Designation: D 6466 – 99

Standard Test Method for Diameter of Wool and Other Animal Fibers By Sirolan-Laserscan Fiber Diameter Analyser[1]

This standard is issued under the fixed designation D 6466; 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 (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure, using the Sirolan-Laserscan, for the determination of the average fiber diameter and the fiber diameter variation in wool and other animal fibers in their various forms.

1.2 The values stated in SI units are to be regarded as the standard.

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:*

D 123 Terminology Relating to Textiles[2]

D 584 Test Method for Wool Content of Raw Wool—Laboratory Scale[2]

D 1060 Practice for Core Sampling of Raw Wool in Packages for Determination of Percentage of Clean Wool Fiber Present[2]

D 1776 Practice for Conditioning Textiles for Testing[2]

D 2130 Test Method for Diameter of Wool and Other Animal Fibers by Microprojection[2]

D 2252 Specification for Fineness of Types of Alpaca[2]

D 2816 Test Method for Cashmere Coarse–Hair Content in Cashmere[2]

D 3991 Specifications for Fineness of Wool or Mohair and Assignment of Grade[3]

D 3992 Specifications for Fineness of Wool Top or Mohair Top and Assignment of Grade[3]

E 126 Test Method for Inspection and Verification of Hydrometers[4]

E 1750 Guide for Use of Water Triple Point Cells[4]

2.2 *Federal Standards:*

Official Standards of the United States for Grades of Wool, Section 31.0[5]

Measurement Method for Determining Grade of Wool, Section 31.204[5]

Official Standards of the United States for Grades of Wool Top, Section 31.1[6]

Measurement Method for Determining Grade of Wool Top, Section 31.301[6]

IWTO-8 Method of Determining Wool Fiber Diameter by the Projection Microscope[7]

IWTO-12 Measurement of the Mean and Distribution of Fibre Diameter Using the Sirolan-Laserscan Fibre Diameter Analyser[7]

3. Terminology

3.1 *Definitions:*

3.1.1 *average fiber diameter*, *n*—the arithmetic width of a group of fibers.

3.1.1.1 *Discussion*—In wool and other animal fibers, all animal fibers, regardless of species, can be measured using the Sirolan-Laserscan to determine average fiber diameter.

3.1.2 *grade*, *n—in wool and mohair*, a numerical designation used in classification of fibers in their raw, semi-processed and processed forms based on average fiber diameter and variation of fiber diameter.

3.1.3 *snippet*, *n*—a wool or other animal fiber which has been cut to a specified length.

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

4. Summary of Test Method

4.1 This test method describes procedures for sampling various forms of wool, 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 Laserscan. Snippets cut from the various forms of wool are cleaned where required, and dispersed in a mixture of isopropanol and water. The suspension of snippets is transported through a measuring cell which is positioned in a beam of laser light. The reduction in intensity of the laser beam as the individual snippets pass through the beam of light, approximately 500 μm in diameter,

[1] This test method is under the jurisdiction of ASTM Committee D–13 on Textiles and is the direct responsibility of Subcommittee D13.13 on Wool and Wool Felt.

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[2] *Annual Book of ASTM Standards*, Vol 07.01.

[3] *Annual Book of ASTM Standards*, Vol 07.02.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] *Federal Register*, Vol 30, No. 161, Aug. 20, 1965, pp. 10829–10833.

[6] *Federal Register*, Vol 33, No. 248, Dec. 21, 1968, pp. 19073–19076.

[7] International Wool Textile Organization, International Wool Secretariat, Commercial Development Department, Valley Drive, Ilkley, LS298PB, England.

is sensed by a detector and transformed, using a calibration look-up table, into a diameter in micrometres. Each diameter measurement is allocated to a diameter class, and when the specified number of fibers has been measured, the class contents are statistically analysed to produce the mean and standard deviation of 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 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 the purchaser and the supplier when using this test method for acceptance testing, Test Method D 2130 shall be used as a referee method.

5.2 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 D 2252, D 2816, D 3991, and D 3992.

5.3 The procedures for determining mean fiber diameter and standard deviation of fiber diameter provided in this test method and in IWTO Method 12-93 are in essential agreement.

6. Apparatus and Materials

6.1 *Fiber Diameter Analyser*[7]—Fig. 1, consisting of the following:

6.1.1 A means of transporting fiber snippets in an isopropanol/water mixture through a laser beam.

6.1.2 A means of measuring the reduction of light intensity of the beam due to the passage of a snippet and converting this to digital form.

6.1.3 A system for discrimination against the measurement of fibers that do not properly intersect the beam and contaminants such as fiber fragments, dirt, and vegetable matter particles.

6.1.4 A computing system to transform and collate results.

6.2 *Fiber Sectioning Device*—One or more of the following:

6.2.1 *Guillotine*[8]—Fig. 2, having two parallel cutting edges between 1.8 and 2.0 mm apart.

6.2.2 *Minicore*[8]—Fig. 3, a cylindrical sample holder, designed for large samples, in which a sample is manually packed and a coring head which is driven pneumatically into the sample. The sample is compacted by a spring-loaded platen and 6 minicore tubes with 2-mm diameter tips pass through perforations in the platen when the force supplied by the pneumatic cylinder exceeds the force (300 N) 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.

6.2.3 *Heavy-Duty Sectioning Device*[9]—Fig. 4, comprised of a metal plate with slot and compressing key, and equipped with a propulsion mechanism by which the fiber bundle may be extruded for sectioning. The device is designed to hold a sliver of top or equivalent bulk of fibers.

6.3 *Box for Compressing Loose Fibers*—A box, 300 by 150 by 375 mm deep in inside dimensions, equipped with floating top which 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 obtain a sample.

[8] Sirolan-Laserscan analyser, minicorer, and guillotine obtainable from Loptex S.r.l., Via L. Leoni 20, 2210 0 COMO (Italia). Tel: 39 31 273502; Fax: 39 1 273255.

[9] Obtainable from MICO Instruments, 1944 Main St., P.O. Box 451, Marshfield Hills, MA 02051-0451.

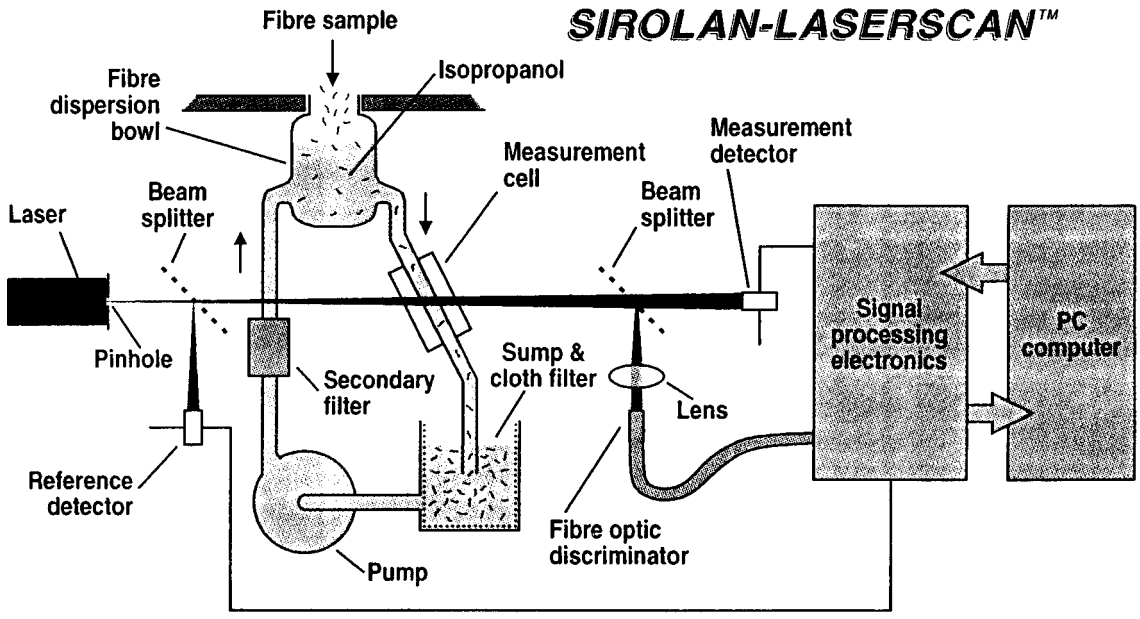

FIG. 1 Block Diagram of Laserscan System

FIG. 2 Guillotine

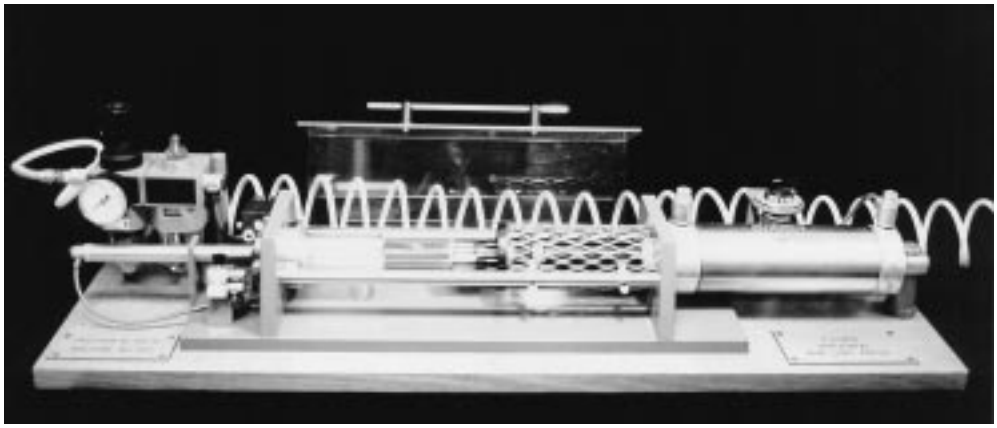

FIG. 3 Minicorer

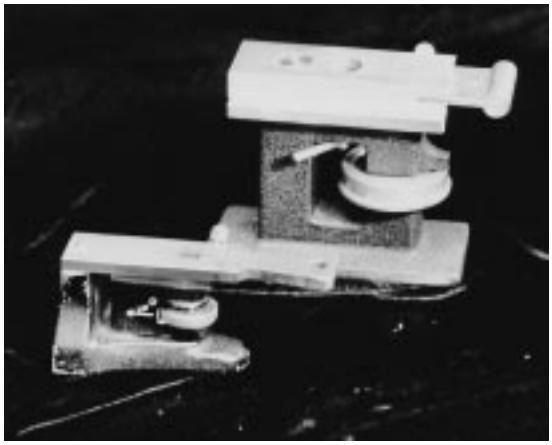

FIG. 4 Heavy Duty Sectioning Device

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

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

[10] Obtainable from Yocom-McColl Testing Laboratories, Inc., 540 West Elk Place, Denver, CO 80216 and Acro Associates, Inc., 163 Merrimac St., Woburn, MA 01801.

6.6 Apparatus for Measuring the Water Content of Isopropanol.

6.6.1 *Hydrometer*, for the range density from 0.800 to 0.900 Mg/m^3 and calibrated in accordance with Test Method E 126.

6.6.2 *Thermometer*, for the range from 0 to 50°C and calibrated in accordance with Guide E 1750.

6.7 *Calibration Standards*, for instrument calibration.

6.7.1 *Current Interwoollabs III Standard Tops*, for wood.[11]

6.7.2 *Current International Mohair Association Standard Tops*, for mohair.[12]

7. Reagents

7.1 The following reagents are used:

7.1.1 *Water*, distilled, or equivalent.

7.1.2 *Alcohol*, isopropyl ($CH_3CH_2CH_2OH$).

7.1.3 *Petroleum Spirit*, boiling range from 40 to 70°C, for cleaning sliver subsamples.

7.1.4 *1,1,1 Trichlorethane* (CH_3CCl_3), for cleaning sliver subsamples.

8. Hazards

8.1 Refer to the manufacturer's material safety data sheet (MSDS) for information on handling, storage, use, and disposal of chemicals used in this test method.

9. Sampling

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

9.1.1 *Lots of Packaged, Grease, Pulled, or Scoured Wool*—Take core samples in accordance with Practice D 1060. Clean or scour the raw wool sample in accordance with Test Method D 584. 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.

9.1.2 *Major Sort*—Packaged grease wool in fleece from which a diameter test is needed, hand sample by drawing one or more handfuls of wool from the major sort portion of at least 50 fleeces taken at random from the lot. The aggregate mass of the sample shall be at least 1.5 kg.

9.1.3 *Piles of Graded or Sorted Wool*—Sample piles of graded or sorted wool by taking from random locations in the pile at lease 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 in accordance with 9.1.2.

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

9.1.5 *Top*—Sample the top by drawing from each 9000 kg or fraction thereof, four 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.

10. Test Samples and Test Specimens, Number and Preparation

10.1 *Test Samples* (one from each laboratory sampling unit):

10.1.1 *Grease Wool, Pulled Wool, and Scoured Wool*:

10.1.1.1 *Sub-Coring*—Randomly pack the core or hand sample (see 9.1.1, 9.1.2, and 9.1.3), into a suitable container (see 6.3) and compress to approximately 14 kPa by loading a weight of 667 N on the floating top. By means of a pressure coring tube (6.4) extract at least five cores to provide a test specimen of at least 20 g of scoured wool. Scour or otherwise clean the test specimen in accordance with Test Method D 584 if it is grease wool or pulled wool.

10.1.1.2 *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 (see 9.1.1), 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.

10.1.1.3 *Gridding and Machine Blending*—For samples other than those specified in 10.1.1 and 10.1.2, 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 specimen of grease or pulled wool.

10.1.2 *Card Sliver*—Strip off portions of each of the ten 600-mm lengths of sliver (see 9.1.4). Combine these portions to form a composite sliver about 600 mm in length. This constitutes the test specimen.

10.1.3 *Top*—Each of the four sections of sliver comprising the sample (see 9.1.5) constitutes a test specimen.

10.2 *Test Specimens*:

10.2.1 Test one test specimen from each bulk subsample and two specimens from each sliver and top subsample. Prepare approximately 0.3 g test specimens by cutting enough fiber snippets to measure the diameters of 2000 fiber segments for each test specimen measured. Obtain snippets using a minicore (10.2.1.1) or guillotine (10.2.1.2). Where required to achieve the necessary quantity of snippets, combine snippets from one sliver subsample or bulk subsample to form the test specimen.

10.2.1.1 *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 give at least 2000 accepted counts. Where appropriate, samples of greasy wool shall be scoured by the procedures outlined in Test Method D 584 before minicoring. (Snippets from tops, aqueous scoured or carbonized wool require no further cleaning.)

10.2.1.2 *Guillotine (Applicable to Card Sliver and Top)*—Cut snippets from the sliver subsample with a guillotine or

[11] Available from Interwoollabs Secretariat, Boit 14, Rue du Luxembourg 19/21, 1040 Brussels, Belgium.

[12] Available from International Mohair Association, Mohair House, 68 The Grove, Ilkley, West Yorkshire, LS29 9PA, England, U.K.

microtome set to a length between 1.8 and 2.0 mm. Make the same number of cuts from each sliver 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.

NOTE 1—A cutter setting appreciably less than 1.8 mm may give biased results. Snippets longer than 2.0 mm may cause blockages in the instrument.

10.2.2 Remove any large pieces of vegetable matter and excessively long fibers from the test specimens.

NOTE 2—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.

11. Preparation and Calibration of Apparatus

11.1 *Preparation*—The operating conditions shall be as follows:

11.1.1 Set up and operate the instrument in the standard atmosphere for testing textiles.

NOTE 3—The temperature within the instrument's cabinet should be maintained to within ±0.1°C of a set value of 20°C to keep the circulating isopropanol at a constant temperature during operation.

11.1.2 Maintain the water content of the isopropanol and water mixture at 8 % (±1 %) water by volume. Check the concentration weekly using a hydrometer.

11.1.3 Set the laser current and adjust the detector position as specified in the maintenance manual. Set up the analyser for a maximum diameter measurable by the calibrated instrument greater than the maximum diameter of any fiber in the calibration tops. Adjust the analyser zero (baseline) within the specifications in the operating manual before measuring a test specimen.

11.1.4 Demonstrate that the analyser is stable by comparing measurements on test specimens from the same type sample (sliver, top) made more than 2 h apart. The results should agree within 0.5 µm for both average and standard deviation. It may be necessary to install a voltage stabilizer or an uninterruptable power supply to meet this requirement.

11.2 *Calibration*—A complete calibration and validation of the analyser will be necessary following any of the conditions outlined: every 3 to 6 months dependent on measurement performance monitoring, change of Interwoollabs top series, any significant instrument hardware changes, or adjustments and hardware maintenance or translocation of the instrument.

11.2.1 Calibrate the analyser to obtain a calibration to pass the appropriate verification tests described in Annex A1.

12. Conditioning

12.1 Precondition and condition all test specimens in accordance with Practice D 1776.

13. Procedure

13.1 A single operator is sufficient for Sirolan-Laserscan testing.

13.2 *Premeasurement Checks*—At the start of each measurement session, ensure that the instrument is set up in accordance with the operating conditions at calibration. Measure one test specimen from a fine wool top of a known diameter and one from a coarse top of known diameter. (See Note 4.) If the result for either test specimen differs by more than 0.3 µm from its assigned value, adjust the fiber diameter analyser and, if necessary, re-calibrate until subsequent test specimens of the tops give the required value.

NOTE 4—These diameters may be assigned to tops by round trials or by using the means of at least ten previous measurements on the fiber diameter analyser. The current Interwoolabs Standard Tops may be used as a further check.

13.3 *Measurement*—Ensure that the instrument settings remain stable. Feed a clean test specimen into the fiber diameter analyser. Run the analyser until 2000 measurements have been made. If the specimen isn't large enough to give 2000 measurements, discard the data and test a new specimen.

NOTE 5—The Sirolan-Laserscan instrument can operate up to a count rate of 100 counts per second. Above 100 counts per second, the instrument stops counting until the count rate falls to a satisfactory level.

NOTE 6—Excessively long fibers or any large pieces of vegetable matter may cause blockages.

13.3.1 Feed the test specimen into the wool dispersion bottle where it is dispersed in a propan-2-ol and water mixture from a header tank. The flow of propan-2-ol and water carries the fibers through a laser beam. A detector responds to the change in the laser beam caused by a snippet passing through; these changes are seen as electrical pulses which are converted to digital form. If the image simultaneously received on the fiber-optic discriminator is assessed as being a single snippet, assign the pulse a micrometre class in accordance with the transform table in use. At the end of the measurement run a frequency table for these micrometre classes, with the integer micrometre as the midpoint of the class interval, is displayed and may be printed together with calculated statistics such as the mean and standard deviation for the distribution reported to 1 decimal place, diameter distribution and coefficient of variation as a percentage to the nearest whole number. Filters remove the fibers from the propan-2-ol and water which is recirculated by a pump.

14. Report

14.1 State that the tests were made in accordance with Test Method D 6466. Describe the material sampled and the method of sampling used.

14.2 Report the following information:

14.2.1 The average fiber diameter.

14.2.2 The fiber diameter distribution.

14.2.3 The standard deviation of fiber diameter.

14.2.4 The coefficient of variation of fiber diameter.

14.2.5 The 95 % confidence limits for the sample average.

15. Precision and Bias

15.1 *Precision*—An international interlaboratory test utilizing five laboratories was conducted in 1992 under the auspices of IWTO. This led to the acceptance of IWTO-12 in 1993. Wool tops and greasy wool cores were utilized in this interlaboratory test. A set of twelve tops covering a broad range of fiber diameters were utilized in the study. The top samples were taken from a continuous 10-m length of each top. Each 10-m length of top was divided into 2 subsamples, 300 mm in length. These subsamples were randomly alloted to the participating

laboratories. Each subsample was guillotined to provide sufficient snippets to obtain a count of 2000 accepted fiber measurements. Twelve 40-g greasy wool core samples were supplied to each laboratory. Each core sample was divided into two 20-g portions, each portion minicored and cleaned. Two test specimens, of 2000 accepted diameter measurements, were taken from each of these cleaned minicore subsamples. Estimates of the components of variance and the 95 % confidence limits for wool tops are shown in Table 1. The estimates for greasy wool cores are shown in Table 2. Table 3 provides the 95 % confidence limits in 5-µm increments for aqueous scoured core samples. Similar information for other animal fibers is being generated by members of Subcommittee D13.13.

15.2 *Bias*—The procedure in Test Method D 6466 for measuring the diameter of wool fibers by Sirolan-Laserscan has no known bias or systematic error and gives results that agree closely with those obtained using the microprojection method.

TABLE 1 Components of Variance (µm²) and the 95 % Confidence Limits[A] (µm), for LASERSCAN Measurements (Guillotined Slivers)

Fibre Diameter Group	Factor	Laserscan Values
Less than 26.0 µm	variance between-laboratory	0.003
	variance within-laboratory	0.015
	95 % confidence limit (3 hanks)	±0.2 µm
26.0 µm and greater	variance between-laboratory	0.016
	variance within-laboratory	0.062
	95 % confidence limit (3 hanks)	±0.3 µm

[A]The 95 % confidence limits in Table 1 were calculated as follows:

95 % confidence limits = 1.95 $\sqrt{\sigma^2}$ within laboratory + σ^2 between laboratory

TABLE 2 Components of Variance (µm²) and the 95 % Confidence Limits[A] (µm) for Laserscan Measurements (Aqueous Scoured Core Samples)

Fibre Diameter Group	Factor	Laserscan Values
Less than 26.0 µm	variance between-laboratory	0.0142
	variance within-laboratory	0.0223
	variance total	0.0365
	95 % confidence limit	±0.37 µm
26.0 µm and greater	variance between-laboratory	0.0283
	variance within-laboratory	0.0406
	variance total	0.0689
	95 % confidence limit	±0.51 µm

[A]The 95 % confidence limits in Table 2 were calculated as follows:

95 % confidence limits = 1.96$\sqrt{\sigma^2}$ within laboratory + σ^2 between laboratory

TABLE 3 95 % Confidence Limits for LASERSCAN Measurements in 5-µm Increments (Aqueous Scoured Core Samples)

Mean Fiber Diameter, µm	Laserscan 95 % C.L., µm
15.0	±0.24
20.0	±0.30
25.0	±0.37
30.0	±0.43
35.0	±0.50
40.0	±0.56

16. Keywords

16.1 animal fibers; diameter; wool fiber

ANNEX

(Mandatory Information)

A1. CALIBRATION OF THE SIROLAN-LASERSCAN

A1.1 This procedure is the one used by the Sirolan-Laserscan software. It is used in conjunction with the current Interwoollabs IH Standard Wool Tops and International Mohair Association Standard Tops. This allows the calibration to be used to measure individual fibers up to 80 µm. This limit has been shown to give acceptable results even though it is an extrapolation beyond the highest values usually found in calibration tops.

NOTE A1.1—A second range up to 160 µm is available on Sirolan-Laserscan. The second range (from 0 to 160 µm) is to be used when calibrating with mohair tops.

A1.1.1 Measure test specimens from at least eight, preferably twelve, current Interwoollabs IH Standard Tops, which have known Projection Microscope (PM) average diameters on the analyser and obtain the frequency histogram using a linear transform. Calculate the average values separately for the Interwoollabs tops from the analyser readings and regress this data against the PM values for the tops to determine the coefficients α, β, and ϕ of Eq A1.1.

$$DIA = \alpha + \beta\,(FDA)^{\phi} \qquad (A1.1)$$

where:

FDA = Sirolan-Laserscan measurements, and
DIA = assigned values of the tops.

A1.1.2 Use Eq A1.1 to recalculate the values of the class midpoints in the measured histograms and recalculate the average values for each top. Regress these new values against the assigned PM values and obtain a new equation of the same form. Use this resulting equation to calculate a table to enable readings on fibers from the analyser to be assigned to 1-µm class intervals.

A1.2 The set up the Instrument governs the maximum diameter fiber that will be measured by the instrument. Adjust the set-up values to those in the manual. To check whether these values give the required range, follow the calibration procedure. For this check, measure only 2000 snippets of each calibration top material. If the maximum ADC value is reached before the greatest value of diameter present in the calibration

tops, adjust the analyser. If the ADC value is not reached before the maximum micrometre class (usually 80) allowed by the software, adjust the analyser until near the maximum ADC value to obtain the best resolution.

NOTE A1.2—Fibers outside the 0 to 80 range are counted and automatically recorded in the "over bin" of the Sirolan-Laserscan. In such cases the results are likely to be inaccurate.

A1.2.1 When testing mohair, calibrate the analyser using International Mohair Association Calibration Tops instead of the wool calibration tops.

A1.3 *Preparation of Snippets*—Prepare snippets from the current series Interwoollabs III Standard Calibration Tops in accordance with 10.2.1. It is recommended that the total available length of each standard top be separated into six equal lengths and a cut taken from each length to make the test specimens for calibration. This ensures a greater number of fibers sampled and reduces any problems of short-term variation that may occur in tops.

A1.4 *Measurement of Calibration Tops*—Ensure that the instrument has been allowed sufficient time to stabilize and that the checks and adjustments required by the instrument manuals have been carried out. A total of 10 000 accepted fiber measurements should be obtained for each of the calibration tops using the "linear range."

A1.5 *Calculation*—An iterative procedure is used to calculate the coefficients in Eq A1.1. The differences between the calculated and assigned mean values of the calibration tops should have a mean square error of less than 0.1 µm. Use the resulting equation to calculate a calibration table which can be used by the computer to assign the pulse height values (in analogue to digital converter units) of each fiber to 1-µm class intervals. In software, the class boundaries are calculated with the exact micrometre value as the class midpoint, for example, for the 20-µm class all fibers greater than or equal to 19.5 µm and less than 20.5 µm will be included.

A1.6 *Verification of the Calibration*:

A1.6.1 *Analyser Settings*—On completion of the calibration, check the calibration table to ensure that the first occurrence of the maximum analogue-to-digital converter unit corresponds to a fiber diameter greater than the highest fiber diameter declared in any of the projection microscope distributions. If this is not the case, adjust the analyser ted and repeat the calibration.

A1.6.2 *Verification with Wool Tops*—Measure a test specimen from each of eight wool tops with known Projection Microscope (PM) mean and standard deviation values. These may be eight earlier Interwoollabs standard reference tops.

NOTE A1.3—The first set of Interwoollabs tops which has these data was the Series 10 Airflow Calibration tops which were first issued in 1993.

A1.6.3 *Mean Fiber Diameter*—For each top, calculate the difference, d_i, between the measured mean and the PM mean, m_i, assigned by Interwoollabs. Calculate the mean difference, the variance of the difference and the *t* value, using Eq A1.2-A1.4.

$$d = \frac{\Sigma d_i}{n} \tag{A1.2}$$

$$s_d^2 = \frac{\Sigma d_i^2 - \frac{(\Sigma d_i)^2}{n}}{n-1} \tag{A1.3}$$

$$t \text{ value } = \frac{d\sqrt{n}}{S_d} \tag{A1.4}$$

where:
n = number of reference tops,
d = mean difference, and
S_d^2 = variance of the difference.

A1.6.3.1 Calculate the gradient of the regression of the differences against the Interwoollabs PM means. To test whether the gradient is significantly different from zero, calculate a new mean difference, variance of difference and *t* value, using Eq A1.5-A1.7.

$$g = \frac{n\Sigma d_i m_i - \Sigma d_i \Sigma m_i}{n\Sigma m_i^2 - (\Sigma m_i)^2} \tag{A1.5}$$

$$s_g^2 = \frac{\frac{n\Sigma d_i^2 - (\Sigma d_i)^2}{n\Sigma m_i^2 - (\Sigma m_i)^2} - g^2}{n-2} \tag{A1.6}$$

$$t \text{ value } = \frac{g}{S_g} \tag{A1.7}$$

where:
m_i = Interwoollabs PM mean value for the ith top,
S_g = gradient, and
S^2_g = variance of gradient.

NOTE A1.4—An acceptable alternative to the statistical significance of the *T*-test is an *F*-test (*F* value is the square root of the *t* value).

A1.6.3.2 The calibration is deemed to be unsatisfactory if either: (*1*) the absolute difference is greater than 0.1 µm and the *t* value is significant at the 5 % level for $n - 1$ df, or (*2*) the absolute value of the gradient of the regression is greater than 0.004 and the *t* value is significant at the 5 % level of $n - 2$ df. If the calibration is unsatisfactory, the analyser must be adjusted and recalibrated until it passes the preceding tests, if unable to pass the tests, the machine must be checked by the distributor.

NOTE A1.5—The analyser software (Version 2) has a menu option which performs the verification procedure previously described.

A1.6.4 *Standard Deviation (SD) of Fiber Diameter*—The verification procedure for SD is dependent upon valid SD data being available and at this stage is not mandatory. The verification procedure used for the mean value as described in A1.6.3 can also be applied to SD values. The differences should be calculated against the Interwoollabs Standard Deviation (SD) PM values. A gradient limit of 0.01 can be used. If verification of the SD values fails, this may be improved by repeating the calibration and verification procedure.

NOTE A1.6—The analyser has a software menu option which conducts the verification procedure in the outlined manner for additional information only.