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Test Method for Finishes in Textiles: Identification

1. Purpose and Scope

1.1 This test method gives basic procedures for qualitative identification of various finish components present on textile materials.

1.2 The method is applicable to textile fabrics, yarns or fibers.

2. Principle

2.1 The identification scheme may involve any or all of the following approaches:

2.1.1 Sequential solvent extractions followed by identification of extracts by infrared spectroscopy (IR), gas chromatography (GC), high performance liquid chromatography (HPLC), thin layer chromatography (TLC), mass spectrometry (MS) or other instrumental or wet-chemical methods.

2.1.2 Direct measurement of elemental or chemical species on fabric by X-ray fluorescence (XRF) spectroscopy, infrared reflectance spectroscopy, furnace atomic absorption (AA) spectroscopy, inductively coupled plasma (ICP) spectroscopy and other instrumental or wet-chemical analysis methods.

2.1.3 Identification of specific finishing components by chemical spot tests on the textile or extracts from textiles.

3. Referenced Documents

3.1 AATCC TM20, Test Method for Fiber Analysis: Qualitative (see 16.1).

3.2 *Analytical Methods for a Textile Laboratory*, Fourth Edition, pending, American Association of Textile Chemists and Colorists, Research Triangle Park, NC (see 16.1).

3.3 *Infrared Spectra for the Identification of Chemical Finishing Agents from Textiles*, Part I, ARDS-S-47, October 1974, Agricultural Research Service, U.S. Dept. of Agriculture.

3.4 *Handbook of Chemical Specialties*, John E. Nettles, Chapter on Textile Fiber Processing, Preparation and Bleaching, John Wiley & Sons, New York.

3.5 *Textile Chemist and Colorist*, Vol. 5, No. 12, December 1973, p279 (see 16.1).

3.6 Walker, J. F. *Formaldehyde*, Reinhold, NY, 1964.

3.7 Feigl, F., *Spot Tests in Organic Analysis*, 7th Edition, Elsevier, Amsterdam, 1966.

3.8 VanLoo, W. J., Jr., et al., *American Dyestuff Reporter*, Vol. 45, 1956, p397.

3.9 Feigl, F., *Spot Tests in Inorganic*

Analysis, 7th Edition, Elsevier, Amsterdam, 1966.

3.10 *Textile Chemist and Colorist*. Vol. 21, No. 26, June 1989, p13 (see 16.1).

4. Terminology

4.1 **chemical finish**, n.—chemical material other than colorants and residual processing chemicals added to textiles to impart desired functional or aesthetic properties to the textile product.

5. Safety Precautions

5.1 The safety precautions specified in the method/procedure are ancillary to the testing procedures and are not intended to be all inclusive.

5.2 It is the user's responsibility to reference applicable safety data sheets, use safe and proper techniques, and wear appropriate personal protective equipment in handling materials in this standard.

5.3 Users MUST consult manufacturers for specific details such as equipment operating instructions and other recommendations. Consult and follow all applicable health and safety regulations (e.g., OSHA standards and rules).

5.4 Refer to each procedure for additional safety precautions regarding handling acids, hexane and methanol flammability, and reflux precautions.

6. Limitations

6.1 This test method is a flexible set of guidelines for identification of textile finishes. Appropriate adjustments and modifications will be needed as finishing chemicals change and as new analytical test methods are developed.

6.2 Any chemical which is on the textile prior to finishing may be present on the finished fabric and could be removed and/or detected as finish. Warp sizes, [e.g., starches, acrylics, waxes, polyesters, poly(vinyl alcohol)], dyes, dye fixatives, binders, spin finishes, natural gums, sugars, optical brighteners, and processing chemicals (e.g., surfactants, bleach stabilizers, dye carriers, and oils) may be present by design or unintentionally. A working knowledge of these chemicals is needed to properly detect and identify them. Where possible, supplier should be consulted for further information.

7. Sample Preparation

7.1 Dry textile samples at $105 \pm 1^\circ\text{C}$ ($221 \pm 2^\circ\text{F}$) and desiccate to constant

weight prior to weighing for subsequent extractions.

7.2 Additional preparation of samples, apparatus, and reagents is described in subsequent sections. Read all sections to ensure proper preparation and safety precautions before starting.

8. Solvent Extraction Procedures

8.1 Extraction with various solvents is intended to remove as many finishing chemicals as possible in a sequential order. The extraction scheme presented here (see Table I) was chosen on the basis of solvent polarity, solubility, volatility, economy and safety.

8.2 Use chemical goggles or face shield, impervious gloves and an impervious apron during dispensing and mixing of alkalis, acids and organic solvents. Handle concentrated acids only in an adequately ventilated laboratory hood. CAUTION: Always add acid to water.

8.2 Changes in chemical finishes, safety considerations or personal preferences may dictate the use of other solvents. The user is responsible for determining which finish types may be removed by each solvent.

8.3 Some finishes may not be soluble in any solvent. An alternate method for insoluble finishes such as fully-crosslinked polymers [e.g., some acrylics, polyurethanes, silicones and poly(vinyl acetates)] is to dissolve all of the fibrous material with a suitable solvent, leaving the residue of finish for further analysis. Refer to AATCC TM20, Table V (see 3.1).

8.4 Procedure. Extract a weighed textile sample (1-10 g depending on the size of the extractor) in sequence by the solvents in Table I, using Soxhlet reflux, beaker or pressurized solvent extractions depending on equipment availability. Carry out reflux procedure in a well-ventilated hood with an electrical heater or a water bath as the heating source. Use chemical goggles or face shield, impervious gloves and an impervious apron when handling organic solvents.

Extraction times may range from 30 min to several hours depending on the boiling point, exchange rate, apparatus and knowledge of the history of finish removal for the textile sample in question. Dry samples completely before beginning the next solvent in the sequence.

After the extractions are complete, filter each solvent and evaporate or distill, leaving the residual finish extract. The extract may be transferred to a weighing

Table I

Group	Solvent	Polarity Index*	Typical Finishing Chemicals Removed
1	Cold Hexanes	0.1	Oils, silicone oils
2	Hot Hexanes	1.0	Oils, waxes, silicones, some softeners, ester lubricants
3	Methanol	5.1	Unfixed cellulose reactants, organic salts, sulfonated organics
4	Water	10.2	Unfixed cellulose reactants, urea, linear starches, poly(vinyl alcohol)
5	0.1 N Hydrochloric acid	10.2	Fixed cellulose reactants, branched starches, inorganic salts

*Burdick and Jackson

NOTE: Some types of fibers may be partially dissolved by the solvents. Pre-check the fiber types to avoid errors. Refer to AATCC TM20 (see 3.1).

CAUTION: Hexanes and methanol are flammable liquids and should be stored in the laboratory only in small containers away from heat, open flames and sparks. These chemicals should not be used near an open flame.

pan prior to complete dryness; drying can then be completed in an oven at 105 ±1°C.

It may be useful to neutralize the acid extract with dropwise addition of dilute sodium hydroxide to avoid further degradation of the finish during evaporation. The quantitative results will reflect the presence of sodium chloride in the dried residue. In addition, contamination of the solution by sodium carbonate from the reaction of sodium hydroxide with atmospheric carbon dioxide may interfere with interpretation of subsequent infrared spectra.

Calculate the percent extract using Eq. 1:

Extract =

$$100 \times \frac{\text{Weight of Residue}}{\text{Weight of Original Fabric}} \quad (\text{Eq. 1})$$

The percent extract, coupled with subsequent identification, will be useful in distinguishing true finish components from residual non-finishing chemicals that may have been left on the fabric; e.g., natural waxes from softeners.

Identify the extracted finish by one of the analytical techniques described below. Additional information may be found in *Handbook of Chemical Specialties*, Textile Fiber Processing, Preparation and Bleaching chapter (see 3.4).

9. Infrared Spectroscopic Procedures

9.1 Infrared spectroscopy offers a convenient way to identify extracted finish from textiles. The basis for infrared spectroscopy is the structural information about a compound that can be found from its infrared spectrum. Infrared absorption bands in the 4000 to 250 wavenumbers region result from specific functional groups in a molecule. These can be compared with spectra of finishes in published or personal libraries. Visual in-

spection by a trained analyst will quickly identify the general type of finish. Computerized search-and-match capabilities on many infrared systems will be helpful. For more complete information, including typical wavenumbers for comparison of spectra of textile materials, descriptions of newer techniques such as Fourier transform infrared spectroscopy (FTIR), attenuated total reflectance spectroscopy (ATR) and primary references, refer to *Analytical Methods for a Textile Laboratory* (see 3.2). For representative spectra of chemical finishes, refer to *Infrared Spectra for the Identification of Chemical Finishing Agents from Textiles* (see 3.3).

9.2 FTIR spectrometers are capable of producing high quality spectra for identification and reference purposes. Other spectroscopic techniques such as laser-Raman spectroscopy, mass spectrometry and UV-Visible spectroscopy may also be utilized, if available, to further identify the extracts. Figs. 1-6 represent infrared spectra run on an FTIR spectrometer at a resolution of 4 wavenumbers of a fatty ester softener, a silicone softener, a dimethyldihydroxyimidazolidinone (DMDHI, aka DMUG) cellulose reactant, a glycolated dimethyloldihydroxyethylene-urea (DMDHEU) cellulose reactant, a tetrakis-(hydroxymethyl)phosphonium chloride (or sulfate) flame retardant and a C-6 fluorochemical oil/water repellent, respectively. Fig. 7 represents a laser-Raman spectrum of an aqueous solution of a DMDHEU cellulose reactant using an argon ion laser source.

9.3 Infrared Sample Preparation.

9.3.1 Techniques. Prepare finish samples for infrared analysis by dissolving the extract in suitable solvent for analysis as a liquid or as a cast film on an ATR crystal.

9.4 Infrared Analysis.

9.4.1 Obtain a spectrum of the sample or extract prepared by one of the above

techniques. Compare the sample spectrum with infrared spectra of known finishes. For laboratories that possess an FTIR instrument, it is strongly recommended that an in-house spectral library of appropriate finishing agents be developed. For more accurate identification, the spectrum of the known compound should be obtained with the same application and extraction history as the unknown finish.

9.4.2 Softeners. Quaternary ammonium compounds, polyethylenes, polyoxyethylene glycols, polyurethanes, polyacrylates and silicones are soluble or partially soluble in hot hexanes. Concentrate the extract and transfer to a clean ATR crystal.

9.4.3 Durable Press Reactants. Since most durable press reactants for cellulose can be extracted with either water or hydrochloric acid, this provides an alternate method for removing finishes for infrared spectroscopy.

Use chemical goggles or face shield, impervious gloves and an impervious apron during dispensing and mixing of alkalis, acids and organic solvents. Handle concentrated acids only in an adequately ventilated laboratory hood. CAUTION: Always add acid to water.

Reflux approximately 0.2 g fabric, cut into small squares, for 5 min in 0.1 N HCl. Carry out reflux procedure in a well-ventilated hood with an electrical heater or a water bath as the heating source. Use chemical goggles or face shield, impervious gloves and an impervious apron when handling organic solvents.

Decant the solution into a 50-mL round-bottom flask with 350 mg spectral grade KBr.

Evaporate to dryness on a rotary evaporator while the flask is immersed in an ice-salt bath to prevent further hydrolysis.

Complete final drying over phosphorus pentoxide.

Mix the residue with more KBr (in a 50 mg:300 mg ratio) and press into a disk.

Obtain a spectrum and compare to that of known compounds, with spectra of hydrolysates of known compounds preferable for accurate comparison.

Another method of sample preparation for identification of durable press cellulose reactants by infrared spectroscopy is to grind finished and unfinished fabrics and pelletize the two samples with spectral grade KBr. The unfinished pellet is run as the background and its spectrum subtracted from the spectrum of the finished sample pellet. It is important to use the same weights of both fabric samples to compensate for the cellulose bands. The resulting spectrum from the subtraction process represents the spectrum of the finish.