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# Test Method for Mercerization in Cotton

## 1. Purpose and Scope

1.1 This test method provides a method for the determination of the presence of mercerization in dyed and undyed cotton yarns and fabrics. In addition, the test will give an indication of the completeness of the reaction between the cotton and the mercerization bath.

## 2. Principle

2.1 Carefully scoured specimens of the cotton to be tested and unmercerized cotton are immersed in separate baths of barium hydroxide solution for a definite time period. Aliquot portions of each soaking bath and of fresh barium hydroxide solution are then titrated with hydrochloric acid.

2.2 The ratio of the amount of barium hydroxide absorbed by the mercerized specimen to that absorbed by the unmercerized specimen multiplied by 100 gives the barium activity number.

## 3. Terminology

3.1 **mercerization**, *n.*—a process for irreversibly altering the physical characteristics and appearance of natural cellulose fibers by swelling in strong alkali.

## 4. Safety Precautions

NOTE: These safety precautions are for information purposes only. The precautions are ancillary to the testing procedures and are not intended to be all inclusive. It is the user's responsibility to use safe and proper techniques in handling materials in this test method. Manufacturers MUST be consulted for specific details such as material safety data sheets and other manufacturer's recommendations. All OSHA standards and rules must also be consulted and followed.

4.1 Good laboratory practices should be followed. Wear safety glasses in all laboratory areas.

4.2 All chemicals should be handled with care.

4.3 Use chemical goggles or face shield, impervious gloves and an impervious apron during dispensing and mixing of barium hydroxide, sodium carbonate and hydrochloric acid. Concentrated acids should be handled only in an adequately ventilated laboratory hood. CAUTION: Always add acid to water.

4.4 Petroleum solvent can be a combustible or flammable liquid, depending on which solvent is used, and presents a

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

4.4.1 Carry out reflux procedure in a well ventilated hood with an electrical heating mantle or a water bath as the heat source.

4.4.2 Use chemical goggles or face shield, impervious gloves and an impervious apron when handling organic solvents.

4.5 An eyewash/safety shower should be located nearby and an organic vapor respirator and a self-contained breathing apparatus should be readily available for emergency use.

4.6 Exposure to chemicals used in this procedure must be controlled at or below levels set by governmental authorities [e.g., Occupational Safety and Health Administration's (OSHA) permissible exposure limits (PEL) as found in 29 CFR 1910.1000; see web site: [www.osha.gov](http://www.osha.gov) for latest version]. In addition, the American Conference of Governmental Industrial Hygienists (ACGIH) Threshold Limit Values (TLVs) comprised of time weighted averages (TLV-TWA), short term exposure limits (TLV-STEL) and ceiling limits (TLV-C) are recommended as a general guide for air contaminant exposure which should be met (see 13.7).

## 5. Limitations

5.1 The test cannot be used satisfactorily if durable finishes or fibers other than cotton are present.

5.2 Near-infrared spectroscopy (NIR) prediction of barium number may be an alternate method to titration. NIR has the advantages of being fast (values in minutes versus days) and is non-destructive. However, an NIR library must first be established to accurately predict barium number. This library must contain multiple known samples encompassing the barium number range of interest. Typically, these known representatives will be from titrated fabrics.

## 6. Apparatus

6.1 Burette (preferably automatic) (see 13.4).

6.2 Flask, Erlenmeyer, with reflux tube.

6.3 Flasks, glass-stoppered, 250 mL.

6.4 Flasks, Erlenmeyer, 125 mL.

6.5 Bottles, storage, 250-500 mL.

6.6 Beaker, 1500 mL.

6.7 Pipette, 10 mL.

6.8 Drying oven.

## 7. Reagents and Materials

7.1 Hydrochloric acid (HCl) standardized approx. 0.1N.

7.2 Barium hydroxide reagent [Ba(OH)<sub>2</sub>] approximately 0.25N (see 13.1).

7.3 Phenolphthalein.

7.4 Petroleum solvent [BP 30-60°C (86-140°F)].

7.5 Alcohol (95% ethanol or anhydrous methanol).

7.6 Enzyme, starch-solubilizing.

7.7 Soap, neutral, granular (see 13.2).

7.8 Water, distilled.

7.9 Cotton yarn, unmercerized, for reference (standard cotton) (see 13.3).

7.10 Cotton fabric, unmercerized, for reference (AATCC Crocking squares) (see 13.4).

## 8. Test Specimens

8.1 A minimum of 5 g of each sample and of the unmercerized standards are scoured as directed after which a 2 g specimen of each scoured sample is weighed and placed in clean, dry glass-stoppered flasks.

## 9. Procedure

9.1 Scouring. The purpose of the scouring operation is to remove all extraneous matter, leaving the cotton cellulose in as pure a form as possible and without changing it chemically.

9.1.1 The samples to be tested (at least 5 g each), together with the standard unmercerized cotton, are refluxed together successively for 1 h with petroleum solvent [boiling point 30-60°C (86-140°F)], 1 h with alcohol (95% USP ethanol, No. 30 specially denatured alcohol, 95% or anhydrous methanol may be used), and for 1 h with distilled water (see 13.4).

9.1.2 Following these three extractions, starches are removed as follows:

9.1.3 Cover the sample with distilled water containing 3% of a commercial starch-solubilizing malt enzyme solution and heat to 60 ± 5°C (140 ± 9°F). Maintain the solution at this temperature for a period of 1 h. Pour off the enzyme solution, rinse and then scour as follows.

9.1.4 Boil the samples together for 1 h in 1 L of water containing 10 g of a neutral soap and 2 g of soda ash. Wash repeatedly in warm water until free from soap and alkali, i.e., until neutral to phe-