



Designation: D6500 – 00 (Reapproved 2012)<sup>ε1</sup>

## Standard Test Method for Diameter of Wool and Other Animal Fibers Using an Optical Fiber Diameter Analyser<sup>1</sup>

This standard is issued under the fixed designation D6500; 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.

<sup>ε1</sup> NOTE—The terminology section was updated in July 2012.

### INTRODUCTION

Instruments based on image analysis have been designed to reduce the level of operator skill required and to speed up the measurement process while concurrently maintaining acceptable levels of precision and accuracy. An additional advantage of image analysis instruments is the ability of the operator to see the measurement points and to audit the process, if required, though this is not carried out during routine measurement. As with projection microscope measurements, and the Sirolan-Laserscan, the Optical Fiber Diameter Analyser (OFDA) system covered by this test method provides a count of readings grouped into diameter classes. Because the fiber snippets are measured automatically by an optical and image processing system, controls are provided in the image processing software to minimize the inclusion of multiple measurements on the same fiber and false diameter readings that arise from non-fiber material.

### 1. Scope

1.1 This test method covers a procedure that uses an Optical Fiber Diameter Analyser (OFDA) for the determination of the average fiber diameter and the fiber diameter variation in wool and other animal fibers in their various forms.

NOTE 1—This test method may also be applied to other fibers having a round cross section such as some polyamides, polyesters, and glass; it may also be applied to a limited number of polyacrylics and regenerated cellulose-type fibers.

NOTE 2—In subsequent sections of this test method, the term “wool” also signifies other animal fibers where applicable.

NOTE 3—For fineness specifications of wool, wool top, mohair, mohair top, alpaca, and cashmere, refer to Specifications D3991, D3992, D2252, and Test Method D2816, respectively.

1.2 The OFDA reports average fiber diameter and standard deviation of fiber diameter in micrometer units ( $\mu\text{m}$ ). The coefficient of variation of fiber diameter is reported as a percentage.

1.3 *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:<sup>2</sup>

- D123 Terminology Relating to Textiles
- D584 Test Method for Wool Content of Raw Wool—Laboratory Scale
- D1060 Practice for Core Sampling of Raw Wool in Packages for Determination of Percentage of Clean Wool Fiber Present
- D1776 Practice for Conditioning and Testing Textiles
- D2130 Test Method for Diameter of Wool and Other Animal Fibers by Microprojection
- D2252 Specification for Fineness of Types of Alpaca
- D2816 Test Method for Cashmere Coarse-Hair Content in Cashmere
- D3991 Specifications for Fineness of Wool or Mohair and Assignment of Grade
- D3992 Specifications for Fineness of Wool Top or Mohair Top and Assignment of Grade
- D4845 Terminology Relating to Wool

#### 2.2 Federal Standards:

- Official Standards of the United States for Grades of Wool, Section 31.0<sup>3</sup>

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D13 on Textiles, and is the direct responsibility of Subcommittee D13.13 on Wool and Felt.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> *Federal Register*, Vol 30, No. 161, August 20, 1965, pp. 10829-10833.

Measurement Method for Determining Grade of Wool, Section 31.204<sup>3</sup>

Official Standards of the United States for Grades of Wool Top, Section 31.1<sup>4</sup>

Measurement Method for Determining Grade of Wool Top, Section 31.301<sup>4</sup>

USDA Grade Standards for Grease Mohair and Mohair Top<sup>5</sup>

2.3 IWTO Standards:<sup>6</sup>

IWTO-8-66 Method of Determining Wool Fiber Diameter by the Projection Microscope

IWTO-12-93 Measurement of the Mean and Distribution of Fibre Diameter Using a Sirolan-Laserscan Fibre Diameter Analyser

IWTO-19-98 Determination of Wool Base and Vegetable Matter Base of Core Samples of Raw Wool

IWTO-47-98 Measurement of the Mean and Distribution of Fibre Diameter of Wool Using an Optical Fibre Diameter Analyser (OFDA)

### 3. Terminology

3.1 For all terminology related to D13.13, refer to Terminology D4845.

3.1.1 The following terms are relevant to this standard: average fiber diameter, grade, *in wool and mohair*, snippet.

3.1.2 For all other terminology related to textiles, refer to Terminology D123.

### 4. Summary of Test Method

4.1 This test method describes procedures for sampling wool in various physical forms, the reduction of the sample to small test specimens, and measurement of the diameter of a number of fibers from the test specimens using the OFDA. Snippets comprising a test specimen cut from the various forms of wool are cleaned where required, conditioned, and spread uniformly over the surface of a microscope slide. A cover slide is placed over the specimen and the slide placed on a microscope stage, that is moved under computer control. The slide is stepped through the field of view of a low-power microscope objective. At each step, the video system is instructed to capture and analyze a fiber image frame. Each diameter measurement is allocated to a diameter class and, at the completion of the slide, the class contents are statistically analysed to produce the mean and standard deviation of the fiber diameter for the specimen. Full distribution data are also available in the form of a printed histogram.

### 5. Significance and Use

5.1 This test method specifies sampling and testing procedures for the measurement of average fiber diameter and variation in diameter of animal fibers.

5.2 This test method is considered satisfactory for acceptance testing of commercial shipments of wool and other

animal fibers in raw and sliver form because current estimates of between-laboratory precision are acceptable. In cases of disagreement arising from differences in values reported by two or more laboratories when using this test method for acceptance testing, the statistical bias, if any, between the laboratories should be determined with each comparison being based on the testing of specimens randomly drawn from one sample of material of the type being evaluated. Test Method D2130 shall be used as a referee test method.

5.3 This test method may be used for determining compliance with average fiber diameter and diameter variation to assign grades when determining conformance of shipments to material specifications given in Specifications D2252, D3991, and D3992, and Test Method D2816.

5.4 The procedures for determining mean fiber diameter and standard deviation of fiber diameter provided in this test method and in IWTO Method 47-98 are in essential agreement.

### 6. Apparatus, Materials, and Reagents

6.1 *Optical Fiber Diameter Analyser*<sup>7</sup>, consisting of a transmission light microscope, fitted with a stage (motor-driven and controlled by a computer), stroboscopic illumination that is synchronised with the stage movement, and a CCD camera; an image acquisition and analysis hardware system; a means for controlling the interaction between the camera, stage motors and illumination unit; a data acquisition and processing computer, with optionally, control and reporting software; and, a video monitor, capable of displaying each image frame in real time, for audit purposes. See Fig. 1.

6.2 *Glass Microscope Slides*<sup>7</sup>, of float glass, sufficiently robust to withstand repeated handling having dimensions 70 by 70 by 2 mm. Two identical slides are taped together so that one supports the fiber samples with the other serving as a cover slide. Slides that are scratched on their inside surfaces are unsuitable as they may lead to erroneous measurements.

6.3 *Cleaning and Conditioning Apparatus and Facilities*, suitable for cleaning and drying the subsamples in accordance with Test Method D584 and conditioning them as described in Practice D1776.

6.4 *Apparatus for Snippet Preparation*, having either two parallel cutting edges between 1.8 and 2.0 mm apart (for example, guillotine<sup>7,8</sup> or snippeter<sup>9</sup>; see Figs. 2 and 3), or a cutting diameter of between 1.9 and 2.1 mm (for example, minicore<sup>7,10</sup>; see Fig. 4). A minicore consists of a cylindrical sample holder, designed for relatively large samples, in which a sample is manually packed, then compressed, and a coring head is driven pneumatically into the sample. The sample is compacted by a spring-loaded platen. Six or more minicore tubes with 2-mm diameter tips pass through perforations in the

<sup>7</sup> Available from BSC Electronics Pty, Ltd., 1A Thurso Rd., Myaree, Western Australia, 6154.

<sup>8</sup> Available from Symtech Systems and Technology, I-85 and Bryant Rd., PO Box 2627, Spartanburg, SC 29304.

<sup>9</sup> Available from CSIRO, Division of Wool Technology, PO Box 21, Belmont, VIC 3216, Australia.

<sup>10</sup> Available from the South African Wool Testing Bureau, Gomery Ave., Summerstrand, PO Box 1867, Port Elizabeth 6000, South Africa.

<sup>4</sup> *Federal Register*, Vol 33, No. 248, December 21, 1968, pp. 19073-19076.

<sup>5</sup> *Federal Register*, Vol 36, No. 129, July 3, 1971, pp. 12681-12658.

<sup>6</sup> Available from the International Wool Textile Organization, International Wool Secretariat, Commercial Development Department, Valley Drive, Ilkley, Yorkshire LS29, 8PB, England, UK.



FIG. 1 The Optical Fiber Diameter Analyser

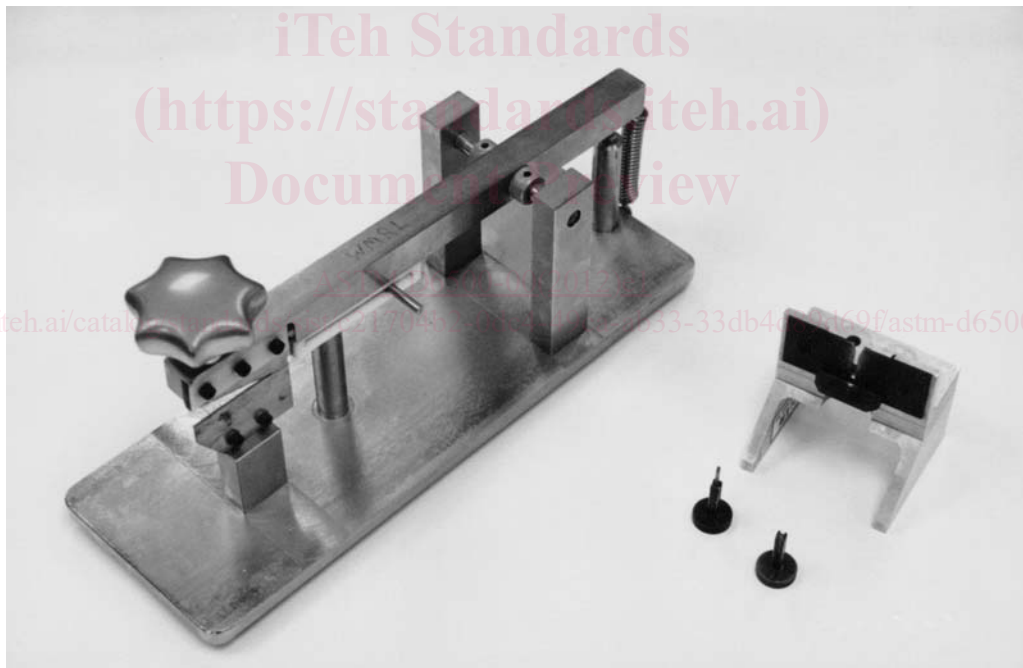


FIG. 2 Guillotine and Snippeter

platen when the force supplied by the pneumatic cylinder exceeds the force from the preloaded spring. At the end of the stroke, the cutting tips have penetrated to within 0.5 mm of the base of the sample holder. The sample collected by the minicore tubes is automatically expelled into a collection device upon retraction of the coring head.

6.5 *Heavy-Duty Sectioning Device*<sup>11</sup>, comprised of a metal plate with slot and compressing key and equipped with a

<sup>11</sup> Available from MICO Instruments, 1944 Main St., PO Box 451, Marshfield Hills, MA 02051-0451.



FIG. 3 Guillotine

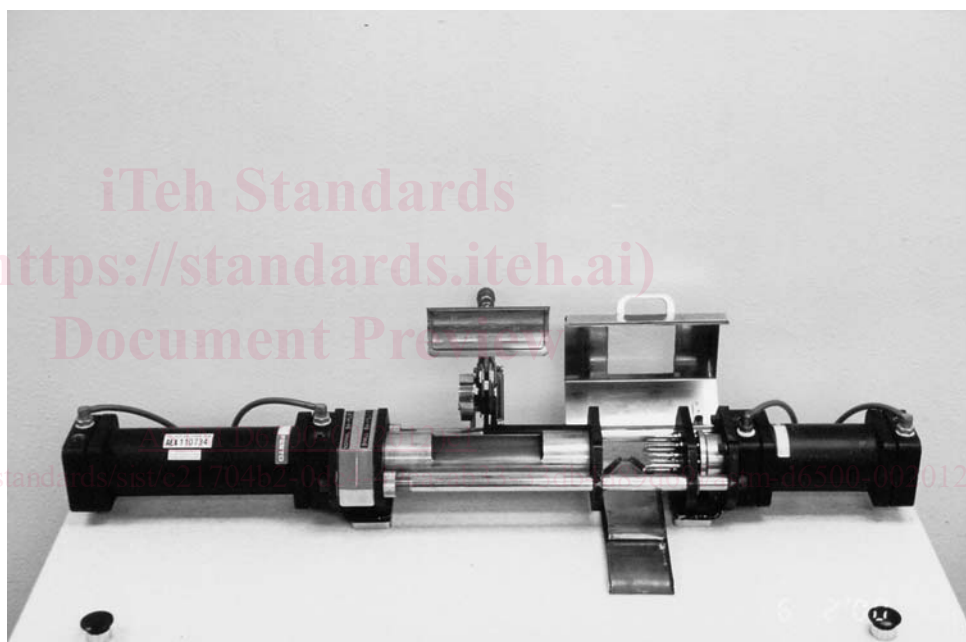


FIG. 4 Micoring Device

propulsion mechanism by which the fiber bundle may be extruded for sectioning. The instrument is designed to hold a sliver or top or equivalent bulk of fibers, yarn, or fabric (see Fig. 1 of Test Method D2130). Alternatively, this instrument can be used to generate the snippets.

6.6 *Safety Razor Blades*, single-edge or double-edge (if used with blade holder).

6.7 *Slide Preparer*<sup>7</sup> capable of uniformly spreading a portion of the cleaned, conditioned snippet sample over the surface of a clean glass slide at a predetermined, controlled density. For the OFDA, the optimum obscured areas, that is, the ratio of fiber to the total field area, is between 15 and 25 %. There are different versions of slide preparers (spreaders) available and it must be ensured that the same slide preparer is used for both calibration and routine OFDA measurements. See Fig. 5.

6.8 *Box for Compressing Loose Fibers*, 300 by 150 by 375 mm deep, inside dimensions, equipped with a floating top that has 16 randomly spaced holes 20 mm in diameter over its area. The sample may be firmly compressed by applying pressure on the top. The top is held in place by two rods extending through holes in the side of the box and over the top. The coring tube is thrust through the holes in the top to sample the wool.

6.9 *Pressure Coring Tube*, 13-mm inside-diameter metal tube, approximately 760 mm long, reamed and tapped on one end to hold a sharp 10 or 13-mm cutting tip. The tube is fitted with a “T” cross bar about 500 mm long.

6.10 *Core Extruder*, 6-mm wood dowel or aluminum rod slightly longer than the coring tube to push the sample from tube.



FIG. 5 Slide Spreader

6.11 *Solvents*—Petroleum spirit (boiling range 40 to 70°C) and 1,1,1, trichloroethane. When the preparation method calls for the cleaning of sliver subsamples, one of these two solvents shall be used. **Warning**—Both solvents have associated hazards in terms of volatility, toxicity, and, in the case of petroleum spirit, flammability. In both cases, care should be taken in storage, handling, use, and disposal in accordance with the appropriate safety procedures. Refer to manufacturers' material safety data sheets (MSDS).

6.12 *Calibration Standards*—Used for instrument calibration. For wool, use current Interwoollabs IH Standard Tops<sup>12</sup> and for mohair, use current International Mohair Association Standard Tops<sup>13</sup>.

## 7. Sampling

7.1 *Loose Fibers*—The method of obtaining a representative sample of wool differs according to circumstances. The sampling procedures and major circumstances encountered are as follows:

7.1.1 *Lots of Packaged, Grease, Pulled, or Scoured Wool*—Take core samples as directed in Practice D1060. Clean or scour the raw wool sample as directed in Test Method D584. If a representative portion of the scoured wool core sample resulting from the test for clean wool fiber present is available, it may be used for fiber diameter determination. If core sampling is not feasible, take at random, by hand, at least 50 handfuls of wool from not less than 10 % of the packages. The aggregate mass of the sample shall be at least 1.5 kg.

7.1.2 *Major Sort*—For packaged grease wool in fleece form for which a diameter test is needed for only the major sort of the fleece, hand sample by drawing one or more handfuls of

<sup>12</sup> Available from Interwoollabs Secretariat, Boite 14 Rue de Luxembourg 19/21, 1040 Brussels, Belgium.

<sup>13</sup> Available from International Mohair Association, Mohair House, 68 The Grove, Ilkley, West Yorkshire, LS29 9PA, England, UK.

wool from the major sort portions of at least 50 fleeces taken at random from the lot. The aggregate mass of the sample shall be at least 1.5 kg.

**7.1.3 Piles of Graded or Sorted Wool**—Sample piles of graded or sorted wool by taking from random locations in the pile at least 50 handfuls of wool, the aggregate mass of which shall be at least 1.5 kg. If the wool is in fleece form and a test is needed for only the major sort, take the sample as directed in **7.1.2**.

**7.1.4 Card Sliver**—Sample the wool card sliver by drawing 10 600-mm lengths at random from the lot, preferably during the carding operation.

**7.1.5 Top**—Sample the top by drawing from each 9000 kg or fraction thereof, 4 sections of sliver, each of which shall be at least 1 m in length and taken from different balls of top selected at random. Take only one ball from any one bale or carton. For broken top, take an equivalent aggregate length of sliver at random.

## 8. Test Samples and Test Specimens, Number and Preparation

**8.1 Test Samples (One from Each Lab Sampling Unit):**

**8.1.1 Grease Wool, Pulled Wool, Scoured Wool:**

**8.1.1.1 Sub-Coring**—Randomly pack the core or hand sample (see **7.1.1**, **7.1.2**, and **7.1.3**) into a suitable container (see **6.8**) and compress to approximately 14 kPa by loading a weight of 667 N on the floating top. By means of a 10- or 13-mm tipped pressure coring tube, extract at least 5 cores to provide a test specimen of at least 20 g of scoured wool. Scour or otherwise clean the test specimen if it is grease wool or pulled wool as directed in Test Method **D584**.

**8.1.1.2 Gridding, Core Test Residue**—If the sample comprises an adequate amount of scoured wool resulting from core testing a lot for clean wool fiber present, divide the sample into 40 portions of approximately equal size. From each portion, draw at random at least 0.5 g. Mix or blend these 40 portions to form the test specimen.

**8.1.1.3 Gridding and Machine Blending**—For samples other than those specified in **8.1.1.1** and **8.1.1.2**, divide the sample into 40 portions of approximately equal size. From each portion draw at random a sufficient quantity of fiber to provide a clean test specimen of 20 g. Scour or otherwise clean the test specimen of grease or pulled wool.

**8.1.2 Card Sliver**—Strip off portions of each of the 10 600-mm lengths of sliver (see **7.1.4**). Combine these portions to form a composite sliver about 600 mm in length. This constitutes the test specimen.

**8.1.3 Top**—Each of the 4 sections of sliver comprising the sample (see **7.1.5**) constitutes a test specimen.

**8.2 Test Specimens:**

**8.2.1 Test 1 test specimen from each bulk subsample and 2 specimens from each sliver and top subsample.** Prepare approximately 25-mg test specimens by cutting enough fiber snippets to measure the diameters of at least 2000 fiber segments for each test specimen measured. Obtain snippets using a minicore (**8.2.1.1**) or guillotine (**8.2.1.2**). When re-

quired to achieve the necessary quantity of snippets, combine snippets from one sliver subsample or bulk subsample to form the test specimen.

**8.2.1.1 Minicore (Applicable to Raw Wool, Card Sliver, or Top)**—Minicore each sliver subsample or each bulk subsample, as appropriate, using cutting tips between 1.8 and 2.0 mm in diameter. If the whole sliver subsample or bulk subsample cannot fit into the minicore, divide the coring sample into approximately equal portions of a size to produce at least 2000 individual fiber measurements. Where appropriate, samples of greasy wool shall be scoured by the procedures outlined in Practice **D584** before minicoring. Snippets from tops, aqueous scoured, or carbonized wool should be solvent washed, dried, and conditioned before measuring.

**8.2.1.2 Guillotine (Applicable to Staples, Card Sliver and Top)**—Cut snippets from the subsample with a guillotine or microtome set to a length between 1.8 and 2.0 mm. Make the same number of cuts from each subsample. Do not cut snippets within 100 mm of either end of the sliver or make sequential cuts within the length of the longest fibers.

**8.2.2** Remove any large pieces of vegetable matter and excessively long fibers from the test specimens. During removal of large pieces of vegetable matter and excessively long fibers, handling of the specimen must be kept to a minimum to avoid preferential separation of fibers of differing diameter.

## 9. Calibration of OFDA

**9.1** A complete calibration and validation of the analyzer will be necessary following any of the following conditions: every 3 to 6 months, dependant upon measurement performance monitoring; after a change of Interwoollabs standard top series or a change of International Mohair Association standard top series; whenever any significant instrument hardware or software changes or adjustments are made; and, after moving the instrument.

**9.2** Calibration and verification tests for the OFDA are described in **Annex A1**.

## 10. Conditioning

**10.1** Condition the cleaned, dried samples for at least 4 h in the standard atmosphere for testing textiles,  $65 \pm 2$  % relative humidity and  $21 \pm 1^\circ\text{C}$ , as directed in Practice **D1776**.

**NOTE 4**—Experience to date has involved only sub-sampling and test specimen preparation from conditioned samples and subsamples. Subsequently, it is only necessary to store and measure the test specimens under standard conditions. Theoretically, it seems reasonable that small amounts of snippets could be cleaned and dried such that only the test specimen would need to be conditioned. This approach has not yet been fully investigated and can not, therefore, be recommended.

## 11. Procedure

**11.1** A single operator is sufficient for OFDA testing.

**11.2 Pre-Measurement Checks**—At the start of each measurement session, ensure that the instrument is set up according to the operating manual. Measure at least one test specimen from a fine top of known diameter and one from a coarse top of known diameter. During the pre-measurement check, check the microscope focus and adjust if necessary according to the