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ISO RECOMMENDATION

R 1136

AIR PERMEABILITY METHOD

FOR MEASURING THE MEAN DIAMETER OF WOOL FIBRES

1st EDITION

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BRIEF HISTORY

The ISO Recommendation R 1136, *Air permeability method for measuring the mean diameter of wool fibres*, was drawn up by Technical Committee ISO/TC 38, *Textiles*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question led to the adoption of a Draft ISO Recommendation.

In January 1968, this Draft ISO Recommendation (No. 1245) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	India	Romania
Belgium	Iran	South Africa, Rep. of
Brazil	Israel	Spain
Canada	Italy	Sweden
Cuba	Japan	Switzerland
Czechoslovakia	Korea, Rep. of	Turkey
Denmark	Netherlands	U.A.R.
France	New Zealand	United Kingdom
Germany	Norway	U.S.S.R.
Greece	Poland	
Hungary	Portugal	

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in November 1969, to accept it as an ISO RECOMMENDATION.

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AIR PERMEABILITY METHOD FOR MEASURING THE MEAN DIAMETER OF WOOL FIBRES

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. Owing to the speed and simplicity of the method it is particularly suitable for quality control in mill testing laboratories.

Since the method is indirect, the apparatus must first be 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 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 may need special calibrations as described in Annex D.

1. SCOPE

This ISO Recommendation describes a method of measuring the mean diameter of wool fibres, using an apparatus which passes a current of air through a bundle of fibres.

The method is applicable to clean, unmedullated wool fibres dispersed in a uniform, open state. It is particularly suitable for combed slivers. The method is also applicable to oil-combed slivers without cleaning, if the oil content is constant and the apparatus suitably calibrated.

The method is less accurate for lambswool which is appreciably medullated (see Annex D).

2. DEFINITIONS

- 2.1 *Laboratory sample.* The conditioned sample of fibres, representative of the bulk, from which the test specimens are weighed out. In many cases the laboratory sample will consist of one or more short lengths of sliver.
- 2.2 *Test specimen.* The weighed amount of fibre which is packed into the constant volume chamber.

3. 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.

4. APPARATUS

4.1 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 microns. Since less accuracy in weighing the specimen is required, this method has some advantages for mill use.

4.2 Detailed parts

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

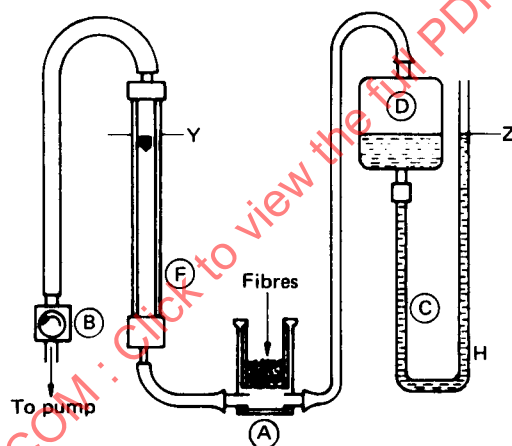


FIG. 1 — General arrangement of apparatus

- 4.2.1 *Air valve (B)*, giving sufficiently fine control of the air supply, such that the lever of the flowmeter or manometer may be quickly adjusted to the working value.
- 4.2.2 *Suction pump* of a type providing a smooth output of at least 30 litres per minute at 200 mmH₂O 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).
- 4.2.3 *Constant volume chamber (A)*, of brass, hardened steel, or any other suitable metal, suggested dimensions of which are given in Figure 2.
This comprises the following three 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 should be smooth so that the plunger slides easily into the base without trapping fibres.
- 4.2.4 *Reservoir (D)* of fluid manometer as specified in the Table on page 7, mounted at sufficient height to give a clear working distance ZH of 350 mm in the glass limb of the manometer.
The manometer (C) is made of glass tube 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 limb ZH as described in Annex A, clause A.3.1.

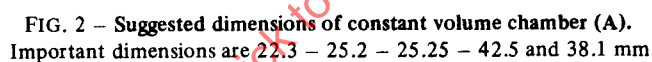


TABLE – Manometer and flowmeter characteristics

Characteristics considered	Constant flow	Constant pressure
Minimum diameter of reservoir	150 mm	60 mm
Type of manometer fluid	<i>n</i> -propyl alcohol	Distilled water
Working range of flowmeter	10 to 20 litres/minute	5 to 25 litres/minute

4.2.6 *Rubber tube* connecting the manometer reservoir (D) to the chamber (A), which should be pressure tubing of small internal diameter to avoid constriction at the bends.

4.2.7 Rubber or plastic tube from the chamber (A) to the flowmeter (F), which should be of internal diameter not less than 6 mm, should be as short as possible and should not be twisted or kinked between calibration of the apparatus and its subsequent use.

4.3 Balance

Provide a 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.

5. CONDITIONING AND TESTING ATMOSPHERE

- 5.1 Whenever possible, condition the laboratory sample to equilibrium and test in the standard atmosphere for testing defined in ISO Recommendation R 139*, *Standard atmospheres for conditioning and testing textiles*.
- 5.2 If tests are not carried out in the standard atmosphere for testing, condition the laboratory sample to equilibrium near the apparatus and note the relative humidity of the atmosphere at the time of test; then correct the final results by the factors given in Annex C.

NOTE. — A source of error may occur if the moisture of the specimen changes during test. This may 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 minutes.

6. PREPARATION OF TEST SPECIMENS

6.1 Cleaning

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

If the laboratory sample is known to be dry-combed with fatty matter content below 1 % the test specimens may be taken from it without cleaning.

If the laboratory sample is known to be oily with fatty matter content between 3 and 4 %, the test specimens may be taken from it without cleaning provided the apparatus has been calibrated from oil-combed slivers.

6.2 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 .

6.3 Selection of specimens

Take the specimens from different places in the laboratory sample. In the case of balls of sliver the laboratory sample should be made up of pieces of sliver from both inside and outside the ball.

6.4 Specimen mass

For the constant flow method the specimen mass should be 1.5 ± 0.002 g. For the constant pressure method the specimen mass should be 2.5 ± 0.004 g.

6.5 Preparation

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

For slivers with pulled ends, remove and discard about five hand draws, then weigh out the specimens by taking several successive hand draws.

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

7. TEST PROCEDURE

- 7.1 Ensure that the meniscus of the manometer is at the zero mark and if required carry out an orifice plate check as detailed in Annex A, clause A.3.3.
- 7.2 Pull out the weighed test specimen into a long thin sliver and feed it evenly into the constant volume chamber (A), packing the fibres down with a smooth rod from time to time. Insert the plunger and screw down the cap to the furthestmost extent so that the lip of the plunger is in contact with the base.
- 7.3 Depending on the method to be used, adjust the air valve as follows :
 - (a) for the constant flow method adjust the air valve until the top of the float of the flowmeter coincides with the reference mark Y and note the fluid level of the manometer to the nearest millimetre or $0.1 \mu\text{m}$ (see Annex A, clause 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 H and note the position of the float of the flowmeter to the nearest millimetre or $0.1 \mu\text{m}$ (see Annex A, clause A.3.2).
- 7.4 Remove the specimen from the constant volume chamber, tease out the fibres by hand, repack in the constant volume chamber without loss of fibre, insert the plunger and screw down the cap.
Note the reading as before.
- 7.5 Repeat the operation described in clause 7.4 so that a total of three readings on each test specimen is obtained.

8. EXPRESSION OF RESULTS

Calculate the average of the three readings for each specimen and express the result to the nearest $0.1 \mu\text{m}$.

9. TEST REPORT

The test report should state, in addition to the results,

- the reference to this ISO Recommendation and the method used;
- whether the sample was tested after cleaning in petroleum ether or without cleaning;
- the relative humidity and temperature of conditioning and testing atmosphere and whether the result has been corrected for the relative humidity;
- all operating conditions not specified in this ISO Recommendation as well as any incidents that may have influenced the results.

ANNEX A

CALIBRATION OF APPARATUS

A.1 LEAKAGE TEST

After assembling the apparatus as in Figure 1, remove the cap and plunger from the constant volume chamber (A) and insert a rubber stopper. By means of a Hoffman clip close the rubber tube between (A) and (F) after introducing a pressure difference causing the meniscus in the manometer to alter by about 150 mm. Note the position of the meniscus periodically for several minutes; if it changes, the apparatus should be examined 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 g or 2.5 g);
- (b) whether oil-combed or dry-combed samples are required.

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

A.3 GRADUATING THE SCALE

A.3.1 Constant flow apparatus

Make a horizontal mark Y (see Fig. 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 described in sections 5 and 6, 1.5 g specimens of each sample of reference sliver and test according to the procedure described in section 7, noting the distance in millimetres below the zero to which the meniscus falls. Do not clean the sliver before test. Test three 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 microns 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 microns, or a scale may be graduated in microns and fixed behind the manometer.

Adjustment of results by the least squares method

The relation between d and h is of the form $hd^b = \text{constant}$ 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 Y_1 , X_2 and Y_2 , etc.) are obtained.

First calculate the following quantities :

$$\Sigma X = X_1 + X_2 + \dots + X_n$$

$$\Sigma Y = Y_1 + Y_2 + \dots + Y_n$$

$$\Sigma Y^2 = Y_1^2 + Y_2^2 + \dots + Y_n^2$$

$$\Sigma XY = X_1 Y_1 + X_2 Y_2 + \dots + X_n Y_n$$

$$\Sigma y^2 = \Sigma Y^2 - \frac{(\Sigma Y)^2}{n}$$

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

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

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

$$X = \frac{\Sigma X}{n} + b \left(Y - \frac{\Sigma Y}{n} \right) \quad \dots (1)$$

Finally, construct a table relating h to d by taking values of h at 5 mm intervals, finding $\log h$, substituting in equation (1) to obtain X and so tabulating $d = \text{antilog } X$ 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 Z of the manometer. Fix a scale graduated in millimetres behind the flowmeter (F) 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 described in sections 5 and 6 and test according to the procedure described in section 7, noting the distance y in millimetres of the float of the flowmeter from zero. Do not clean the slivers before test. Test three 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 the equation

$$y = a + bd + cd^2 \quad \dots (2)$$

by solving the equations

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

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

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

The equation (2) is then used to graduate a scale in microns 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 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 above described conditions.

At least once a day orifice plates are clamped in the apparatus so that air enters through the central hole only and the readings are noted. Variations in the readings given by the scale should 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

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 metre 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 $3n$ 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 $3n$ readings. From the work of various authors it appears that the value of σ is about $0.2 \mu\text{m}$ for wool of $20 \mu\text{m}$, rising to about $0.4 \mu\text{m}$ for wool of $30 \mu\text{m}$.

“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 concerned with 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 may occur.