Mercerization in Cotton

Developed in 1958 by AATCC Committee RA66; editorially revised 1974, 1984, 1986, 1988, 1990, 1992; reaffirmed 1974, 1977, 1980, 1989, 1998, 2003; editorially revised and reaffirmed 1985, 1994.

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 cellulosic 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 of January 1, 1989]. 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.

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. 0.1*N*.

7.2 Barium hydroxide reagent [$Ba(OH)_2$] approximately 0.25*N* (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).

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 phenolphthalein, squeeze and dry. The samples and the standard unmercerized cotton are dried in an oven at 100°C (212°F) until thoroughly dry. The samples are then allowed to come to room conditions. Each sample should then be cut into

small pieces [approximately 3 mm (0.125 in.) square] for subsequent weighing.

9.2 Testing. Prepare and test duplicate specimens from each sample. Weigh 2 g of each scoured sample and of the scoured standard cotton into dry 250-mL flasks equipped with stoppers. (Ground glass stoppers are recommended.) Add 30 mL 0.25*N* barium hydroxide (see 13.5) to each flask containing a test specimen and to two empty flasks for blank determinations. Stopper each flask immediately upon addition of the barium hydroxide and store them for at least 2 h in a water bath at 20-25°C (68-77°F) (room temperature). Shake the flasks at frequent intervals. After 2 h, transfer 10 mL of solution (see 13.6) from each container, including the blanks and titrate with 0.1*N* hydrochloric acid, using phenolphthalein as an indicator.

10. Calculations

10.1 Using the titration figures, determine the ratio of barium hydroxide absorbed by a mercerized specimen to that absorbed by the unmercerized standard. Multiply this ratio by 100 to obtain the barium activity number.

Example: 10 mL of barium hydroxide solution (blank) required 24.30 mL of 0.1*N* HCl; 10 mL of barium hydroxide from an unknown sample of cotton required 19.58 mL of 0.1*N* HCl; 10 mL of barium hydroxide from unmercerized cotton (standard) required 21.20 mL of 0.1*N* HCl. Therefore the barium number of the unknown sample is:

$$\frac{24.30 - 19.58}{24.30 - 21.20} \times 100 = 152$$

10.2 Barium numbers should be run in duplicate and should be reported separately. Duplicate runs should not be off by more than four units. The titrations should be within 0.1 mL for check results. Skilled operators can estimate to within 0.05 mL. A difference of more than four units between duplicate runs indicates inaccuracy in running the test (see Table I).

11. Interpretation

11.1 A barium activity number in the range of 100-105 indicates no mercerization. A barium activity number above 150 indicates substantially complete reaction between the cotton and the mercerizing bath. Intermediate numbers indicate either incomplete reaction or use of a weak mercerizing bath.

Table I—Barium Numbers: Interlaboratory Tests on Mercerized Fabrics

Cloth	Lab A	Lab B	Lab C[1]	Lab C[2]	Lab C[3]
80 × 80—35° Tw	118	118	117	120	114
80 × 80—55° Tw	130	131	128	132	125
108 × 58—55° Tw	141	145	143	143	140
136 × 64—55° Tw	122	123	123	122	120
88 × 50—55° Tw	139	140	136	140	133

[1] Fabrics scoured by Lab C, unmercerized 80 × 80 as standard.
[2] Fabrics scoured by Lab A, unmercerized 80 × 80 as standard.
[3] Fabrics scoured by Lab C, 40/2 combed mercerized yarn as standard.

12. Precision and Bias

12.1 *Precision.* Precision for this test method has not been established. Until a precision statement is generated for this test method, use standard statistical techniques in making any comparisons of test results for either *within-laboratory* or *between-laboratory* averages.

12.2 *Bias.* Mercerization in cotton can be defined only in terms of a test method. There is no independent method for determining the true value. As a means of estimating this property, the method has no known bias.

13. Notes

13.1 The barium hydroxide reagent is prepared by shaking distilled water with slightly more than the calculated quantity of barium hydroxide, allowing it to stand overnight in a stoppered bottle, and then siphoning the clear solution into a clean storage bottle.

13.2 Available from AATCC, P.O. Box 12215, Research Triangle Park NC 27709; tel: 919/549-8141; fax: 919/549-8933; e-mail: orders@aatcc.org.

13.3 Unmercerized standard cotton skeins (40/2 ply) as used for the Draves Wetting Tests are particularly satisfactory. They may be obtained from Testfabrics Inc., P.O. Box 26, 415 Delaware St., W. Pittston PA 18643; tel: 570/603-0432; fax: 570/603-0433; e-mail: testfabric@aol.com.

13.4 If it is known that all samples that test do not contain finish or starch, then the scouring procedure may begin with the soap-and-soda ash treatment. If one sample requires solvent extraction and the enzyme treatment, then all samples including the standard cotton must be treated together with the complete scouring procedure to insure the same final state for the whole set of samples.

13.5 An automatic burette is most convenient for the addition of the barium hydroxide solution to the specimens. The air outlet must be equipped with an absorption tube containing soda lime to remove carbon dioxide. The latter must not be allowed to enter any burette that might be used, because formation of barium carbonate not only affects the concentration of the reagent but forms a film which interferes with burette readings. A cork is fitted to the bottom of the burette in such a manner that the 250 mL Erlenmeyer flasks containing the specimens under test are locked into place without exposure to air during the addition of barium hydroxide. The barium hydroxide solution should cover the specimens, tilting the flasks if necessary to accomplish this end.

13.6 For the removal of 10 mL of barium hydroxide when equilibrium has been reached, a 10 mL pipette is used. The same pipette, burette, etc., should be used for the whole set of determinations and the same technique for emptying or filling pipettes and burettes should be used on each determination. The hydrochloric acid burette is also equipped with a cork to which the 125 mL flask can be attached during the titration of the 10 mL aliquot portions of barium hydroxide, thereby eliminating titration errors resulting from carbon dioxide absorption by the alkaline solution. In removing the 10 mL aliquot portion of barium hydroxide from flasks containing specimens, the operator should use the end of his pipette to push the cotton against the wall of the flask and to express the excess liquor. In this way, a larger amount of the solution will be available for drawing up into the pipette.

13.7 Available from Publications Office, ACGIH, Kemper Woods Center, 1330 Kemper Meadow Dr., Cincinnati OH 45240; tel: 513/742-2020.