



# Standard Test Method for Maturity of Cotton Fibers (Sodium Hydroxide Swelling and Polarized Light Procedures)<sup>1</sup>

This standard is issued under the fixed designation D 1442; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

## 1. Scope

1.1 This test method covers the determination of the percentage of mature fibers in a sample of loose, chemically untreated cotton fibers, whether taken before processing or unravelled from a textile product.

1.2 This test method gives two optional procedures for determining maturity, as follows:

1.2.1 Procedure 1—Sodium Hydroxide Swelling.

1.2.2 Procedure 2—Polarized Light.

NOTE 1—For other test methods for the determination of maturity of cotton fibers refer to Test Methods D 1464 and D 2480.

1.3 The values stated in SI units are to be regarded as standard. No other units of measure are included in this standard.

1.4 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 1440 Test Method for Length and Length Distribution of Cotton Fibers (Array Method)<sup>2</sup>

D 1442 Test Method for Maturity of Cotton Fibers (Sodium Hydroxide Swelling and Polarized Light Procedures)<sup>2</sup>

D 1447 Test Method for Length and Length Uniformity of Cotton Fibers by Fibrograph Measurement<sup>2</sup>

D 1464 Test Method for Differential Dyeing Behavior of Cotton<sup>2</sup>

D 1769 Test Method for Linear Density of Cotton Fibers (Array Sample)<sup>3</sup>

D 1776 Practice for Conditioning Textiles for Testing<sup>2</sup>

<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 Fibers.

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

<sup>3</sup> Discontinued 1988; see 1987 Annual Book of ASTM Standards, Vol 07.01.

D 2480 Test Method for Maturity Index and Linear Density of Cotton Fibers by the Causticaire Method<sup>4</sup>

## 3. Terminology

3.1 Definitions:

3.1.1 cotton fiber maturity, *n*—the degree of fiber wall development; the ratio of fiber wall width to lumen width.

3.1.1.1 Discussion—When cotton fibers are treated with a sodium hydroxide solution, mature fibers have a total wall width equal to or greater than the lumen width, and immature fibers have a total wall width less than the lumen width.

3.1.2 immature fibers, *n*—in testing with sodium hydroxide solutions, fibers that have either swollen and assumed a spiral form, or remained flat, thinly outlined, and almost transparent with total wall width less than the lumen width.

3.1.2.1 Discussion—See Fig. 1 and Fig. 2.

3.1.3 immature fiber, *n*—observed under polarized light, fibers that appear purple, indigo, or blue that turn orange or yellow-orange upon rotation to the subtractive position and show parallel extinction upon removal of the selenite plate.

3.1.3.1 Discussion—See Table 1.

TABLE 1 Colors of Cotton Fibers Viewed with Polarized Light<sup>A</sup>

Fiber Classification	Without Selenite Plate		With Selenite Plate	
	First Order	Additive Colors		Subtractive Colors
		Second Order	First Order	
Mature	light yellow white	yellow green	light yellow yellow	
Immature	gray-blue gray	blue purple	orange-yellow orange	

<sup>A</sup> Classified according to Mary Anna Grimes, "Polarized Light Preferred for Maturity Tests," *Textile World*, February, 1945.

3.1.4 lumen, *n*—in vegetable fibers, the central canal of the fiber.

3.1.5 mature fibers, *n*—in testing with sodium hydroxide solutions, fibers that have swollen into unconvoluted and almost rod-like shapes where total wall width is equal to or greater than the lumen width.

<sup>4</sup> Discontinued 1992; see 1991 Annual Book of ASTM Standards, Vol 07.01.

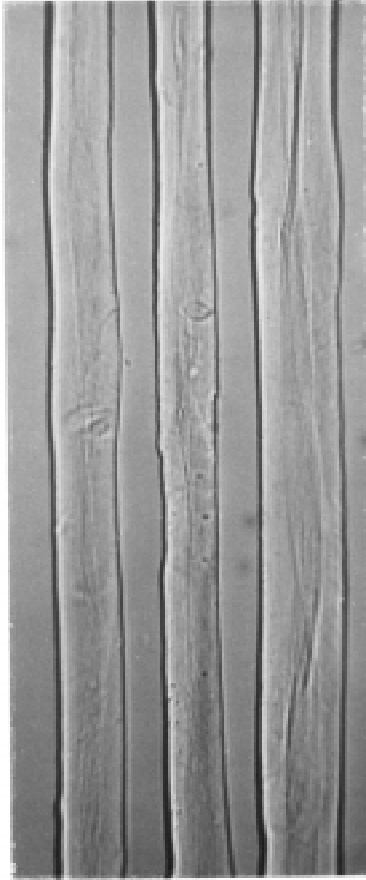


FIG. 1 Mature Fiber

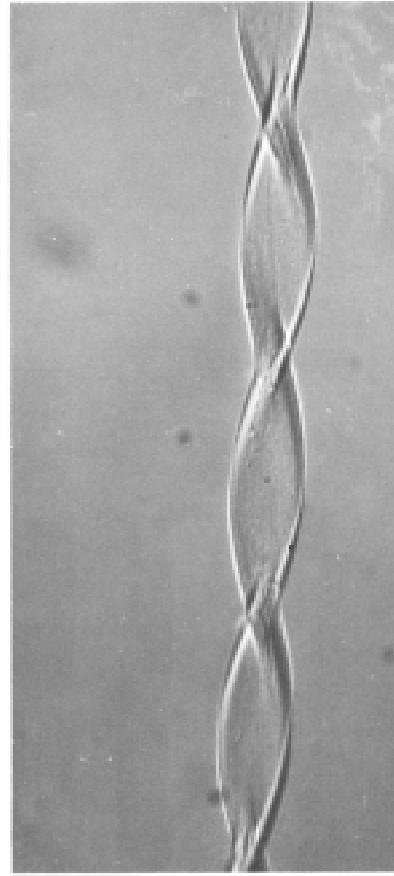


FIG. 2 Immature Fiber (Type A)

3.1.5.1 *Discussion*—See Fig. 3.

3.1.6 *mature fibers, n*—observed under polarized light, fibers that appear yellow, yellow-green, or green and are yellow or light yellow upon rotation to the subtractive position (through 90°) and show little or no parallel extinction upon removal of the selenite plate.

3.1.6.1 Cotton fibers observed under polarized light that appear blue or green may be classified separately for a finer distinction of fiber maturity.

3.1.6.2 *Discussion*—See Table 1.

3.1.7 *micronaire reading, n*—a relative measurement of fiber fineness derived from the porous plug air-flow method.

3.1.8 For definitions of other terms used in this method, refer to Terminology D 123.

3.1.9 *test specimen, n*—in cotton maturity tests, the series of slides observed by one technician as one-half of the test.

#### 4. Summary of Test Method

4.1 Fibers are laid parallel on a microscope slide, covered with a cover glass, treated with a mounting medium, and the magnified images are then classified as mature or immature fibers.

4.2 The method offers two procedures for classifying the fibers as mature or immature:

4.2.1 *Procedure 1, Sodium Hydroxide Swelling*, which uses an 18 % solution of sodium hydroxide as the mounting

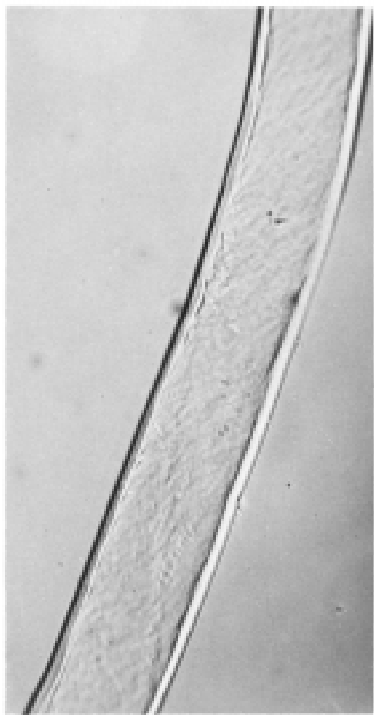
medium and a laboratory microscope for viewing the fibers at a magnification of 400×.

4.2.2 *Procedure 2, Polarized Light*, which uses water or clear mineral oil as the mounting medium and requires a polarizing microscope giving a magnification of 100×. Fibers are classified according to their second-order interference colors, using a first-order red selenite plate (Table 1).

#### 5. Significance and Use

5.1 Information regarding the percentage of immature fibers is desirable because immature fibers: (1) break easily during processing; (2) have a tendency to form neps; (3) have a tendency to become entangled around particles of trash and leaf, thus making cleaning more difficult and increasing the amount of fiber removed with foreign matter; (4) adversely affect yarn and fabric appearance; and (5) may appear differently after dyeing.

5.2 Maturity has a high positive correlation with linear density, but genetic differences and differences in wall thickness caused by plant diseases, soil, and water conditions during the growing season interfere with this relationship. Thus two cottons having the same linear density, or having the same average wall thickness as indicated by air-flow instruments, may vary greatly in maturity, that is, a cotton having extremely variable wall thickness may contain more immature fibers than another cotton of the same Micronaire reading composed of fibers having very uniform wall thickness.



**FIG. 3 Immature Fiber (Type B)**

5.3 The Sodium Hydroxide Swelling (Procedure 1) has been used in judging other maturity tests such as the Causticaire and the differential dye methods, in which the individual fibers are not examined.

5.4 Finer distinctions between different degrees of fiber wall development can be made with the Polarized Light procedure than with the Sodium Hydroxide Swelling procedure. The Polarized Light procedure gives a view of the fiber in its natural state so that fibrillar structure, striations, reversals, etc., are clearly visible as are growth abnormalities and variations in wall thickness. This method may be preferred by botanists, geneticists, and plant physiologists, while the Sodium Hydroxide Swelling procedure may be preferred for routine testing of large numbers of samples. Technicians are more easily trained for the latter method. Arbitrary classification as to maturity must be made with both methods.

5.5 This method is not considered satisfactory for acceptance testing because between laboratory precision can be poor. In some cases the purchaser and seller may have to test a commercial shipment of one or more specific material by an appropriate method even though the method has not been recommended for acceptance testing of commercial shipments. In such a case, if there is a disagreement arising from differences in values reported by the purchaser and seller in using this method for acceptance testing, the statistical bias, if any, between the laboratory of the purchaser and the laboratory of the seller should be determined with comparison based on tested specimens randomly drawn from one sample of material of the type being evaluated.

## 6. Apparatus and Reagents

### 6.1 Procedure 1:

6.1.1 *Microscope or Microprojector*, which will give a magnification of approximately 400×, equipped with a mechanical stage, microscope lamp, and viewing aid such as a Euscope or projection screen.<sup>5</sup>

6.1.2 *Metal Comb*, rake-type.<sup>6</sup>

6.1.3 *Microscope Slides*, 2 by 3 in. (50 by 75 mm), and appropriate cover glasses.

6.1.4 *Forceps, Dissecting Needles, and Tweezers*.

6.1.5 *Multiple Counter* with totalizer or a pair of *Single Counters*.

6.1.6 *Balance*,<sup>7</sup> with a capacity of 3 mg and a sensitivity of 0.005 mg (needed for specimens taken from array length groups only).

6.1.7 *Mounting Medium*, sodium hydroxide (NaOH) solution, 18 %, sp gr  $1.197 \pm 0.002$  at 60 to 70°F (16 to 20°C) in a dropping bottle.

6.2 *Procedure 2:*

6.2.1 *Polarizing Microscope*<sup>5</sup> equipped with a polarizer, an analyzer, a first-order red selenite plate, a cross-hair eyepiece mounted so that the hairs make a 45-deg angle with the plane of polarization, a rotatable, mechanical stage, and a microscope lamp. The possible magnification must be at least 100×.

6.2.2 *Mounting Medium*, water or clear mineral oil in a dropping bottle.

6.2.3 *Other Apparatus* as specified in 6.1.2-6.1.6 for Procedure 1.

## 7. Safety Precaution

7.1 The sodium hydroxide solution used in Procedure 1 is caustic and corrosive. Use care in its preparation and application to avoid contact with the skin or with equipment, especially the microscope objective, which may be permanently damaged if the solution is not removed immediately following contact. Clear water and a soft tissue will remove the solution.

## 8. Sampling and Preparation of Specimens

8.1 Three sources of specimens may be used with either procedure. If Suter-Webb array length groups are not available, either of the other two sources of specimens may be used.

8.1.1 *Option A—Suter-Webb Array Length Groups*—Prepare the array length groups as directed in Method D 1440. From one array discard the  $\frac{1}{16}$ -in. (1.6-mm) and  $\frac{3}{16}$ -in. (4.8-mm) length groups and any other length groups containing less than 1 mg of fibers. From each length group remaining, remove a bundle of approximately 100 fibers by lengthwise separation beginning with the longest group. Place the fibers on a microscope slide, spread them carefully to a width of 30 to 40 mm. Cover the fibers with a cover glass and apply a drop of the mounting medium to one corner. Tap the cover glass to cause the mounting medium to spread more rapidly and help prevent air bubbles. Mark the slide with the length group identification.

<sup>5</sup> Suitable microscopes are obtainable from Bausch and Lomb, Inc., 635 Paul St., Rochester, NY 14602.

<sup>6</sup> A suitable comb may be obtained from the Alfred Suter Co., 200 Fifth Ave., New York, NY 10010.

<sup>7</sup> Torsion balances meeting these requirements are obtainable from Alfred Suter Co., 200 Fifth Ave., New York, NY 10010; The Laboratory Equipment Co., Charlotte, NC; or Spinlab, Inc., 312 W. Vine Ave., Box 2018, Knoxville, TN 37902.

The series of slides shall constitute a test specimen. Have a second operator prepare a second test specimen from a second array of the sample.

NOTE 2—The sampling method described in 8.1.1 has been used for a longer period of time and given slightly more reliable results than the other sampling methods.

8.1.2 *Option B, Laboratory Blended Samples*—Take a subsample consisting of a section of sliver approximately 2 in. (50 mm) long from the blended laboratory sliver. Twist one end of the subsample, hold it firmly and place the loose ends near the edge of a microscope slide. By means of a second slide held perpendicularly, grip a few fibers, hold them lightly and pull the subsample away gently. Repeat the process until approximately 200 fibers have been extracted. Pull the fibers from the entire width of the subsample and do not purposely discard any fibers. Spread the extracted fibers and separate them as evenly as possible, keeping them nearly parallel. A dissecting needle may be used to move the fibers while holding them lightly with a second slide or a cover glass. A minimum amount of overlapping will greatly facilitate fiber classification. Cover the fibers with a cover glass and apply a drop of the mounting medium to one corner, tap the cover glass to cause the solution to spread more rapidly and help prevent air bubbles. Two technicians shall each prepare three slides in this way to secure the two specimens needed for a test.

8.1.3 *Option C, Fibrograph Beards*—Prepare beards on Fibrograph combs as directed in Test Method D 1447. Remove the beards from the two combs, divide each in half and use three of these sections as subsamples. Roll up each of the three sections leaving the combed ends free. Prepare specimens as described in 8.1.2 using the rolled-up sections of the beards as subsamples.

## 9. Preparation of Microscope

9.1 For Procedure 1, properly center and align the microscope, condenser, stage, and lighting system. Adjust the microscope to obtain a magnification of approximately 400 $\times$ .

9.2 For Procedure 2, set the analyzer and polarizer at extinction (crossed) with the selenite plate removed. Insert the

selenite plate with slow direction as indicated by the arrow on the plate, at 45 deg to the plane of polarization. Set the eyepiece so that one of the cross-hairs is parallel to the arrow of the selenite plate.

## 10. Conditioning

10.1 If the arrays for Option A have not been stored in an air-conditioned laboratory, condition them in the standard atmosphere for testing textiles for at least 4 h before preparing and weighing the specimens. Preconditioning is not necessary.

10.2 Conditioning or preconditioning is not necessary for the other sampling techniques, but a relative humidity between 55 and 70 % facilitates the mounting of fibers.

## 11. Procedure

11.1 Have each of two technicians prepare and examine a test specimen (3.1.9) in order to avoid variation associated with individual technicians.

11.2 Place a prepared slide on the stage of the microscope, locate the last fiber on one side of the mounted fibers. Move the slide to bring the fibers into view, successively classify them as mature or immature, and record on the counters the number of mature fibers and the total number of fibers on the slide.

NOTE 3—Intermediate degrees of maturity may be observed for Procedure 2 according to the information in Table 1.

11.3 For the array test specimens, examine the fibers near the middle of their lengths, but for the randomly selected specimens view the fibers about 0.25 in. (6 mm) from the end held down during extraction. Short fibers will otherwise be overlooked.

11.4 When there is difficulty in distinguishing between mature and immature fibers, proceed as directed in 11.4.1 or 11.4.2.

11.4.1 *Procedure 1*—Focus up and down or move the slide so that a different portion of the fiber is visible. Move the slide back to its original position before moving to the next fiber.

11.4.2 *Procedure 2*—Observe the extinction characteristics by removing the selenite plate and observing the appearance of the fibers against a black background.

**TABLE 2 Work Sheet—Maturity of Cotton Fibers (Array Sample)**

Test No. .... Date .....

Sample Marketing and Description: \_\_\_\_\_

(1) Length	(2) $W_1$ Weight of Fibers on Slide	(3) $N_1$ Number of Fibers on Slide	(4) $N_2$ Number of Mature Fibers on Slide	(5) $M_1$ Maturity ( $N_2/N_1$ ) × 100	(6) $W$ Weight of Com- bined Length Groups	(7) $N$ Number of Fibers in Length Group $WN_1/W_1$	(8) ( $NM_1$ )
$\frac{1}{16}$ in.	mg			percent	mg		
41							
39							
37							
35							
33							
31							
29							
27							
25							
23							
21							
19							
17							
15							
13							
11							
9							
7							
5							
Totals							

Maturity:  $M = \Sigma(NM_1)/\Sigma N = \dots$  percent

11.5 Record the data from the counters, reset the counters to zero, and examine each remaining slide.

**12. Calculation**

12.1 Recording and calculation of data are facilitated by the use of work sheets such as those shown in Tables 2 and 3.

12.2 Calculate the percentage of mature fibers as directed in the appropriate table. Figures for Table 2, column 6 (Weight of Length Group) may be taken from the array data sheet (Fig. 2, Test Method D 1440).

**13. Report**

13.1 State that the tests were made as directed in Test Method D 1442. Describe the material sampled and the method of sampling used.

13.2 Report the following information:

13.2.1 Procedure used for classification of fibers and the sampling option used, and

13.2.2 Average maturity of each sample to the nearest 1 %.

**14. Precision and Bias**

14.1 *Laboratory Data*—Although no formal interlaboratory test has been run, the following components of variance expressed as standard deviations are representative of those found in a typical laboratory using well trained operators:

Within—operator component—3.0 percentage points

Between—operator component—1.2 percentage points

14.2 *Precision*—For the component of variance reported in 14.1, two averages of observed values should be considered significantly different at the 95 % probability level if the difference equals or exceeds the critical differences listed below for various number of test specimens:

Critical Differences Percent Mature, by Procedure 1,  
Percentage Points, for the Conditions Noted<sup>A</sup>

Number of Test Specimens in Each Average	Within-Laboratory Precision
2	6.3
4	4.8
8	3.8

<sup>A</sup> Half of the test specimens in each average would be evaluated by one of a pair of operators. The following equation calculates CD, the critical difference:

$$CD = 2.77 [(S_B^2/2) + (S_W^2/n)]^{1/2}$$

where:

- $S_B$  = 1.2 %, the between-operator component,
- $S_W$  = 3.0 %, the within-operator component,
- $n$  = total test specimens in each average,
- 2 = number of operators used, and
- 2.77 = square root of 2 multiplied by 1.96, the value of Student's  $t$  for two-sided limits, infinite degrees of freedom, and a 95 % probability level.

NOTE 4—The tabulated values of the critical differences should be considered to be a general statement. Before a meaningful statement can

**TABLE 3 Work Sheet—Cotton Fiber Maturity (Random Sample Method)**

 Test No. .... Date received .....  
 Sample Marketing and Description: .....

Sample Identification	Fibers	Observations						Average Percent Maturity
		1st Technician			2nd Technician			
		1	2	3	1	2	3	
	Total number							
	Number mature							
	Percent mature							
	Technician	Avg			Avg			
	Total number							
	Number mature							
	Percent mature							
	Technician	Avg			Avg			
	Total number							
	Number mature							
	Percent mature							
	Technician	Avg			Avg			
	Total number							
	Number mature							
	Percent mature							
	Technician	Avg			Avg			
	Total number							
	Number mature							
	Percent mature							
	Technician	Avg			Avg			

Calculated by ..... Checked by .....

be made about two specific laboratories, the amount of statistical bias, if any, between them must be established, with each comparison being based on recent data obtained from randomly drawn subsamples of at least one material to be tested.

14.3 *Bias*—Procedure 1 and sampling Option A of Test Method D 1442 is the procedure by which the accuracy of other methods is judged (see 5.3).

14.3.1 Observations made on specimens obtained by either Option A or B agree very closely except for tests on immature cottons. Test results based on sampling very immature cottons

using Option A (array length groups) may be three to five percentage points lower than the test results on specimens sampled by Option B (laboratory blended samples).<sup>8</sup>

## 15. Keywords

15.1 cotton; maturity

<sup>8</sup> This method formerly appeared as part of the Standard Method of Test for Fiber Weight per Unit Length and Maturity of Cotton Fibers (Array Method) (ASTM Method D 1442), 1958 *Book of ASTM Standards*, Part 10.

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