

Designation: D 1464 - 90 (Reapproved 2002)

# Standard Test Method for Differential Dyeing Behavior of Cotton<sup>1</sup>

This standard is issued under the fixed designation D 1464; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

- 1.1 This test method covers a test for the characterization of certain dyeing behavior of cotton. This test method is especially applicable to raw cotton fibers, but may also be adapted to cotton yarns and fabrics.
- 1.2 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:
- D 123 Terminology Relating to Textiles<sup>2</sup>
- D 1441 Practice for Sampling Cotton Fibers for Testing<sup>2</sup>

# 3. Terminology

- 3.1 Definitions:
- 3.1.1 differential dyeing behavior, n—of cotton, the tendency of cotton fibers to absorb and retain selectively varying proportions of different dyes from a binary dye bath.
- 3.1.2 For definitions of other textile terms used in this test method, refer to Terminology D 123.

## 4. Summary of Test Method

4.1 Specimens are dyed under specified conditions in a bath containing a mixture of prescribed red and green dyes. The resultant colors are compared with those of cottons of known dyeing behavior or fiber properties.

## 5. Significance and Use

- 5.1 This test method is not recommended for acceptance testing because the information obtained is qualitative rather than quantitative in nature (see Section 12).
- 5.2 The response of fibers to the dye mixture is indicative of the uniformity of dyeing, the probability of dyeing defects due to neps, and the difficulties to be expected from heterogeneity of fibers that differ in dyeing characteristics. In general, the red dye is retained predominantly by the thick-walled fibers and

the green by the thin-walled fibers.

5.3 The color response of the specimens to the dyes in the binary mixture reflects the behavior to be expected with many other dyes. The colors from red to green may be influenced by factors such as weather during the growing period, exposure of open bolls in the field before harvest, natural color, fineness, and degree of wall development.

### 6. Apparatus

- 6.1 *Balance*, having a capacity of 1000 g and a sensitivity of 0.05 g.
- 6.2 Beakers, preferably of stainless steel, having capacities of 400, 1200, 2000, and 3000 mL.
  - 6.3 Clothes Wringer, or Centrifuge.
- 6.4 *Cylinders*, graduated, having capacities of 5, 10, 25, 50, 100, and 500 mL or *Burets* (automatic, screw-top, acid-bottle type preferred).
  - 6.5 Büchner Filter Funnel, coarse, fritted-disk, 80-mm.
  - 6.6 Source of Suction.
  - 6.7 Sewing Machine, or Needle and Thread.
  - 6.8 Timer (with Second Hand), or Stop Watch.
  - 6.9 Wiley Mill, laboratory, intermediate model.

## 7. Reagents and Material

- 7.1 *Dyes*:
- 7.1.1 CI Direct Green 26<sup>3</sup> (Color Index No. 34045), 1 g/100 mL stock solution.
- 7.1.2 CI Direct Red 81<sup>4</sup> (Color Index No. 28160), 0.5 g/100 mL stock solution.
  - 7.2 Distilled Water.
- 7.3 Sodium Carboxymethyl Cellulose, low viscosity ("Carbose D").<sup>5</sup>
- 7.4 Sodium Carboxymethyl Cellulose Solution (1%)—Dissolve 0.1 g of sodium carboxymethyl cellulose in distilled water and dilute to 100 mL.

Note 1—Any commercially available distilled water or water singly distilled in laboratory glassware is acceptable.

<sup>&</sup>lt;sup>1</sup> This test method is under the jurisdiction of ASTM Committee D13 on Textiles, and is the direct responsibility of Subcommittee D13.11 on Cotton and Fibers.

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<sup>&</sup>lt;sup>2</sup> Annual Book of ASTM Standards, Vol 07.01.

<sup>&</sup>lt;sup>3</sup> Solophenyl Green BL, available from Ciba-Geigy Corp., Dyestuffs and Chemical Div., P.O. Box 11422, Greensboro, NC 27409, or equivalent, has been found suitable.

<sup>&</sup>lt;sup>4</sup> Diphenyl Red 5 BL, available from Ciba-Geigy Corp., Dyestuffs and Chemical Div., P.O. Box 11422, Greensboro, NC 27409, or equivalent, has been found suitable.

<sup>&</sup>lt;sup>5</sup> Available from Carbose Corp., 100 Maple St., Somerset, PA 15501.



- 7.5 Sodium Chloride Solution (1%)—Dissolve 1 g of reagent grade NaCl in 100 mL of distilled water.
- 7.6 *Bleached Gauze*, such as cheesecloth, or gauze having a fabric count of 28 by 24 (approx 11 by 9.5/10 mm).

# 8. Sampling, Selection, and Number of Specimens

- 8.1 Take a lot sample and laboratory sample as directed in the applicable material specification or by a plan agreed upon by the parties interested in the test results or, in the absence of such specification or agreement, as directed in Practice D 1441.
- 8.2 Take a 3 or 4-g test specimen from the sample and place loosely in a closable bleached cotton gauze bag. Alternatively spread out a group of such specimens, slightly separated, between two pieces of cotton gauze, allowing an area of about 4 by 4 in. (100 by 100 mm) for each specimen, and sew a seam around each specimen to form a compartment for it. Identify each specimen by some appropriate coding system. Consider the group as a bagged specimen in the subsequent discussion.

# 9. Conditioning

9.1 It is not necessary to precondition or condition specimens.

Note 2—If samples are obviously wet, dry them in the prevailing room atmosphere.

### 10. Procedure

- 10.1 Weigh the bagged specimen (specimen and gauze) to obtain the air-dry weight of the total amount of cotton to be dved.
- 10.2 Prepare a dye bath containing the following for each gram (air-dry weight) of the cotton (specimen and gauze), 2.4 mL of the red dye stock solution, 2.8 mL of the green stock solution, and distilled water to make a total volume of 40 mL. The dye bath will weigh 40 times the weight of the specimen and gauze.
- 10.3 Heat the dye bath to boiling, add the bagged specimens without prewetting, and boil for 15 min. Keep the specimens submerged and stir at approximately 3-min intervals. After 15 min at the boil, lift the specimens above the bath and add, with stirring, 2.5 mL of the sodium chloride stock solution per gram of the total weight (specimen and gauze), and sufficient distilled water to maintain the 40-mL volume for each gram of the total weight. Submerge the specimens in the boiling bath for a second 15-min dyeing period; then lift the specimen above the bath and add, while stirring, a second 2.5 mL of the sodium chloride stock solution, per gram of the original total weight. Replace the specimens in the boiling dye bath for a final 15 min. The total dyeing time is 45 min.

10.4 Lift the dyed specimen from the bath, squeeze, cool, and wash with two changes of distilled water at room temperature in the proportion of 50 mL of water to 1 g of air-dry total weight. Squeeze out the excess water and pull apart the matted fibers while in the gauze bag. Dip the bagged specimen into vigorously boiling distilled water (50 times the original total weight) (Notes 2 and 3) for  $30 \pm 1$  s with vigorous stirring, lift out, and remove the excess water as rapidly as possible by squeezing in a clothes wringer or by centrifuging. Rinse the stripped dyed cotton twice in water at room temperature to

remove the unbound dye, and squeeze or centrifuge. Handle each bagged specimen separately during this operation, but treat all in as nearly the same manner as possible to ensure uniform treatment and rapid uniform removal of the hot water.

Note 3—The after-treatment with boiling water has a partial and selective stripping effect. The excess red dye is removed with a relatively small proportion of the green dye, thus eliminating grayness and causing more clearly defined colors, especially green.

Note 4—The time of contact of the dyed material with boiling water is critical with respect to the final shade. Variations in after-treatment are largely responsible for difficulties in reproducing dyeings.

- 10.5 Remove the dyed specimens from the bags of the sewed gauze, fluff, and allow to dry in the prevailing room atmosphere.
- 10.6 Observe the over-all color and rank the dyeings according to relative distributions of red and green fibers in the specimens. They may range in color from a pronounced red to a distinct green.
- 10.7 If a more permanent record is desired, cut the specimen nearly to a powder, using a Wiley mill with 20-mesh screen. Disperse the cut cotton in an adhesive consisting of a 1 % solution of sodium carboxymethyl cellulose (1 g of cotton to 40 mL of solution). Filter by suction on an 80-mm coarse, fritted-disk funnel. Apply air pressure to the stem of the funnel to remove the cotton, and dry as a smooth flat pad. Place a gummed label on the rough upper side for identification. Keep the smooth underside for color inspection. Rank the series according to color, ranging from red to green, and record the order.

# 11. Report

- 11.1 State that the specimens were tested as directed in ASTM Test Method D 1464. Describe the sample including all available information as to the origin and history of the cotton, for example, crop year, variety, place of growth, and method of sampling.
  - 11.2 Report the following information:
- 11.2.1 The ranking of the dyed specimens in fiber form, and in pad form if determined, according to their color and shades of color from red to green. State the level of the color range from colors of cottons of known dye behavior or fiber properties.
- 11.2.2 A prediction of the response of products made from the same lots of cotton to similar or other dyes from their behavior in the binary dye mixture.

#### 12. Precision and Bias

- 12.1 *Precision*—Since the test results obtained with this method are qualitative in nature, no justifiable statement can be made on the precision of Test Method D 1464 for the differential dyeing behavior of cotton.
- 12.2 *Bias*—No justifiable statement on the bias of Test Method D 1464 for the differential dyeing behavior of cotton can be made since the true value of this property cannot be established by an accepted referee method.

# 13. Keywords

13.1 cotton; dyeing



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