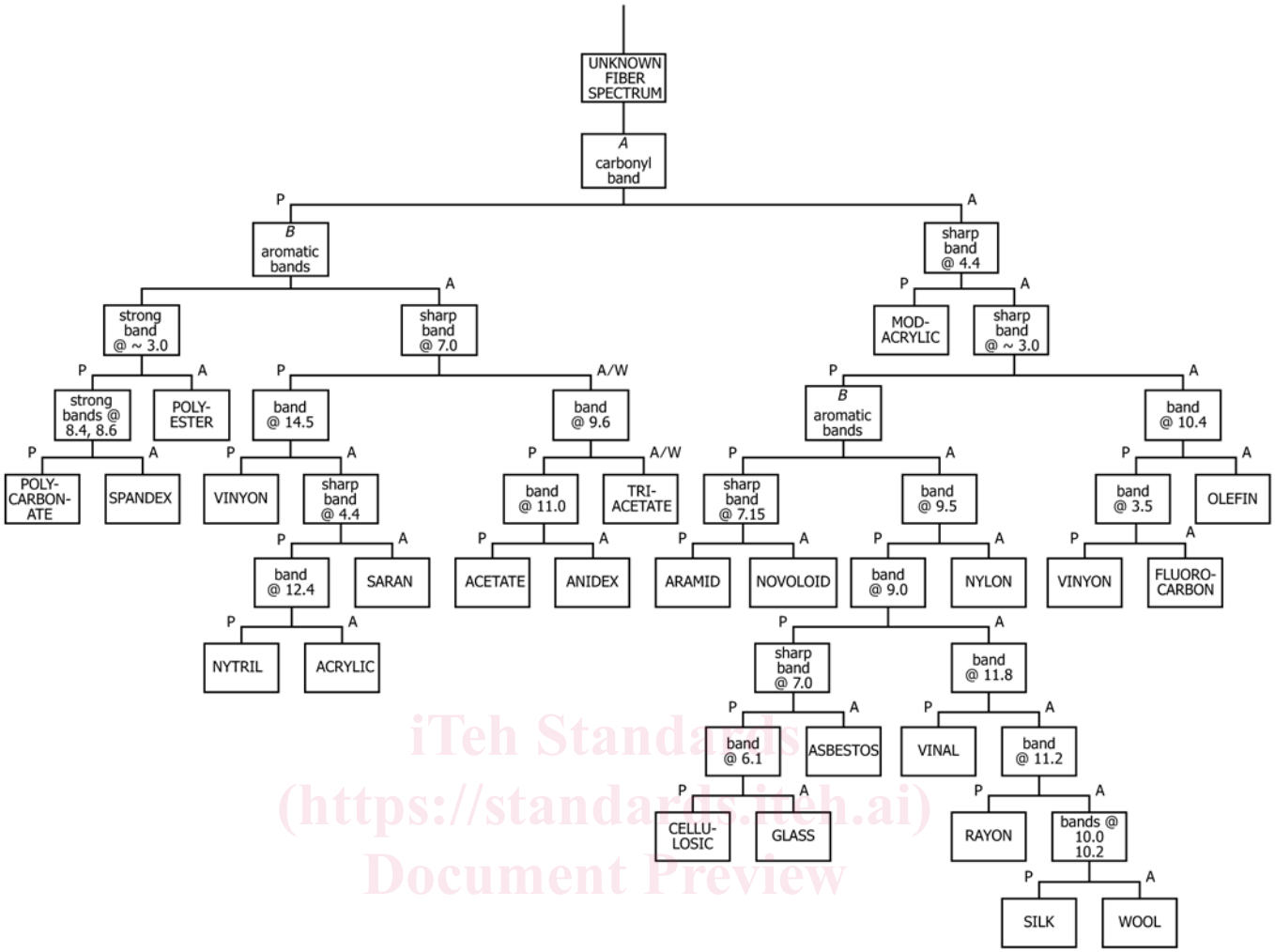
6.1 The quantity of material per specimen and the number of specimens required differ according to the types of analyses that are to be conducted. It is possible to make an identification using a sample of less than 10 mg of each type of fiber present.

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 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 D629) 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.



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

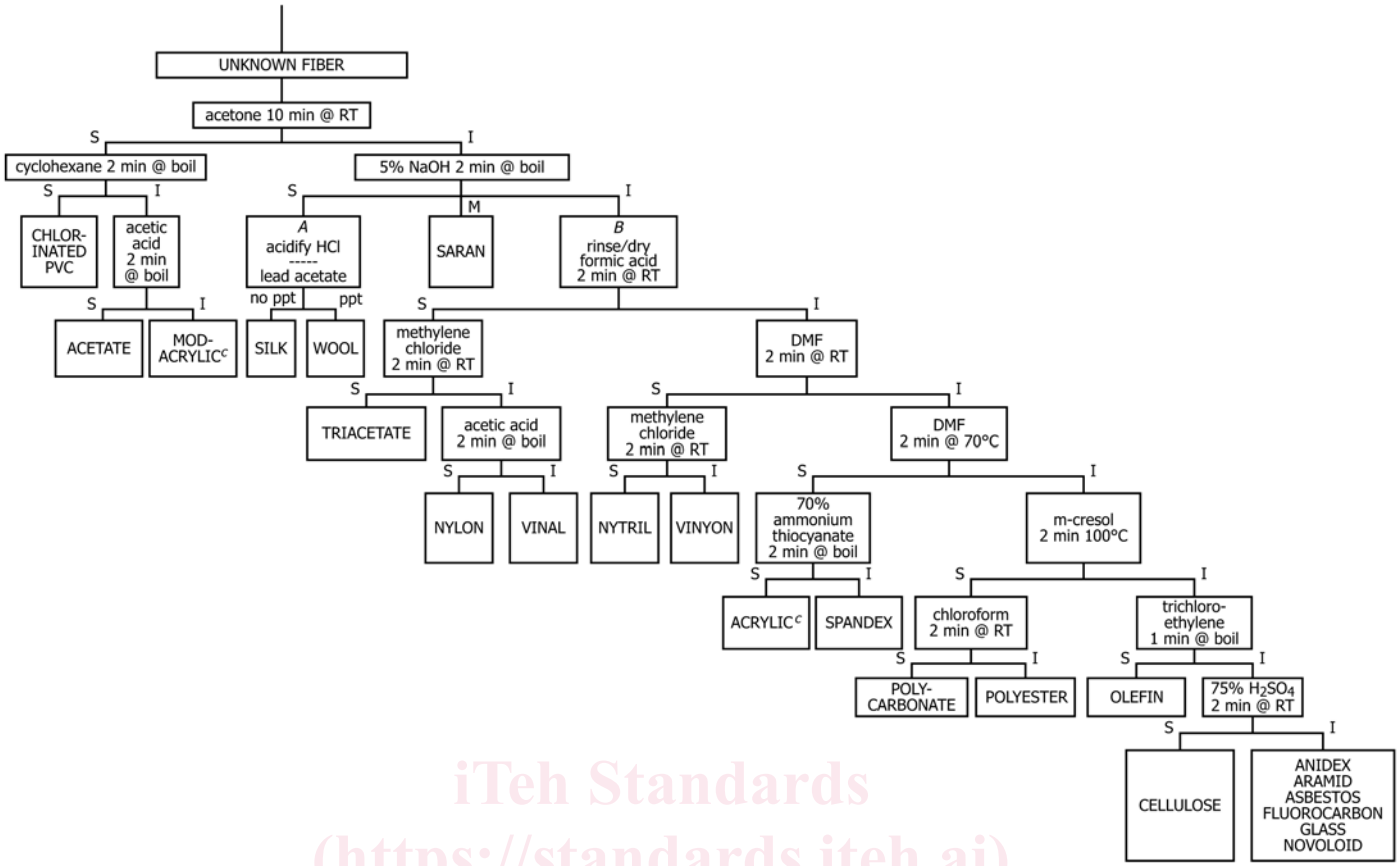
6.3.3 Warp and filling yarns may be of different types and not every yarn in the warp (or filling) is necessarily made from the same type of fiber.

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



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

specifications are available.[6] 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 D276 for microscopical examination since the test result is nonquantitative.

[6] *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.

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.

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.

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