



Standard Test Methods for Quantitative Analysis of Textiles¹

This standard is issued under the fixed designation D629; 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 D629-59 T, Quantitative Analysis of Textiles, were discontinued in 1969 because the responsible subcommittee failed to recommend their adoption as a standard after several years of 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 method was published, and that the techniques required for their identification were lacking in the text, allowing it to become out of date. The procedures included in the text, however, are believed to be reliable for the fiber types named and the techniques described are currently being used in the trade and are referenced by other standards sponsored by Committee D-13 on Textiles. Reinstatement as a standard using the previously assigned number was requested since the listed procedures were reliable and the text 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 D629-72. Editorial changes have again been made throughout the text, and statements on precision and bias and suitability for acceptance testing have been added.

The text of Methods D629-59 T was published by the American Association of Textile Chemists and Colorists in that society's Technical Manual as "Test Method 20A-1959" issued in the years 1959 through 1974. The AATCC Method was revised completely in 1975 and since published as "Test Method 20A-1975."

1. Scope

1.1 These test methods cover procedures for the determination of the fiber blend composition of mixtures of the fibers listed in 1.2. Procedures for quantitative estimation of the amount of moisture and certain nonfibrous materials in textiles are also described, for use in the analysis of mixtures, but these are not the primary methods for the determination of moisture content for commercial weights.

1.2 These test methods cover procedures for the following fiber types:

1.2.1 Natural Fibers:

1.2.1.1 Cellulose-Base Fibers:

Cotton

Hemp

Flax

Ramie

1.2.1.2 Protein-Base Fibers:

Animal hairs (other than wool)

Silk, cultivated

Silk, Tussah

Wool

1.2.2 Man-Made Fibers:

1.2.2.1 Cellulose-Base Fibers:

Acetate (secondary)

Rayon, viscose or cuprammonium

Triacetate

1.2.2.2 Synthetic-Base Fibers:

Acrylic

Aramid

Modacrylic

Nylon 6, Nylon 6-6, others

Olefin

Polyester

Spandex

1.3 These test methods include the following sections and tables:

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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1.4 The analytical procedures described in the test methods are applicable to the fibers listed in 1.2. The test methods are not satisfactory for the separation of mixtures containing fibers that fall within the same generic class but differ somewhat, either physically or chemically, from each other. These test methods are not satisfactory for the determination of bicomponent fibers.

NOTE 1—For other methods of analysis covering specific determinations, refer to: Test Methods D461, Test Method D584, Methods D885, Test Method D1113, Test Method D1334, and Test Method D2130. Methods for moisture are covered in Methods D885, Test Method D1576, Test Method D2462, Test Method D2495 and Test Methods D2654. For the determination of commercial weight, refer to Test Method D2494.

1.5 *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.*

2. Referenced Documents

2.1 ASTM Standards:²

D123 Terminology Relating to Textiles

D276 Test Methods for Identification of Fibers in Textiles

² 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.

D461 Test Methods for Felt (Withdrawn 2003)³

D584 Test Method for Wool Content of Raw Wool—Laboratory Scale

D885 Test Methods for Tire Cords, Tire Cord Fabrics, and Industrial Filament Yarns Made from Manufactured Organic-Base Fibers

D1113 Test Method for Vegetable Matter and Other Alkali-Insoluble Impurities in Scoured Wool

D1193 Specification for Reagent Water

D1334 Test Method for Wool Content of Raw Wool—Commercial Scale

D1576 Test Method for Moisture in Wool by Oven-Drying

D1909 Standard Tables of Commercial Moisture Regains and Commercial Allowances for Textile Fibers

D2130 Test Method for Diameter of Wool and Other Animal Fibers by Microprojection

D2462 Test Method for Moisture in Wool by Distillation With Toluene

D2494 Test Method for Commercial Mass of a Shipment of Yarn or Manufactured Staple Fiber or Tow

D2495 Test Method for Moisture in Cotton by Oven-Drying

D2654 Test Method for Moisture in Textiles (Withdrawn 1998)³

D4920 Terminology Relating to Conditioning, Chemical, and Thermal Properties

2.2 AATCC Method:

20 A Test Method for Fiber Analysis: Quantitative⁴

3. Terminology

3.1 For definitions of textile terms used specifically in these test methods, refer to Terminology D4920.

3.2 For definitions of other generic textile terms used in these test methods, refer to Terminology D123.

4. Summary of Test Methods

4.1 Summaries of the specific methods used for different tests are given in the appropriate sections.

4.2 The methods for fiber analysis are grouped under three headings, as follows: Mechanical Separation or Dissection, Chemical Test Methods, and Microscopical Analysis.

NOTE 2—It is assumed that the analyst knows from qualitative tests (as directed in Test Methods D276) what fibers are present and, therefore, which method of analysis is applicable. The choice of method will depend upon the nature of the material to be analyzed and, in some cases, on the accuracy required.

5. Significance and Use

5.1 Qualitative and quantitative fiber identification is actively pursued by committee RA24 (Fiber Identification) of AATCC and presented in AATCC Test Methods 20 and 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.

³ The last approved version of this historical standard is referenced on www.astm.org.

⁴ AATCC Technical Manual, available from the American Association of Textile Chemists and Colorists, P.O. Box 12215, Triangle Park, NC 27709, www.aatcc.org.

However, because there is valuable information still present in the ASTM standards, D13.51 has agreed Test Methods D276 and D629 will be maintained as active standards by ASTM.

5.2 Test Methods D629 for the determination of quantitative analysis of textiles may be used for acceptance testing of commercial shipments but caution is advised since information on between-laboratory precision is lacking. Comparative tests as directed in 5.2.1 or in Standard Tables D1909 may be advisable.

5.2.1 In case of a dispute arising from differences in reported test results using Test Methods D629 for acceptance testing of commercial shipments, the purchaser and the supplier should conduct comparative tests to determine if there is a statistical bias between their laboratories. Competent statistical assistance is recommended for the investigation of bias. As a minimum, the two parties should take a group of test specimens which are as homogeneous as possible and which are from a lot of material of the type in question. The test specimen should then be randomly assigned in equal numbers to each laboratory for testing. The average results from the two laboratories should be compared using Student's *t*-test and an acceptable probability level chosen by the two parties before the testing began. If a bias is found, either its cause must be found and corrected or the purchaser and supplier must agree to interpret future test results in the light of the known bias.

5.3 The effects of the various reagents used in the chemical methods on the residual fibers in a blend depend upon the

history of the fibers and, unless otherwise stated, are generally too small or too uncertain to warrant the application of correction factors.

5.4 Fiber composition is generally expressed either on the oven-dry mass of the original sample or the oven-dry mass of the clean fiber after the removal of nonfibrous materials. If nonfibrous materials are not first removed from the textile before the fiber analysis is carried out, or if the treatments described in Section 8 are incapable of removing them, any such materials present will increase the percentage of the fiber constituent with which they are removed during the analysis, assuming they are soluble in the solvent used.

5.5 The analytical methods are intended primarily for the separation of binary mixtures of fibers. These procedures may also be used for the analysis of mixtures containing more than two types of fibers by selecting the best combination of methods to use (Table 1). Since a sequence of solvents on a given fiber may produce different results than the expected results from a single solvent, it is advisable to determine the results of such sequential effects when testing multiple fiber blends. It is sometimes more convenient to separate mechanically the yarns in a textile which are of similar types, and then use the appropriate chemical method to analyze each of the components. Table 2 shows the solubilities of the various fibers in different chemical reagents.

TABLE 1 Chemical Methods for Analysis of Fiber Mixtures^A

	Wool	Spandex	Silk	Rayon	Polyester	Olefin	Nylon	Mod-acrylic	Cellulosic, Natural	Aramid	Acrylic	Triacetate
Acetate	1		1	1	1	1	1	1	1		1	1
Triacetate	3		(5)	(7 ^B)	9	3 ⁹	(5)	9	(5)		3	
Acrylic	(6)		(6)	(7 ⁵)	10	(8)	10	10	(5 ⁷)			
Aramid	(6)											
Cellulosic, Natural	(6) ⁵	(10)	(6)	(4)	5	5	(3)	(2)				
Modacrylic	2	2	2	2	2	2	2					
Nylon	3	(10)	(6)	3	9	(8)						
Olefin	(6)		(6)	(7 ⁵)	8							
Polyester	(6)	(10)	(6)	(7)								
Rayon	5		(6)									
Silk		6										
Spandex	(6)											

^AKey to Methods and Reagents:

Method No. 1—80 % acetone(cold)

Method No. 2— *N*-Butyrolactone

Method No. 3—90 % formic acid

Method No. 4—59.5 % sulfuric acid

Method No. 5—70 % sulfuric acid

Method No. 6—Sodium hypochlorite solution

Method No. 7—Curpammonia solution

Method No. 8—Hot xylene

Method No. 9—90 % formic acid

Method No. 10—N,N-dimethylacetamide

^BEach analytical method is identified by a number and where possible, two methods of analysis are provided for each binary mixture of fibers. The number or numbers inside parentheses refers to the method that dissolves the fiber shown at the top of the diagram. The number or numbers outside the parentheses indicates the method that dissolves the fiber listed at the left side of the diagram. Where two methods are listed for a specific binary mixture, the non-superscript method number represents the method of choice.

TABLE 2 Solubilities of Fibers in Solvents Used in Chemical Methods^A

	Method No. (1)	(2)		(3) and (9)	(4)	(5)	(6)	(7)	(8)	(10)
	80 % Acetone	Butyrolactone		90 % Formic Acid	59.5% H ₂ SO ₄	70% H ₂ SO ₄	NaOCl Solu- tion	Curpam- monia Solution	Hot Xylene	N,N-dimethyl acetamide
		(A) RT	(B) 75°C							
Acetate	S	S	PS	S	S	S	I	I	...	...
Triacetate	I	PS	PS	S	I	I	I	I	...	S
Acrylic	I	S	S	I	I	I	I	I	I	S
Aramid										
Cellulosic, Natural	I	I	I	I	SS	S	I	S	...	I
Modacrylic	I	S	S	I	I	I	I	I	...	I
Nylon	I	I	I	S	S	S	I	I	I	I
Olefin	I	I	I	I	I	I	I	I	S	I
Polyester	I	I	I	I	I	I	I	I	I	I
Rayon	I	I	I	I	S	S	I	S	I	I
Silk	I	I	I	PS	S	S	S	...	...	S
Spandex	...	I	I	PS	I	I	I	...	...	S
Wool and Hair Fibers	I	I	I	I	I	I ^B	S	...	...	...

^AKey to Symbols:

S = Soluble

PS = Partially Soluble

SS = Slightly Soluble (a correction factor may be applied)

I = Insoluble

^BReworked wools are soluble in 70 % H₂SO₄ depending upon their previous history.

6. Purity of Reagents

6.1 Use reagent-grade chemicals 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.⁵ 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.

6.2 Unless otherwise indicated, references to water shall be understood to mean water conforming to Type I grade of Specification **D1193**.

7. Sampling

NOTE 3—It is not possible to provide specific instructions for all types of textile materials to which these analytical methods may be applicable, but a few general recommendations shall be followed.

7.1 Sampling for Acceptance Testing:

7.1.1 *Lot Sample*—As a lot sample for acceptance testing, take at random the number of shipping containers directed in an applicable material specification or other agreement between the purchaser and the supplier. Consider shipping containers to be the primary sampling units.

NOTE 4—An adequate specification or other agreement between the purchaser and the supplier requires taking into account the variability between and within shipping containers so as to provide a sampling plan with a meaningful producer's risk, consumer's risk, acceptable quality level, and limiting quality level.

⁵ *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.

7.1.2 *Laboratory Sample*—As a laboratory sample for acceptance testing, proceed as follows:

7.1.2.1 *Fiber in Bulk*—Take one composite sample from each bale or other shipping container in the lot sample with each such composite sample being taken from at least five places in the shipping container.

7.1.2.2 *Textile Strands*—Take one package at random from each shipping container in the lot sample.

7.1.2.3 *Fabric*—Take a full width swatch 1 m long or a swatch long enough to contain one pattern repeat, whichever is longer, from the end of each roll of fabric in the lot sample, after first discarding a minimum of 1 m of fabric from the very outside of the roll.

7.1.2.4 *Garments*—Take one garment at random from each shipping container in the lot sample.

7.1.2.5 *Other Textiles*—Take one laboratory sample from each shipping container in the lot sample as agreed between the purchaser and the supplier.

7.1.3 *Test Specimens*—Unless otherwise directed in the test method(s) of interest, prepare a test specimen having a mass of 1.0 to 1.5 g by placing it in a weighing bottle. Place the weighing bottle and specimen in an oven and heat for 1 h at a temperature of 105° to 110°C. Transfer the weighing bottle to a desiccator and allow it to cool to room temperature. For fabrics containing a repeating weave pattern, include all yarns in the pattern within the test specimen. Take the specimen from other units when more than one unit is being tested or analyzed.

7.2 *Sampling for Other Purposes*—In general, follow the principles specified for samples for acceptance testing. Insofar as the amount of material available and the purposes of the analysis dictate, take a lot sample, laboratory sample, and test specimens in such a manner that all fibers present have a proportional probability of being sampled.

8. Nonfibrous Materials

8.1 *Scope*—These procedures are intended for the removal of the nonfibrous natural constituents of the fiber and substances added by the manufacturer. Starch, China-clay, soaps, some waxes, some nondrying oils, certain resins, and the usual natural constituents are in this category and are removed by the procedures described. However, general directions for the removal of all possible substances which may be present cannot be included. Inorganic pigments which are frequently used as delustrants in the manufacture of man-made fibers and types of dyes are not removed by the procedures described in 8.4. Some of the newer finishes present special problems and the analyst will have to deal with these cases as they arise. When it is necessary to modify the procedure to remove nonfibrous constituents, the analyst shall make sure that cleaned (Note 5) specimens of the fibers under consideration do not lose weight when subjected to the same treatment. The treatments described in 8.4 will allow removal of most, but not all, nonfibrous materials that may be present on textiles. The total amount of nonfibrous materials removed by these treatments may be determined by subjecting the specimen in turn to each of the treatments described. When the type of finish present is known, only the appropriate treatment need be used.

8.2 Reagents:

8.2.1 *Ethyl Alcohol*—Pure ethyl alcohol or ethyl alcohol denatured with methyl alcohol.

8.2.2 *Solution*, of 5 % urea/1.5 % phosphoric acid (85 %).

8.2.3 *Trichlorotrifluoroethane*.⁶

8.3 *Test Specimen*—Take a specimen having a mass of approximately 5 g. Take care to prevent the loss of fibers in the treatments. Dry the specimen at 105 to 110°C to constant mass to obtain the oven-dry mass of the original specimen, mass *C*, and then subject the specimen to one or more of the treatments described in 8.4.

8.4 Procedure:

8.4.1 *Solvent Extraction* (for removal of oils, fats, waxes, certain thermoplastic resins, etc.)—Extract the dried specimen for 2 h with trichlorotrifluoroethane (Note 5) in a Soxhlet or similar extractor, siphoning over a minimum of six times. Air dry the specimen and then dry in an oven at 105 to 110°C to constant mass.

NOTE 5—Trichlorotrifluoroethane is nonflammable, has a very low level of toxicity, and a high degree of stability. It is recommended in preference to carbon tetrachloride, which has been used historically for its excellence as a solvent for oils, fats, and the like. This preference is based on the toxicity of carbon tetrachloride by absorption or inhalation and its tendency to decompose in the presence of moisture, with the formation of hydrochloric acid.

8.4.2 *Alcohol Extraction* (for removal of soaps, cationic finished, etc.)—Extract the dried specimen for 2 h with ethyl alcohol in a Soxhlet or similar extractor, siphoning over a minimum of six times. Air-dry the specimen and then dry in an oven at 105 to 110°C to constant mass.

8.4.3 *Aqueous Treatment* (for removal of water-soluble materials)—Immerse the dried specimen for 30 min in water at

50°C using a liquid to fabric ratio of approximately 100:1. Agitate the specimen occasionally by stirring with a glass rod or by mechanical means. Rinse three times in fresh portions of warm (50°C) water and dry in an oven at 105 to 110°C to constant mass.

8.4.4 *Enzyme Treatment* (for removal of starch, gelatin, etc.)—Immerse the specimen in an aqueous solution of a starch and protein-solubilizing enzyme preparation using the concentration, liquid to fabric ratio, temperature, and time of immersion recommended by the supplier of the enzyme preparation. After this treatment, thoroughly rinse the specimen with fresh portions of hot water. Thorough rinsing is necessary to remove China-clay or similar material from heavily filled fabrics. Dry the rinsed specimen in an oven at 105 to 110°C to constant mass.

8.4.5 *Removal of Amino-Aldehyde Resins*—Treat the specimen with 1.5 % phosphoric acid (85 %) and 5 % urea at 75°C for 15 to 30 min with occasional stirring. Rinse thoroughly in hot (80°C) water, and dry the specimen in an oven at 105 to 110°C to constant mass, mass *D*.

NOTE 6—The fiber mass remaining after being subjected to the treatments described in 8.4.1 – 8.4.5 is referred to as “clean fiber” in the following sections.

8.5 Calculation:

8.5.1 Calculate the total percentage of nonfibrous materials in the specimens using Eq 1:

$$\text{Nonfibrous materials, \%} = \frac{100(C - D)}{C} \text{ or } 100\left(1 - \frac{D}{C}\right) \quad (1)$$

where:

C = oven-dry mass of the original specimen, g, and
D = oven-dry mass of the specimen after acid treatment, g.

8.5.2 The percentage of nonfibrous materials removed by any of the individual treatments described in 8.4 may also be calculated using the dry mass of the specimen determined after specific stages.

8.6 Report:

8.6.1 State that the amount of nonfibrous material was determined as directed by Test Methods D629.

8.6.2 Describe the material(s) or product(s) sampled and the method of sampling used.

8.6.3 State the type of extraction used.

8.6.4 Report the individual and the average values of percent nonfibrous materials.

8.7 Precision and Bias:

8.7.1 *Precision*—The precision of the procedure for nonfibrous materials in Test Methods D629 is being established.

8.7.2 *Bias*—The procedure in Test Methods D629 for measuring the nonfibrous materials in textiles has no known bias because the value of the nonfibrous materials in textiles can be defined only in terms of a test method.

9. Moisture Content or Moisture Regain

9.1 *Scope*—This test method may be used to determine the amount of moisture in a textile either as it is received or when

⁶ The solvent grade of the trichlorotrifluoroethane is available under various trademarks including Freon, Isotron, Ucon, and also Genesolve.

it is in moisture equilibrium for testing in the standard atmosphere for testing textiles, as defined in Terminology D123.

9.2 Summary of Method—Specimens are heated until they reach constant mass; the loss in mass is considered moisture.

NOTE 7—When textiles are heated under the conditions described in 9.5, volatile materials in addition to moisture may be removed. If this possibility is known or suspected, it should be reported that the percentage loss in mass of the textile does or may include volatile substances as well as moisture.

9.3 Apparatus:

9.3.1 Weighing Bottle, glass, approximately 100-mL capacity, fitted with a ground-glass cover, or an aluminium weighing can, approximately 100-mL capacity, and having a tight-fitting cover.

9.3.2 Desiccator, containing anhydrous calcium sulfate (CaSO_4) or other suitable dehydrating agent.

9.3.3 Analytical Balance, capable of weighing to 0.1 mg.

9.3.4 Oven, maintained at 105 to 110°C.

9.3.5 Special Equipment, for drying specimens to constant mass, generally available in textile laboratories (conditioning ovens, etc.) may also be used. The apparatus listed in 9.3.1 – 9.3.4 and the procedure described in 9.5 are provided for laboratories not so equipped and shall be used in cases of dispute, with regard to the fiber composition of mixtures.

9.4 Test Specimen—Take a specimen having a mass of approximately 1 g and weigh it to the nearest 0.01 g.

9.5 Procedure:

9.5.1 Place the weighing bottle and cover separately in the oven. After heating for 1 h at a temperature of 105 to 110°C, replace the cover, transfer the weighing bottle to the desiccator, and allow it to cool to room temperature. Remove the cover momentarily to equalize the pressure, and with the cover in place, weigh the container. Repeat the heating, cooling, and weighing until the mass of the empty weighing bottle is constant to within ± 0.001 g.

9.5.2 Place the specimen to be tested in the container, cover, and weigh. Subtract the mass of the empty container (9.5.1) from this mass to obtain the as-received mass of the original specimen, mass *A*.

9.5.3 Place the uncovered weighing bottle and specimen in the oven for 1.5 h at a temperature of 105 to 110°C. Cover the container and transfer it and the specimen to a desiccator. When the container has cooled to room temperature, remove the cover momentarily to adjust the pressure, replace the cover, and weigh the container and the specimen. Repeat the heating for periods of not less than 20 min, cooling and weighing until the mass is constant to within ± 0.001 g. Subtract the mass of the empty container (9.5.1) from this mass to obtain the oven-dry mass of the original specimen, mass *B* (Note 7).

9.6 Calculation:

9.6.1 Calculate the moisture content of the specimen using Eq 2 and the moisture regain of the specimen using Eq 3:

$$\text{Moisture content, \%} = \frac{100(A - B)}{A} \text{ or } 100\left(1 - \frac{B}{A}\right) \quad (2)$$

$$\text{Moisture regain, \%} = \frac{100(A - B)}{B} \text{ or } 100\left(\frac{A}{B} - 1\right) \quad (3)$$

where:

A = as-received mass of the specimen, g, and

B = oven-dry mass of the specimen, g.

9.7 Report:

9.7.1 State that the moisture content of the specimen was determined as directed by Test Methods D629.

9.7.2 Describe the material(s) or product(s) sampled and the method of sampling used.

9.7.3 Report the individual and the average values.

9.8 Precision and Bias:

9.8.1 Precision—The precision of the procedure for determining moisture content in Test Methods D629 is being established.

9.8.2 Bias—The true value of the moisture content in textiles can only be defined in terms of a specific test method. Within this limitation, the procedure for determining the moisture content in Test Methods D629 has no known bias.

MECHANICAL SEPARATION OR DISSECTION

10. Fiber Analysis by Dissection

10.1 Scope—This method is intended for mechanical separation of yarns in a textile, provided the different fibers are segregated.

10.2 Summary of Method—A specimen is carefully selected to include all the yarns in a complete repeat of the pattern. The component yarns are mechanically separated, dried, and weighed separately after heating at 105 to 110°C to constant mass. The percentage composition is calculated for each fiber type present.

10.3 Apparatus:

10.3.1 Analytical Balance, accurate to 0.1 mg.

10.3.2 Weighing Bottle, approximately 25 mL capacity and having a tight-fitting cover.

10.3.3 Oven, maintained at 105 to 110°C.

10.3.4 Desiccator, with anhydrous calcium sulfate or equivalent.

10.4 Test Specimen—Take a specimen having a mass of approximately 1 g.

10.5 Procedure:

10.5.1 Select the specimen carefully to include all the yarns in a complete repeat of the pattern.

10.5.2 Place the entire specimen in a weighing container preweighed to the nearest 0.001 g.

10.5.3 Place the weighing container and specimen in an oven at 105 to 110°C for 1.5 h.

10.5.4 Remove the weighing container and specimen from the oven and transfer them to a desiccator. When the container has cooled to room temperature, weigh the container and the specimen to the nearest 0.001 g. Subtract the mass of the empty container from this mass to obtain the oven-dry mass of the specimen, mass *B*.

10.5.5 Separate the component yarns and place each of the fiber types into separate preweighed weighing containers.

10.5.6 Place the weighing containers with specimens in an oven for 1.5 h at 105 to 110°C.

10.5.7 Remove the weighing container and specimen from the oven and transfer to a desiccator. Allow the container to cool to room temperature and then weigh the containers and specimens to the nearest 0.001 g. Subtract the mass of the empty containers from these masses to obtain the oven-dry mass of the specimen, mass A_1 , A_2 , etc.

10.6 Calculation:

10.6.1 Calculate the percent composition of each fiber type, to the nearest 0.1 %, of dry sample using the following equation:

$$\text{Fiber type } A_1, \% \text{ dry} = 100(A_1/B) \quad (4)$$

where:

A_1 = separate fiber type dry mass from 10.5.7, and

B = original dry specimen mass from 10.5.4.

10.7 Report:

10.7.1 State that the specimens were tested as directed by mechanical separation or dissection of Test Methods D629.

10.7.2 Describe the material(s) or product(s) sampled and the method of sampling used.

10.7.3 Report the individual and the average values of percent of each fiber type, based on dry sample.

10.8 Precision and Bias:

10.8.1 *Precision*—An interlaboratory study has been organized. The analysis data will be completed within the year 1999 and the precision statement will be balloted for inclusion in the test method by the end of the year 2000.

10.8.2 *Bias*—The procedure in Test Methods D629 for measuring the fiber content of textiles by mechanical separation or dissection has no known bias because the values of fiber content of textiles by mechanical separation or dissection can be defined only in terms of a test method.

CHEMICAL TEST METHODS

11. Summary of Methods

11.1 A diagrammatic arrangement of binary mixtures of fibers and appropriate methods of analysis are shown in Table 1. Each analytical method is identified by a number and, where possible, two methods of analysis are provided for each binary mixture of fibers. The number or numbers inside parentheses refers to the method that dissolves the fiber shown at the top of the diagram. The number or numbers outside the parentheses indicates the method that dissolves the fiber listed at the left side of the diagram. Where two methods are listed for a specific binary mixture, the nonsubscript method number represents the method of choice.

11.2 An indication of the solubilities of various textile fibers in the solvents specified in the chemical methods of separation herein described is given in Table 2. These solubilities may be used as a guide in selecting appropriate methods for analyzing mixtures that contain more than two types of fibers.

11.3 In these methods of analysis, provision is made for expressing fiber composition either on the oven-dry mass of

clean fiber after the removal of nonfibrous materials or on the moisture regain basis.

12. Specimens and Symbols

12.1 For each of the methods of analysis described in Sections 13 – 22, take a specimen having a mass of approximately 1 g. Treat the specimen as directed in 8.4 and determine the mass of the clean fiber after oven-drying, mass F .

12.2 If requested, determine the mass of the original specimen after oven-drying (see 9.4), to the nearest 0.001 g, mass E .

12.3 Use the symbols E and F in the number equations in Sections 13 – 22, as follows:

$$E = \text{mass of the original specimen after oven - drying, g, and} \quad (5)$$

$$F = \text{mass of original dry clean specimen (after oven - drying), g.} \quad (6)$$

13. Chemical Test Method No. 1—Acetate Mixed With Other Fibers

13.1 *Scope*—This test method is intended primarily for the separation of acetone-soluble types of acetate, and should be used for mixtures of acetate (secondary) fibers and some other fibers including modacrylic fibers.

13.2 *Summary of Method*—Regular secondary acetate is dissolved in 80 % cold acetone and the remaining fiber is determined quantitatively by gravimetric methods. Percent composition is calculated on oven-dry or commercial moisture regain basis.

13.3 Apparatus:

13.3.1 *Analytical Balance*, accurate to 0.1 mg.

13.3.2 *Fritted Glass Filtering Crucible*, of medium porosity.

13.3.3 *Weighing Bottle*, large enough to hold the fritted glass crucible.

13.3.4 *Erlenmeyer Flask*, glass-stoppered, having a capacity of 500 mL.

13.3.5 *Ice Bath*, in container large enough to submerge the Erlenmeyer flask.

13.3.6 *Suction Flask*, with adapter.

13.3.7 *Oven*, maintained at 105 to 110°C.

13.3.8 *Desiccator*, with anhydrous calcium sulfate (CaSO_4) or equivalent.

13.4 Reagents and Materials:

13.4.1 *Acetone – Water Mixture*—Mix 80 volumes of acetone with 20 volumes of water at 0 to 2°C in the 500-mL flask, cooling by means of an ice bath.

NOTE 8—**Precaution:** In addition to other precautions, acetone is flammable and should be handled away from any ignition source. It is also very volatile and breathing the vapors should be avoided. The use of a hood is strongly recommended.

NOTE 9—Some types of acetate are not completely soluble in an acetone-water mixture, but are soluble in glacial acetic acid.

NOTE 10—If there are no modacrylic fibers present, the analysis may be carried out using 100 % acetone at room temperature (15 to 25°C).

13.5 Procedure:

13.5.1 Weigh the dried specimen and weighing container to the nearest 0.001 g. Label as Tare and Sample.

13.5.2 Place the specimen in an Erlenmeyer flask and cover with 100 mL of the acetone – water mixture at 0 to 2°C (Note 10).

13.5.3 Agitate vigorously the flask containing the specimen for 15 min at a room temperature of 15 to 30°C.

13.5.4 Transfer the contents of the flask to a fritted glass funnel fitted into a suction flask.

13.5.5 Filter out the residual fiber.

13.5.6 Repeat 13.5.2 – 13.5.5 two additional times.

13.5.7 Dry the residue on the funnel by suction and wash with several portions of 70°C water.

13.5.8 Collect the residue from above and blot dry on paper towels. Exercise care not to lose any fibers in the process. Examine for complete extraction and, if necessary, repeat 13.5.2 – 13.5.7.

13.5.9 Place the specimen in an open weighing container and dry at 105 to 110°C for at least 1.5 h. Do not put wet specimens in the oven while other specimens are drying.

13.5.10 Remove the weighing container from the oven and immediately place it uncovered into a desiccator over CaSO₄, allowing it to cool for at least 30 min.

13.5.11 Place cover on specimen container, remove the assembly from desiccator, and weigh to the nearest 0.001 g. Label as Tare and Treated.

13.5.12 Remove the specimen and weigh the empty container to the nearest 0.001 g. Label as Tare.

13.6 Calculations:

13.6.1 Calculate the percent composition, to the nearest 0.1 %, of dry sample using the following equations:

$$\text{Fiber dissolved, \% dry} = \frac{100(F - G)}{F} \text{ or } 100\left(1 - \frac{G}{F}\right) \quad (7)$$

where:

F = original dry clean specimen mass, g, (Tare and Sample) – (Tare) from 13.5.1 and 13.5.12, and

G = extracted dry clean residual specimen mass, g, (Tare and Treated) – (Tare) from 13.5.11 and 13.5.12.

$$\text{Fiber undissolved, \% dry} = 100 - \text{Fiber dissolved, \% dry} \quad (8)$$

13.6.2 If requested, calculate the percent composition, to the nearest 0.1 % on a commercial moisture regain basis (MRB) using the following equations:

$$G_1 = [(\text{Fiber dissolved, \% dry})(100 + MR_1)/100] \quad (9)$$

$$G_2 = [(\text{Fiber undissolved, \% dry})(100 + MR_2)/100] \quad (10)$$

where:

MR_1 = moisture regain % for the dissolved fiber, and

MR_2 = moisture regain % for the undissolved fiber.

$$\text{Fiber dissolved, \% MRB} = 100 G_1 / (G_1 + G_2) \quad (11)$$

$$\text{Fiber undissolved, \% MRB} = 100 - \% \text{Fiber Dissolved, \% MRB} \quad (12)$$

13.7 Report:

13.7.1 State that the specimens were tested as directed in Chemical Test Method 1 of Test Methods D629.

13.7.2 Describe the material(s) or product(s) sampled and the method of sampling used.

13.7.3 Report the individual and the average values of percent dissolved and undissolved fiber, based on the clean, dry sample.

13.7.4 Report the calculated composition, moisture regain basis, if requested.

13.8 Precision and Bias:

13.8.1 *Precision*—An interlaboratory study has been organized. The analysis data will be completed within the year 1999 and the precision statement will be balloted for inclusion in the test method by the end of the year 2000.

13.8.2 *Bias*—The procedure in Test Methods D629 for measuring the fiber content of textiles by chemical test methods has no known bias because the values of the fiber content of textiles by chemical test methods can be defined only in terms of a test method.

14. Chemical Test Method No. 2—Modacrylic Mixed With Cellulosic Fibers or Wool

14.1 *Scope*—This test method is applicable to mixtures of most modacrylic with cellulosic fibers or wool.

14.2 *Summary of Method*—Modacrylic fibers are dissolved in γ -butyrolactone leaving cellulosic fibers or wool. Percent composition is calculated on oven-dry or commercial regain basis.

14.3 Apparatus:

14.3.1 *Analytical Balance*, accurate to 0.1 mg.

14.3.2 *Weighing Container*, glass or metal.

14.3.3 *Beakers and Flasks*, and other laboratory glassware, as required.

14.3.4 *Oven*, maintained at 105 to 110°C.

14.3.5 *Sieve*, stainless steel, 180 μ m (80 mesh).

14.3.6 *Desiccator*, with anhydrous calcium sulfate (CaSO₄) or equivalent.

14.4 Reagents and Materials:

14.4.1 γ -Butyrolactone, American Chemical Society reagent.

NOTE 11—**Precaution:** In addition to other precautions, avoid contact of γ -butyrolactone with eyes, skin, or clothing. γ -butyrolactone causes irritation. Wear safety equipment when using this solvent.

14.5 Procedure:

14.5.1 Weigh the dried specimen and weighing container to the nearest 0.001 g. Label as Tare and Sample.

14.5.2 Place the specimen in a 250-mL beaker and cover with 100 mL of γ -butyrolactone.

14.5.3 Let stand for 60 ± 5 min at a room temperature of 15 to 30°C, stirring every 5 min.

14.5.4 Decant the liquid through the 550- μ m (30-mesh) strainer returning the specimen to the beaker.

14.5.5 Repeat 14.5.2 – 14.5.4 allowing the specimen to remain in γ -butyrolactone for 30 ± 5 min.

14.5.6 Wash the specimen in running water for at least 10 min. If the specimen is fabric, wash it in the original beaker. If the specimen is fibrous or in small pieces, wash it in the 180- μ m (80-mesh) sieve.

14.5.7 Collect the residue from above and blot dry on paper towels. Exercise care not to lose any loose fibers in the process. Examine for complete extraction and, if necessary, repeat 14.5.2 – 14.5.6.

14.5.8 Place the specimen in an open weighing container and dry at 150 to 110°C for at least 1.5 h. Do not put wet specimens in the oven while other specimens are drying.

14.5.9 Remove the weighing container from the oven and immediately place it uncovered into a desiccator over CaSO₄, allowing it to cool for at least 30 min.

14.5.10 Place cover on specimen container, remove assembly from desiccator and weigh to nearest 0.001 g. Label as Tare and Treated.

14.5.11 Remove the specimen and weigh the empty container to the nearest 0.001 g. Label as Tare.

14.6 Calculations:

14.6.1 Calculate the percent composition, to the nearest 0.1 %, of dry sample using the following equations:

$$\text{Fiber dissolved, \% dry} = \frac{100(F - G)}{F} \text{ or } 100\left(1 - \frac{G}{F}\right) \quad (13)$$

where:

F = original dry clean specimen mass, g, (Tare and Sample) – (Tare) from 14.5.1 and 14.5.11.

G = extracted dry clean residual specimen mass, g, (Tare and Treated) – (Tare) from 14.5.10 and 14.5.11.

$$\text{Fiber undissolved, \% dry} = 100 - \text{Fiber dissolved, \% dry} \quad (14)$$

14.6.2 If requested, calculate the percent composition, to the nearest 0.01 % on a commercial moisture regain basis (MRB) using the following equations:

$$G_1 = [(\text{Fiber dissolved, \% dry}) \cdot (100 + MR_1)] / 100 \quad (15)$$

$$G_2 = [(\text{Fiber undissolved, \% dry}) \cdot (100 + MR_2)] / 100 \quad (16)$$

where:

MR_1 = moisture regain % for dissolved fiber, and

MR_2 = moisture regain % for the undissolved fiber.

$$\text{Fiber dissolved, \% MRB} = 100 G_1 / (G_1 + G_2) \quad (17)$$

$$\text{Fiber undissolved, \% MRB} = 100 - \% \text{ Fiber dissolved, \% MRB} \quad (18)$$

14.7 Report:

14.7.1 State that the specimens were tested as directed in Chemical Test Method No. 2 of Test Methods D629.

14.7.2 Describe the material(s) and product(s) sampled and the method of sampling used.

14.7.3 Report the individual and average values of percent dissolved and undissolved fiber, based on the clean, dry sample.

14.7.4 Report the calculated composition, moisture regain basis, if requested.

14.8 Precision and Bias:

14.8.1 *Precision*—An interlaboratory study has been organized. The analysis data will be completed within the year 1999 and the precision statement will be balloted for inclusion in the test method by the end of the year 2000.

14.8.2 *Bias*—See 13.8.2

15. Chemical Test Method No. 3—Nylon 6 or Nylon 6–6 Mixed With Natural Fiber or Rayon

15.1 *Scope*—This test method is applicable to mixtures of Nylon 6 or Nylon 6–6 with cotton, rayon, or wool.

15.2 *Summary of Method*—Nylon fibers are dissolved in 90 % formic acid, and the other fibers present (rayon, wool, or cotton) are determined quantitatively by gravimetric methods. Percent composition is calculated either on oven dry or commercial moisture regain basis.

15.3 Apparatus:

15.3.1 *Analytical Balance*, accurate to 0.1 mg.

15.3.2 *Weighing Container*, glass or metal.

15.3.3 *Beakers and Flasks*, and other laboratory glassware, as required.

15.3.4 *Oven*, maintained at 105 to 110°C.

15.3.5 *Sieve*, stainless steel, 180 μm (80 mesh).

15.3.6 *Desiccator*, with anhydrous calcium sulfate (CaSO₄) or equivalent.

15.4 Reagents and Materials:

15.4.1 *Formic acid*, (HCOOH), 90 % purified.

NOTE 12—Precaution: In addition to other precautions, formic acid is corrosive to human tissue. Wear protective equipment and handle with care. Work with adequate ventilation.

15.5 Procedure:

15.5.1 Weigh the dried specimen and weighing container to the nearest 0.001 g. Label as Tare and Sample.

15.5.2 Place the specimen in a 250-mL beaker and cover with 100 to 150 mL of HCOOH (See Note 12).

15.5.3 Let stand for 30 ± 5 min at a room temperature of 15 to 30°C, stirring every 10 min.

15.5.4 Decant the liquid through the 550-μm (30 mesh) strainer, returning the specimen to the beaker.

15.5.5 Repeat 15.5.2 – 15.5.4.

15.5.6 Wash the specimen in running water for at least 10 min. If the specimen is fabric, wash it in the original beaker. If the specimen is fibrous or in small pieces, wash it in the 180-μm (80-mesh) sieve.

15.5.7 Collect the residue from above and blot dry on paper towels. Exercise care not to lose any loose fiber in this process. Examine for complete extraction and, if necessary, repeat 15.5.2 – 15.5.6.

15.5.8 Place the specimen in an open weighing container and dry at 105 to 110°C for at least 1.5 h. Do not put wet specimens in the oven while other specimens are drying.

15.5.9 Remove the weighing container from the oven and immediately place it uncovered into a desiccator over CaSO₄, allowing it to cool for at least 30 min.

15.5.10 Place cover on specimen container, remove the assembly from desiccator, and weigh to the nearest 0.001 g. Label as Tare and Treated.

15.5.11 Remove the specimen and weigh the empty container to the nearest 0.001 g. Label as Tare.

15.6 Calculation:

15.6.1 Calculate the percent composition, to the nearest 0.1 %, of dry sample using the following equations:

$$\text{Fiber dissolved, \% dry} = \frac{100(F - G)}{F} \text{ or } \left(1 - \frac{G}{F}\right) \quad (19)$$

where:

F = original dry clean specimen mass, g, (Tare and Sample) – (Tare) from 15.5.1 and 15.5.11, and

G = extracted dry clean residual specimen mass, g, (Tare and Treated) – (Tare) from 15.5.10 and 15.5.11.

$$\text{Fiber undissolved, \% dry} = 100 - \text{Fiber dissolved, \% dry} \quad (20)$$

15.6.2 If requested, calculate the percent composition, to the nearest 0.1 %, on a commercial moisture regain basis (MRB) using the following equations.

$$G_1 = [(\text{Fiber dissolved, \% dry}) \cdot (100 + MR_1)] / 100 \quad (21)$$

$$G_2 = [(\text{Fiber undissolved, \% dry}) \cdot (100 + MR_2)] / 100 \quad (22)$$

where:

MR_1 = moisture regain, %, for the dissolved fiber, and

MR_2 = moisture regain, %, for the undissolved fiber.

$$\text{Fiber dissolved, \% MRB} = 100 G_1 / (G_1 + G_2) \quad (23)$$

$$\text{Fiber undissolved, \% MRB} = 100 - \% \text{ Fiber dissolved, \% MRB} \quad (24)$$

15.7 Report:

15.7.1 State that the specimens were tested as directed in Chemical Test Method No. 3 of Test Methods D629.

15.7.2 Describe the material(s) or product(s) sampled and the method of sampling used.

15.7.3 Report the individual and average values of percent dissolved and undissolved fiber, based on the clean, dry sample.

15.7.4 Report the calculated composition, moisture regain basis, if requested.

15.8 Precision and Bias:

15.8.1 *Precision*—An interlaboratory study has been organized. The analysis data will be completed within the year 1999 and the precision statement will be balloted for inclusion in the test method by the end of the year 2000.

15.8.2 *Bias*—See 13.8.2

16. Chemical Test Method No. 4—Rayon Mixed With Cotton

16.1 *Scope*—This test method is applicable to mixtures of rayon with cotton.

16.2 *Summary of Method*—Rayon fibers are dissolved in 59.5 % sulfuric acid leaving cotton fibers. Percent composition is calculated either on oven-dry or commercial moisture regain basis.

16.3 Apparatus:

16.3.1 *Analytical Balance*, accurate to 0.1 mg.

16.3.2 *Weighing Container*, glass or metal.

16.3.3 *Beakers and Flasks*, and other laboratory glassware, as required.

16.3.4 *Oven*, maintained at 105 to 110°C.

16.3.5 *Sieve*, stainless steel, 180 μm (80 mesh).

16.3.6 *Desiccator*, with anhydrous calcium sulfate (CaSO_4) or equivalent.

16.4 Reagents and Materials:

16.4.1 *Sulfuric Acid* (H_2SO_4)—American Chemical Society grade, 59.5 ± 0.5 % weight mass, specific gravity $1.493 \pm 0.027 \text{ Mg/m}^3$ at 20°C. Add sulfuric acid (density 1.84 Mg/m^3) slowly to water in the ratio of 1 mL acid to 1.25 mL water.

16.4.2 *Sodium Bicarbonate* (NaHCO_3)—Two percent prepared by adding 20 g of NaHCO_3 to 500 mL water and diluting to 1 L.

NOTE 13—**Precaution:** In addition to other precautions, sulfuric acid is corrosive to human tissue. Wear protective equipment and handle with care.

16.5 Procedure:

16.5.1 Weigh the dried specimen and weighing container to the nearest 0.001 g. Label as Tare and Sample.

16.5.2 Place the specimen (weighing approximately 1.0 g) in a 500-mL beaker and cover with 100 to 150 mL of 59.5 % H_2SO_4 .

16.5.3 Let stand for 15 ± 2 min at a room temperature of 15 to 30°C, stirring every 5 min.

16.5.4 Decant the liquid through the 550- μm (30-mesh) strainer returning the specimen to the beaker.

16.5.5 Repeat 16.5.2 – 16.5.4 but allow the specimen to remain in the acid for 30 ± 5 min.

16.5.6 Repeat 16.5.2 – 16.5.4 but allow the specimen to remain in the acid for 5 ± 1 min.

16.5.7 Wash the specimen in running water for at least 10 min. If the specimen is fabric, wash it in the original beaker. If the specimen is fibrous or in small pieces, wash it in the 180- μm (80-mesh) sieve.

16.5.8 Place the specimen in a 2 % NaHCO_3 solution and allow it to stand for at least 5 min, then repeat 16.5.7.

16.5.9 Collect the residue from above and blot dry on paper towels. Exercise care not to lose any loose fiber in the process. Examine for complete extraction.

16.5.10 Place the specimen in an open weighing container and dry at 105 to 110°C for at least 1.5 h. Do not put wet specimens into the oven while other specimens are drying.

16.5.11 Remove the weighing container from the oven immediately and place it uncovered into a desiccator over CaSO_4 , allowing it to cool for at least 30 min.

16.5.12 Place cover on specimen container, remove the assembly from desiccator, and weigh to nearest 0.001 g. Label as Tare and Treated.

16.5.13 Remove the specimen and weigh the empty container to the nearest 0.001 g. Label as Tare.

16.6 Calculations:

16.6.1 Calculate the percent composition, to the nearest 0.1 %, of dry sample using the following equations:

$$\text{Fiber dissolved, \% dry} = \frac{100 (F - G)}{F} \text{ or } \left(1 - \frac{G}{F} \right) \quad (25)$$

where:

F = original dry clean specimen mass, g, (tare and sample) – (Tare) from 16.5.1 and 16.5.12, and

G = original dry clean specimen mass, g, (tare and treated) – (Tare) from 16.5.11 and 16.5.12.

$$\text{Fiber undissolved, \% dry} = 100 - \text{Fiber dissolved, \% dry} \quad (26)$$

16.6.2 If requested, calculate the percent composition to the nearest 0.1 % on a commercial regain basis (MRB) using the following equations:

$$G_1 = [(\text{Fiber dissolved, \% dry}) \cdot (100 + MR_1)] / 100 \quad (27)$$

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