INTERNATIONAL STANDARD

ISO 1136

Second edition
2015-03-01

Wool — Determination of mean diameter of fibres — Air permeability method

Laine — Détermination du diamètre moyen des fibres — Méthode perméamétrique

Reference number
ISO 1136:2015(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT), see the following URL: Foreword — Supplementary information.

The committee responsible for this document is ISO/TC 38, *Textiles*, Subcommittee SC 23, *Fibres and yarns*.

This second edition cancels and replaces the first edition (ISO 1136:1976), which has been technically revised.

Introduction

When a current of air is passed through a uniformly-arranged mass of fibres packed in a chamber with perforated ends, the ratio of air flow to differential pressure is uniquely determined by the total surface area of the fibres, and by various constants. This was predicted from the hydrodynamic equations of Kozeny and others.

For fibres of circular or near-circular cross-section and constant density, such as non-medullated wool, the surface area of a given mass of fibres is proportional to the average fibre diameter. This principle can be utilized to construct apparatus giving an estimate of fibre diameter. Because of its speed and simplicity, the method is particularly suitable for quality control in mill testing laboratories.

Since the method is indirect, the apparatus is first calibrated from wools of known fibre diameter. For this purpose, eight reference slivers have been provided (see Annex E).

It has been shown that the estimate of fibre diameter actually given by the permeability method is $d(1+c^2)$, where d is the average fibre diameter (length biased) measured by the projection microscope, and c is the fractional coefficient of variation. Since c normally lies within comparatively small limits for unblended slivers, it is usual, however, to calibrate the apparatus directly in terms of d.

The method requires that the fibres be reasonably clean and dispersed in a uniform open state, such as card slivers or combed slivers. It is thus unsuitable for raw wool unless first scoured and carded. Some types of wool need special calibrations as described in Annex D.

The preparation of test specimens for measurement is identical with that used for calibration specimens.

This second edition to ISO 1136 is based on the test method IWTO-6-98, drawn up by the International Wool Textile Organization (IWTO).

Wool — Determination of mean diameter of fibres — Air permeability method

1 Scope

This International Standard specifies a method for the determination of the mean diameter of wool fibres, using an apparatus which passes a current of air through a bundle of fibres.

This International Standard is applicable to clean, unmedullated wool fibres dispersed in a uniform, open state. It provides a method particularly suitable for combed slivers. The dichloromethane extractable matter content of the specimen must not exceed 1,0 %. It is applicable to oil-combed slivers after cleaning with an organic solvent.

The method described in this International Standard is less accurate for lambswool and for wool which is appreciably medullated (see Annex D) and heavily dyed wool.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 139, *Textiles — Standard atmospheres for conditioning and testing*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
laboratory sample
conditioned sample of fibres, representative of the bulk, from which the test specimens are weighed out

Note 1 to entry: In many cases, the laboratory sample will consist of one or more short lengths of sliver.

3.2
test specimen
weighed amount of fibre which is packed into the constant volume chamber

4 Principle

A specified mass of fibres to be tested is compressed to a constant volume in a cylindrical chamber with perforated ends to which a flowmeter and a manometer are connected.

The fibres are packed in such a way that they lie predominantly at right angles to the long axis of the chamber. A regulated current of air is then passed through the compressed fibres and the average fibre diameter read off from a scale on the manometer or the flowmeter.

5 Apparatus

5.1 Forms of apparatus

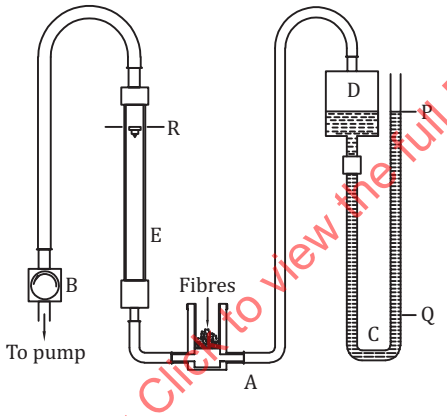
Two alternative forms of apparatus are described: “constant flow” and “constant pressure”. Both forms of apparatus have the same arrangement of parts, as illustrated in Figure 1.

The constant flow apparatus utilizes a specimen mass of 1,5 g; the flowmeter is adjusted to a fixed value and the fibre diameter is read off from the manometer scale. This scale is not linear since the successive intervals, corresponding to 1 µm, decrease with the diameter.

The constant pressure apparatus utilizes a specimen mass of 2,5 g; the manometer is adjusted to a fixed pressure and the fibre diameter is read off from the flowmeter. The constant pressure apparatus gives a nearly linear scale in micrometres. Since less accuracy in weighing the specimen is required, this method has some advantages for mill use.

5.2 Detailed parts

The apparatus consists of the following parts arranged as shown in Figure 1.



Key

A	constant volume chamber
B	air valve
C	manometer
D	reservoir
E	flowmeter
P, Q, R	reference marks

Figure 1 — General arrangement of apparatus

5.2.1 Air valve (B), giving sufficiently fine control of the air supply, such that the lever of the flowmeter or manometer can be quickly adjusted to the working value.

5.2.2 Suction pump, of a type providing a smooth output of at least 30 l/min at 200 mmH_2O with minimal fluctuation of the float of the flowmeter.

A filter to trap any loose fibres may be inserted between the pump and the air valve (B).

NOTE 1 mmH_2O=9 806 65 Pa=9 806 65 N/m^2

5.2.3 Constant volume chamber (A), of brass, hardened steel, or any other suitable metal, comprising the three following parts: the base into which the fibres are packed, the plunger which compresses the fibres, and the screw cap which clamps the plunger to the base.

The finish shall be smooth so that the plunger slides easily into the base without trapping fibres. Suggested dimensions of the constituent elements of the chamber are given in Figure 2.

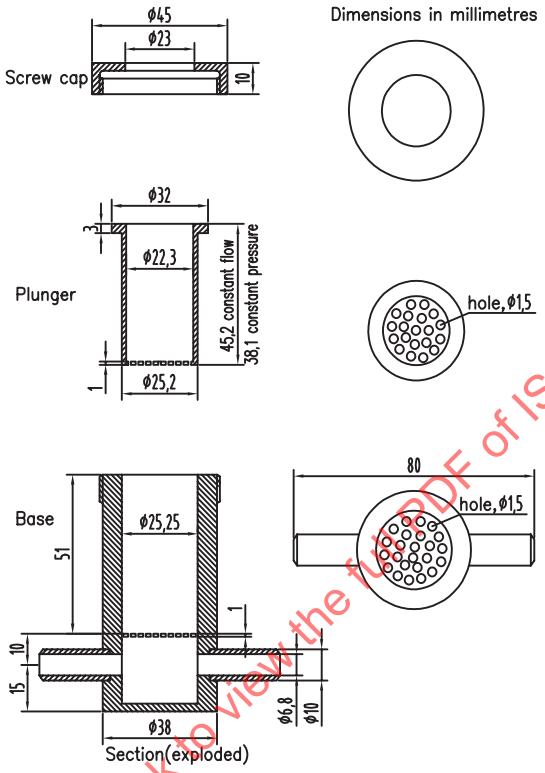


Figure 2 — Suggested dimensions of constant volume chamber (A)

Important dimensions are 22,3–25,2–25,25–42,5 and 38,1 mm.

5.2.4 Manometer (C), made of glass tubing of internal diameter at least 5 mm to reduce surface tension effects.

In both cases, a small amount of dye may be added to the manometer fluid, and where this consists of distilled water, a small trace of chromic acid should be added to give a clear meniscus. A millimetre scale is fixed behind the open limb as described in A.3.1.

5.2.5 Reservoir (D) of the fluid manometer (5.2.4), having the characteristics specified in the following table, and mounted at a sufficient height to give a clear working distance PQ of 350 mm in the open limb of the manometer.

Table 1 — Manometer and flowmeter characteristics

Characteristic	Constant flow	Constant pressure
Minimum diameter of reservoir	150 mm	60 mm
Type of manometer fluid	n-Propyl alcohol	Distilled water
Working range of flowmeter	10 l/min to 20 l/min	5 l/min to 25 l/min

5.2.6 Flowmeter (E), having the characteristics indicated in Table 1.

5.2.7 Rubber tube, connecting the manometer reservoir (D) to the chamber (A), consisting of pressure tubing of small internal diameter to avoid constriction at the bends.

5.2.8 Rubber or plastic tube from the chamber (A) to the flowmeter (E), of internal diameter not less than 6 mm.

The tube shall be as short as possible and shall not be twisted or kinked between calibration of the apparatus and its subsequent use.

5.2.9 Balance, capable of weighing the specimen to an accuracy of ±2 mg for the constant flow method and of ±4 mg for the constant pressure method.

6 Conditioning and testing atmosphere

6.1 The test specimens shall be dried sufficiently and brought to equilibrium and tested in one of the standard atmospheres for testing specified in ISO 139.

NOTE The laboratory sample can be dried in an oven with forced draft circulation or a rapid dryer at between 50 °C to 107 °C. The time required needs to be determined for the specific laboratory situation.

Each laboratory is to carry out investigations on the rate of equilibration, for its particular conditioning system, of wool samples prepared in its specific equipment so that the appropriate conditioning time can be established.

6.2 The tested specimen shall be weighed in the standard atmospheres at the level of accuracy specified in the method.

6.3 If tests are not carried out in the standard atmosphere for testing, the laboratory sample shall be conditioned to equilibrium near the apparatus and the relative humidity of the atmosphere at the time of test noted. The final results shall be corrected by the factors given in Annex C.

NOTE A source of error might occur if the moisture of the specimen changes during test. This could happen if the laboratory sample is allowed insufficient time to attain moisture equilibrium with the testing atmosphere. The minimum time required to ensure conditioning to equilibrium of a length of sliver in an opened-out state in a well-ventilated room is about 60 min.

7 Preparation of test specimens

7.1 Unopened sliver

7.1.1 Cleaning

In general, the laboratory sample shall have a mass of about 8 g and shall first be degreased by rinsing well in two baths each of about 200 ml of petroleum ether before conditioning.

7.1.2 Number of specimens

Unless otherwise specified, test a minimum of two specimens for fibre diameter below 30 µm and a minimum of three specimens for fibre diameter above 30 µm.

7.1.3 Selection of specimens

The specimens shall be taken from different places in the laboratory sample. In the case of balls of sliver, the laboratory sample shall be made up of pieces of sliver from both inside and outside the ball.

7.1.4 Specimen mass

For the constant flow method, the specimen mass shall be 1,5 g ± 0,002 g. For the constant pressure method, the specimen mass shall be 2,5 g ± 0,004 g.

7.1.5 Preparation

For slivers with cut ends, the specimen shall be prepared by cutting off with scissors a length to give as nearly as possible the specimen mass and then making up to the exact mass by adding shorter cut lengths or portions.

For slivers with pulled ends, about five hand draws shall be removed and discarded and the specimens weighed out by taking several successive hand draws.

These two methods of sampling give the same results if carried out properly.

7.2 Opened sliver

7.2.1 Cleaning

The laboratory sample should weigh not less than 10 g, and if it is known to have an oil content not exceeding 1,0 %, the test specimen may be taken from it without cleaning. Otherwise, the sample should first be degreased by rinsing it well in two baths each of about 200 ml of petroleum ether before conditioning.

7.2.2 Preparation

Take from the sample 10 g to 20 g of sliver and deparallelize using a Shirley Analyser or another method to give the laboratory sample.

Pre-condition (see 6.1) and condition the laboratory sample.

For the Shirley Analyser, cut the sliver into lengths of 15 mm to 20 mm before deparallelizing.

Other methods may refer to Shirley Analyser. Laboratories can, according to their own conditions, develop their own method.

7.2.3 Number of specimens

Unless otherwise specified, test a minimum of two specimens, and measurements per test specimen two times.

7.2.4 Selection of specimens

Passing the cut sliver through a Shirley Analyser or other methods thoroughly blends the fibres. Test specimens need not, therefore, be made up from pinches of fibre from different parts of the prepared laboratory sample.

7.2.5 Specimen mass

For the constant flow method, the specimen mass shall be 1,5 ± 0,002 g. For the constant pressure method, the specimen mass shall be 2,5 g ± 0,004 g.

8 Procedure

8.1 Unopened sliver

8.1.1 Ensure that the meniscus of the manometer (5.2.4) is at the zero mark and, if required, carry out an orifice plate check as detailed in A.3.3.

8.1.2 Pull out the weighed test specimen into a long thin sliver and feed it evenly into the constant volume chamber (5.2.3), packing the fibres down with a smooth rod from time to time. Insert the plunger and screw down the cap to the furthermost extent so that the lip of the plunger is in contact with the base.

8.1.3 Depending on the method to be used, adjust the air valve (5.2.1) as follows:

a) for the constant flow method, adjust the air valve until the top of the float of the flowmeter (5.2.6) coincides with the reference mark R and note the fluid level of the manometer (5.2.4) to the nearest 1 mm or 0,1 µm (see A.3.1);

b) for the constant pressure method, adjust the air valve until the fluid level of the manometer coincides with the 180 mm reference mark P and note the position of the float of the flowmeter to the nearest 1 mm or 0,1 µm (see A.3.2).

8.1.4 Remove the specimen from the constant volume chamber (5.2.3), tease out the fibres by hand, repack in the constant volume chamber without loss of fibre, insert the plunger, and screw down the cap.

8.1.5 Repeat the operation specified in 8.1.4 so that a total of three readings on each test specimen is obtained.

8.2 Opened sliver

8.2.1 Ensure that the meniscus of the manometer (5.2.4) is at the zero mark and, if required, carry out an orifice plate check as detailed in A.3.3.

8.2.2 Pack the specimen evenly into the cylindrical base, a small amount at a time, using forceps, not fingers, to handle the specimen, so as not to contaminate or change the moisture regain of the specimen. Push the fibre into the cylindrical base preferably using the short end of the packing rod and taking particular care to ensure that the fibres are uniformly packed and that the walls and bottom of the chamber are not scratched or marked.

8.2.3 Insert and push down the perforated plunger into the cylindrical base. Secure the plunger cap without rotation of the perforated plunger, ensuring no fibres are trapped between the perforated plunger and the chamber and that the shoulder of the plunger rests firmly on the lip of the chamber.

8.2.4 Depending on the method to be used, adjust the air valve (5.2.1) as follows:

a) for the constant flow method, adjust the air valve until the top of the float of the flowmeter (5.2.6) coincides with the reference mark R and note the fluid level of the manometer (5.2.4) to the nearest 1 mm or 0,1 µm (see A.3.1);

b) for the constant pressure method, adjust the air valve until the fluid level of the manometer coincides with the 180 mm reference mark P and note the position of the float of the flowmeter to the nearest 1 mm or 0,1 µm (see A.3.2).

8.2.5 Take the specimen out of the chamber and repack it in reverse direction without teasing it out. Use forceps, not fingers, to handle the specimen, so as not to contaminate or change the moisture regain

of the specimen. Repeat the procedure described in 8.2.1 to 8.2.5 on at least one further test specimen, thus obtaining a total of at least 4 readings.

If using one airflow fineness meter:

Measure 2 test specimens. If the range of the 4 readings is greater than that in Table 2, measure one more test specimen. If the range of the 6 readings so obtained is greater than that shown in Table 2, repeat the test on 3 additional test specimens.

Table 2 — Readings from one airflow fineness meter

Mean fibre diameter (µm)	**Range**	
	Number of test specimens (Reading)	
	2(4)	3(6)
Less than 26 µm	0,5 µm	0,6 µm
26 µm or greater	0,8 µm	0,9 µm

If using two airflow fineness meters:

Measure 2 test specimens (one specimen in each airflow apparatus). If the range of the 4 readings is greater than that in Table 3, measure 2 more specimens (one specimen in each apparatus).

If the range of the 8 readings so obtained is greater than that shown in Table 3, measure an additional 2 specimens (one specimen in each apparatus).

Table 3 — Readings from two airflow fineness meters

Mean fibre diameter (µm)	**Range**	
	Number of test specimens (Reading)	
	2(4)	3(6)
Less than 26 µm	0,7 µm	0,8 µm
26 µm or greater	0,9 µm	1,1 µm

9 Expression of results

Calculate the average of the three readings for each specimen and express the result to the nearest 0,1 µm.

10 Test report

The test report shall include the following particulars:

a) a reference to this International Standard, i.e. ISO 1136;

b) the method used (constant flow or constant pressure);

c) the results obtained in accordance with Clause 9;

d) whether the sample was tested after cleaning in petroleum ether or without cleaning;

e) the relative humidity and temperature of the conditioning and testing atmospheres, and whether the result has been corrected for the relative humidity;

f) all operating conditions not specified in this International Standard, as well as any incidents that might have influenced the results.

Annex A
(informative)

Calibration of apparatus

A.1 Leakage test

After assembling the apparatus, as seen in Figure 1, remove the cap and plunger from the constant volume chamber (A) and insert a rubber stopper. By means of a Hoffmann Clip, close the rubber tube between (A) and (E) after introducing a pressure difference causing the level of the meniscus in the manometer to alter by about 150 mm. Note the position of the meniscus periodically for several minutes; if it changes, examine the apparatus for leaks.

A.2 Samples of slivers

Obtain sufficient quantities of the reference slivers (see Annex E) for calibration. In requesting these, state

a) the test specimen mass for the apparatus to be used (1,5 or 2,5 g), and

b) whether oil-combed or dry-combed samples are required.

Sufficient numbers of each type of sliver are supplied for four specimens.

A.3 Graduating the scale

A.3.1 Constant flow apparatus

Make a horizontal mark R (see Figure 1) near the top of the flowmeter scale, avoiding any position giving marked fluctuation of the float. Fix a scale graduated in millimetres behind the manometer and adjust the zero mark to coincide with the meniscus of the liquid. Then condition and weigh out, according to the procedure specified in Clauses 6 and 7, 1,5 g specimens of each sample of reference sliver and test according to the procedure specified in Clause 8, noting the distance in millimetres below the zero to which the meniscus falls.

Do not clean the sliver before test. Test five specimens from each of the eight reference slivers in this way and calculate the average of the nine readings for each reference sliver.

Plot the average depression h, in millimetres, of the manometer meniscus against the known value of fibre diameter d, in micrometres, and, after inspection to ensure that the points lie about a smooth curve, fit a relation by least squares as given below. From this relation, a conversion table may be prepared, in micrometres, or a scale may be graduated in micrometres and fixed behind the manometer.

Adjustment of results by the least squares method

The relation between d and h is of the form hd^b = constant, and it is thus necessary to take logarithms to obtain a linear relation.

Let $X = \log d$ and $Y = \log h$.

For each of the n lots of sliver used for standardization, two values (X_1 and X_2, X_2 and Y_2, etc.) are obtained.

First, calculate the following quantities:

$$\sum X = X_1 + X_2 + \cdots + X_n$$

$$\sum Y = Y_1 + Y_2 + \cdots + Y_n$$

$$\sum Y^2 = {Y_1}^2 + {Y_2}^2 + \cdots + {Y_n}^2$$

$$\sum XY = X_1Y_1 + X_2Y_2 + \cdots + X_nY_n$$

$$\sum y^2 = \sum Y^2 - \frac{\left(\sum Y\right)^2}{n}$$

$$\sum xy = \sum XY - \frac{\sum X \sum Y}{n}$$

$$b = \frac{\sum xy}{\sum y^2}$$

The regression equation of X and Y which applies to the apparatus is then

$$X = \frac{\sum X}{n} + b\left(Y - \frac{\sum Y}{n}\right) \tag{A.1}$$

Finally, construct a table relating h to d by taking values of h at 5 mm intervals, finding log h, substituting in Formula (A.1) to obtain X and so tabulating d = antilog X for each value of h.

A.3.2 Constant pressure apparatus

Make a horizontal mark at a distance corresponding to 180 mm water pressure from the zero mark Q of the manometer. Fix a scale graduated in millimetres behind the flowmeter (E) so that the zero of this scale coincides with a file mark (Zero) made near the bottom of the flowmeter.

Condition and weigh out 2,5 g specimens of each sample of reference sliver according to the procedure specified in Clause 6 and Clause 7, and test according to the procedure specified in Clause 8, noting the distance y, in millimetres, of the float of the flowmeter from zero. Do not clean the slivers before test. Test five specimens from each of eight reference slivers in this way and calculate the average of the nine readings for each reference sliver.

Plot the average reading, in millimetres, y_1, y_2, etc., against the known values of fibre diameter d_1, d_2, etc. The result will be a nearly linear relation; fit a second degree regression line of y on d. This is done by finding the coefficients a, b, c, in Formula (A.2)

$$y = a + bd + cd^2 \tag{A.2}$$

by solving the equations

$$\sum y = 8a + b\sum d + c\sum d^2$$

$$\sum dy = a\sum d + b\sum d^2 + c\sum d^3$$

$$\sum d^2 y = a\sum d^2 + b\sum d^3 + c\sum d^4$$

Formula (A.2) is then used to graduate a scale, in micrometres, which may be fixed behind the flowmeter.

A.3.3 Orifice plate checks

To make regular daily checks that the apparatus is in good order, the use of the two orifice plates is recommended. These consist of aluminium disks of the same diameter as the inside of the constant volume chamber, each with a central hole. The disks have a rim which in use rests on the annular top of

the constant volume chamber. The diameter of the central hole in one disk is chosen to give a reading of about one-third of the available scale on the manometer (constant flow method) or flowmeter (constant pressure method) when clamped and used in the apparatus under working conditions, with no fibres in the chamber. The diameter of the central hole in the second disk is chosen to give a reading of about two-thirds of the available scale under the conditions described above.

At least once a day, clamp the orifice plates in the apparatus so that air enters through the central hole only and note the readings. Variations in the readings given by the scale shall not exceed 2 mm and 4 mm respectively for the two orifice plates. This provides a useful and quick check on the functioning of the apparatus, particularly as regards the presence of air bubbles in the manometer system.

Annex B
(informative)

Reproducibility of results

It is desirable that the persons to whom a test result is communicated should have some idea of the appropriate confidence limits of each average reading reported. Confidence limits will depend amongst other things on the number of tests, the variability of the material, experimental error, differences between apparatus, and the probability level assumed. There are two important cases about which information is available at present. This information, which is summarized below, should be regarded as illustrative and as applying only to the particular material tested. The original papers should be consulted for further details.

"Within Sample" confidence limits

Suppose about 1 m of sliver is received for test and n test specimens are weighed out and tested, three readings being taken on each in accordance with standard procedure. A total of 3 n readings would be obtained, and since the variance due to repacking is normally about the same as that between different weighings, the 95 % confidence limits of the average readings are given by

$$\pm\frac{1{,}96\sigma}{\sqrt{3n}}$$

where σ is the standard deviation of the 3 n readings. From the work of various authors, it appears that the value of σ is about 0,2 µm for wool of 20 µm, rising to about 0,4 µm for wool of 30 µm.

"Between apparatus" confidence limits

The following confidence limits have been established by a determination carried out by the usual method on two test specimens in any one of 16 laboratories participating in tests with certified apparatus using parts of four identical reference slivers.

Average µm	**95 % confidence limits** µm
20	±0,18
25	±0,29
30	±0,42
35	±0,59

Variability within lots during processing

Although the variability within lots is not related to the reproducibility of the method of test, it is sometimes necessary to take variability within lots into account when comparing results obtained in different laboratories, since the laboratory samples may have been taken at different times from different portions of a non-homogeneous lot. Some tests relating to variations in mean fibre diameter of combed slivers during processing have shown that significant differences might occur.