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Textiles — Quantitative analysis of animal fibres by microscopy — Cashmere, wool, speciality fibres and their blends

Textiles — Analyse quantitative des fibres animales par microscopie — Cachemire, laine, fibres spéciales et leurs mélanges

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Foreword

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International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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ISO 17751 was prepared by Technical Committee ISO/TC 38, *Textiles*, Subcommittee SC 23, *Fibres and yarns*.

This International Standard is based on IWTO-58-00, *Scanning Electron Microscopic Analysis of Speciality Fibres and Sheep's Wool and their Blends*, copyright the International Wool Textile Organisation (IWTO), used with permission of IWTO.

Introduction

Labelling textiles to indicate their composition is necessary according to relevant laws and regulations, not only for the final products but also for the raw materials at different stages of processing. Stringent labelling regulations for textile products at all stages of processing have compelled the manufacturers to state not only the types of fibre but also the mass percentages of the fibres contained in their goods.

Wool and speciality fibres (cashmere, mohair, llama/alpaca, camel hair, angora rabbit hair, etc.) exhibit great similarities in their physical and chemical properties, so that their blends cannot be separated mechanically or chemically. Light microscopy (LM) has traditionally been applied for fibre identification and blend analysis.

Wool has a long tradition as the main substitute in mislabelling when it is blended with animal fibres such as mohair and cashmere. A reliable method, complementing the current and widely used standards based on light microscopy, for distinguishing wool from all other speciality fibres is therefore of major technical and commercial importance.

A technique using scanning electron microscopy (SEM) for the discrimination of wool and speciality animal fibres, based on the assessment of cuticle scale edge heights, was introduced and developed during the 1980s and early 1990s. Although SEM illustrates topographical features extremely well, it is incapable of describing internal fibre structures. Fortunately, this deficiency can be complemented by LM which is capable of illustrating internal features. For all these reasons, it is insufficient to depend on only one form of microscopy and it is advantageous to utilize both LM and SEM techniques.

The identification of animal fibres is so complex that it is often necessary to consider subtle characteristics that require a multidisciplinary microscopic approach.

Textiles — Quantitative analysis of animal fibres by microscopy — Cashmere, wool, speciality fibres and their blends

1 Scope

This International Standard specifies a method for the identification and quantitative analysis of wool and speciality animal fibres using both light microscopy (LM) and scanning electron microscopy (SEM). This standard is also applicable to blends of animal fibres and products made from them.

NOTE 1 Difficulty may be encountered when attempting the analysis of deeply dyed or heavily pigmented fibres by LM. In such cases, mild dye-stripping or pigment-bleaching procedures may be applied prior to analysis.

NOTE 2 SEM is not an appropriate technique for the analysis of blends containing medullated fibres since the medullae will not be visible.

2 Caution

The microscopic analysis of blends of animal fibres requires a high degree of operator skill and experience. Only when authentic reference samples have been successfully identified by multiple replications over a prolonged period, and trial blends of known composition have been tested with acceptable results, should official analysis be performed by an operator.

3 Normative references

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

ISO 6938, *Textiles — Natural fibres — Generic names and definitions*

4 Terms and definitions

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

4.1
CRT
cathode ray tube or display screen

4.2
false scale edge
shoulder
step-like structure on the surface of a cuticle cell, which may be mistaken for the scale edge

4.3
light microscope
optical instrument used to produce magnified images

NOTE Light microscopes may be of the reflected-light, transmitted-light or light-projection type. Either a transmitted-light type or a light-projection type is preferred for this type of analysis.

4.4
medulla
series of cavities formed in the central portion of some animal fibres when cells collapse during growth

4.5
sample
portion representative of the batch of material from which it is taken

4.6
scale
cuticle covering the surface of animal fibres

4.7
scale density
number of scales per millimetre of fibre

4.8
scale edge
thick, distal end of the cuticle cell exposed towards the tip of the fibre

4.9
scale thickness
height of the cuticle at the scale's edge

4.10
scanning electron microscope
electron-optical instrument that examines and analyses the physical information (such as secondary electron, backscattered electron, absorbed electron and X-ray radiation) obtained by generating electron beams and scanning the surface of the sample in order to determine the structure composition and topography of the sample

4.11
secondary electron image
SEI
scanning image which is obtained by modulating the brightness of a cathode ray tube (CRT) with the detected secondary electron signal

4.12
snippet
small sections of fibre cut from a sample

4.13
speciality fibre
any animal source (type) of keratin fibre other than wool: i.e. cashmere goat, angora goat (mohair), angora rabbit hair, camel hair, cashgora goat, llama/alpaca hair, shahtoosh hair, vicuna hair, yak hair, horse hair

NOTE 1 Photographs of the animal fibres listed may be found in AATCC Test Method 20 and IWTO 58-00 (see Bibliography).

NOTE 2 Trade in some animal fibres (e.g. shahtoosh, vicuna, yak) is not always allowed because the animals are protected. Animals under protection are listed in the Washington Convention.

4.14
test specimen
portion taken from randomized snippets for measurement purposes

5 Principle

Following sampling, short fibre snippets are obtained from the material to be tested. The snippets comprising a test specimen are distributed uniformly on suitable sample holders.

For light microscopy (LM), test specimens are analysed optically and measured using a graduated scale. For scanning electron microscopy (SEM), test specimens can be coated with a layer of gold before they are transferred into the microscope. At a magnification of × 1 000, or another suitable magnification, the number of fibres from each animal source is determined by observing and identifying them under the microscope.

With SEM, wool fibres can be differentiated from speciality fibres from all other sources on the basis of the height of their surface scales. The height at the true distal edge of wool cuticle cells (not at “false scale edges” or “shoulders”) reaches a value of 0,6 µm or more, whereas the distal height of speciality fibres is 0,4 µm or less. Edge height is generally a selective indicator for the fibre type. Other characteristics such as scale pattern, scale frequency and diameter are also useful for unequivocal fibre identification.

For a quantitative analysis of a binary blend, the mean diameters, and the related standard deviations, of the fibre components are determined together with the number of fibres of a given type, in order to calculate the percentage fibre content by number of fibres or by mass for each source of fibre. For angora rabbit hair, the reduced mean fibre density, due to consistent medullation, is taken into account.

Practice shows that the experience of the operator with animal fibre identification is an important requirement for conducting reliable fibre analyses.

6 Apparatus and reagents

6.1 Light microscope

6.1.1 Type of microscope

6.1.1.1 Projection type

The microscope proper shall comprise a light source, a light condenser, a stage which supports the mounted specimen of fibres, an objective, an ocular and a circular viewing screen. The stage shall be movable in two directions at right angles by means of sliding mechanisms capable of successive displacements in 0,5 mm steps. The objective and ocular shall be capable of providing a magnification of × 500 at the screen.

The circular screen shall have an associated measurement scale capable of rotation in the plane of the screen and about its centre. If this screen is not transparent, it shall have a movable scale 5 cm long, graduated on its underside in millimetres. The scale shall be capable of movement diametrically across the screen between guides. Transparent screens may incorporate a scale graduated in millimetres along a diameter. A movable scale is generally preferred. The circular screen shall contain a marked central circle whose diameter is equal to one-quarter of the optical distance between the ocular and the centre of the screen. To ensure that any lens aberrations at the objective perimeter are avoided, all measurements shall be made within this circle. However, some modern instruments contain improved optics that ensure uniformity of the observation area, and no marked circle is required. In such cases, the magnification should be checked over the whole projected image by using a certified micrometer scale.

6.1.1.2 Transmitted-light type

The microscope proper shall comprise a light source, a light condenser, a stage, an objective and an ocular. The ocular shall be fitted with a calibrated graticule to permit measurement of the fibre diameter. The stage shall be movable in two directions at right angles by means of sliding mechanisms capable of successive displacements. The objective and ocular shall be capable of providing a magnification of × 150 to × 500.

6.1.2 Slides and cover glasses

Use glass microscope slides measuring 75 mm × 40 mm. Square or rectangular cover glasses with a thickness of 0,13 mm to 0,17 mm can be used.

6.1.3 Mounting medium

Use a mounting medium with the following properties:

— refractive index between 1,43 and 1,53;

— suitable viscosity;

— does not absorb water.

NOTE Cedar wood oil and liquid paraffin are examples of suitable media.

6.1.4 Fibre-cutting devices

6.1.4.1 General

For cutting the fibres to a predetermined length, the fibre holder and pushers described below may be used. Alternatively, a conventional microtome may be used if it is capable of fulfilling the requirements of 7.2 regarding the cutting of pieces of fibre.

6.1.4.2 Fibre holder and pushers

The holder is a short piece of smooth steel about 3 mm thick with a 1,5 mm slot into which slides a tongue. The tongue is fixed by a screw and may thus be adjusted to project different distances into the slot. The pushers consist of three steel stems with shortstop plates near their ends; all the stems have the same width as the slot, namely 1,5 mm. The stem of one pusher extends 0,8 mm beyond the stop plate, that of the second 0,6 mm and that of the third 0,4 mm.

6.2 Scanning electron microscope

6.2.1 Operating conditions

Accelerating voltage:	15 kV to 20 kV.
Beam current:	300 pA to 500 pA.
Pressure in the sample chamber:	$< 10^{-5}$ mbar (10^{-8} Pa).
Image mode:	Secondary electron image.
Resolution of secondary electron image:	Better than 20 nm.
Magnification:	× 10 to × 20 000. For observation of the fibre scale shape and density, × 1 000 may be used. For observation of scale thickness, × 15 000 may be used.

NOTE 1 Other magnification levels may be used to ensure clear images for the operator to make measurements.

Tilting: 0°.

NOTE 2 No special atmosphere is required for preparing SEM specimens as the analysis is performed in a vacuum.

6.2.2 Mounting stubs

Use aluminium or brass holders 13 mm in diameter.

6.2.3 Sputter coater with a gold cathode or vacuum evaporator

6.2.4 Reagents

These are used for the uniform distribution of the snippets on the glass plate:

6.2.4.1 **Acetone** (analytical grade).

6.2.4.2 **Ethyl acetate** (analytical grade).

6.2.4.3 **Petroleum ether** (analytical grade).

6.2.5 Miscellaneous materials

— Single- and double-sided adhesive tape.

— Glass plate measuring approx. 30 cm × 30 cm or 15 cm × 15 cm.

— Stainless-steel rod, 0,5 mm in diameter.

— Razor blade.

— Glass tube, 10 mm to 15 mm in diameter, 35 mm in height.

— Vernier callipers, if necessary (see 8.2.5).

7 Preparation of the test specimens

7.1 General

The general requirement is that the test specimen shall be representative of the batch of material or sample from which it has been taken. The method of obtaining a fibre test specimen will differ depending upon the sample form (loose fibre, fibre blend, yarn, sliver or fabric). No procedures for sampling are given here as the original form of the bulk to be sampled may vary widely.

For suggested methods of preparing test specimens, refer to Annex B.

7.2 Microtoming

7.2.1 For light microscopy, prepare fibre snippets using a microtome device. Take fibre snippets of length 6 mm ± 2 mm, irrespective of fibre diameter. A total mass of 10 mg of fibre snippets is recommended.

7.2.2 For scanning electron microscopy, prepare snippets ⩾ 6 mm in length to minimize coagulation and uneven dispersion of fine or crimped fibres.

7.3 Mounting specimens

7.3.1 For light microscopy measurements, prepare two slides, as follows. Place cut fibres in a few drops of mounting medium on a glass slide. Stir the fibres in the mounting medium with a dissecting needle, employing a circular motion to achieve a uniform distribution on the slide. Lower a cover glass on to the mixture by placing one edge in contact with the slide and gently lowering the opposite edge.

7.3.2 For scanning electron microscopy, collect the fibre snippets in a test tube. Prepare seven stubs, as follows.

Suspend fibres in 1,5 ml ± 0,5 ml of reagent (see 6.2.4) by stirring the snippets with a stainless-steel rod. Without giving the snippets time to settle, pour the suspension onto a glass plate. After all the reagent has evaporated, the fibres should be uniformly distributed in a single-layer spot of diameter 10 cm on the glass plate.

If the snippets have aggregated after the evaporation of the reagent, they shall be recollected by scraping them off the glass plate with a razor blade and the preparation procedure repeated.

Press SEM mounting stubs, that have been attached to lengths of double-sided adhesive tape, onto the fibre layer. Pull off the stubs carefully.

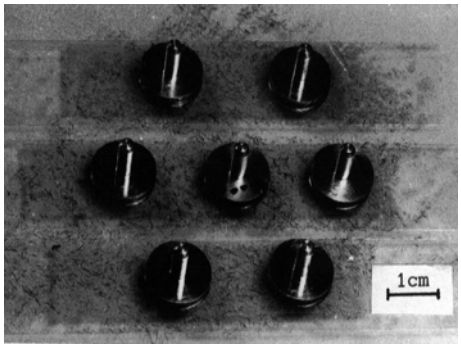

Figure 1 — Arrangement of the specimen stubs on the spot with the fibre snippets

If required by the SEM system, use the sputter coater to apply a 15 nm layer of gold to each test specimen.

8 Procedure

8.1 Analysis by light microscopy

8.1.1 General

For both the projected-light and transmitted-light methods, a total of at least 1 000 fibres shall be examined in the specimens from a sample. At least 100 fibre diameter measurements shall be made on the fibres from each fibre source in the sample.

8.1.2 Verification of calibration

8.1.2.1 The calibration of a projection-type microscope shall be checked periodically using a certified micrometer scale at the × 500 magnification setting. A micrometer scale with 0,01 mm graduations is recommended. The scale is mounted on the microscope stage and the magnification adjusted so that 0,1 mm on the scale shows as 50 mm on the screen.

8.1.2.2 The calibration of a transmitted-light microscope shall be checked periodically using a certified micrometer scale at the × 400 magnification setting.

8.1.3 Measurement of fibres

8.1.3.1 Place a prepared slide on the microscope stage with the cover glass towards the objective. Examine the slide at different depth fields. The distance between the centres of the fields should be greater than the length of the snippets to prevent the measurement of the same snippet twice.

8.1.3.2 Move the slide until a corner of the cover slip is focused. Traverse the slide 0,5 mm from A to B (see Figure 2). Then move the slide 0,5 mm in the transverse direction (towards C) to bring the first field into view on the screen. Measure and record the width of every fibre image lying within the field of view (with the exception of those described in 8.1.3.6).

8.1.3.3 When the objective in a projection-type microscope is too near the slide, the edge of a fibre image will have a white border. When the microscope objective is too far from the slide, the edge of the fibre image will have a black border.

When in focus, the edge of the image shows as a fine line with no border. However, both edges of a fibre image may not be in focus together, since wool fibres have non-circular cross-sections. When measuring an image whose edges are not in focus together, adjust the focusing so that one edge is in focus and the other shows a white line. Measure the width from the edge that is in focus to the inside of the white line.

8.1.3.4 Measurement is made by moving the graduated scale or screen with its length at right angles to the fibre image until a centimetre division coincides with one edge of the image. The width of the fibre image is then read in millimetres. The measurement shall be taken where the longitudinal line of the scale crosses the image. The width is taken as the distance between the extremities of the fibre image, even if the fibre edges coincide with a fibre scale or some other irregularity of the fibre. The stage shall remain stationary during all measurements in a given field.

8.1.3.5 Continue to move the slide in 0,5 mm steps towards C, measuring and recording the fibres in each field of view as before. When C is reached, traverse the slide a suitable distance (see 8.1.3.7) from C to D, and then proceed in 0,5 mm steps in the transverse direction (towards E), measuring and recording the fibres in each field. Continue in this way, following the path shown in Figure 2, until the opposite edge of the cover glass is reached.

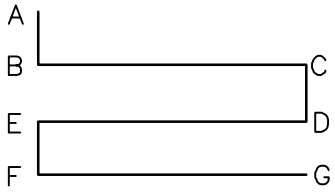

Figure 2 — The sampling path A B C D E F G (not to scale)

8.1.3.6 Images of fibres within each field of view not to be counted or measured are:

a) those with more than half their width outside the central circle;

b) those whose widths are not wholly within the boundary of the measurement screen for systems without central circles;

c) those that end within the width of the 5 cm graduated scale;

d) those that end 2,5 cm or less from the point of measurement;

e) those that cross another image at the point of measurement.

Animal fibre scales are important for the accurate identification of fibre source or type. Shearing, processing, recycling or dyeing may damage fibre scales and hinder the identification of fibre source or type. If all fibres from one source in a blend of fibres are damaged, the damaged fibres contribute to the total mass and shall be used in the calculation of mass fibre content.

Parties shall agree upon the definition of damaged fibres and the inclusion or exclusion of damaged fibres in the total number of fibres measured and in the calculation of mass fractions in blends.

8.1.3.7 From the number of fibres measured in the first traverse (BC in Figure 2) and the total number of measurements desired, estimate the number of traverses and the length of each cross-traverse (e.g. CD in Figure 2) to achieve the necessary number of fibre measurements. If fibres are needed from the second slide prepared, then half of the measurements shall be taken from each slide.

8.1.3.8 When making diameter judgements, if the second edge of a focused fibre image falls between two millimetre divisions of the measurement scale, the diameter shall be recorded as that corresponding to the lower millimetre division. In the subsequent calculation, all such diameters recorded under N shall be assigned a diameter equal to $N + 0{,}5$ mm. In the event that the second edge of a fibre image lies exactly on a millimetre division on the scale, the image shall be assigned alternately to this group and to the lower-diameter group to avoid the assignment of 0,5 mm diameters.

8.1.4 Precision and accuracy

No precision and accuracy studies are available for the analysis of fibre source or fibre diameter by light microscopy.

8.2 Analysis by SEM

8.2.1 General

Place a stub in the scanning electron microscope. Using the $\times$ 10 magnification setting, bring an area near the upper left edge of the stub onto the monitor. Set the magnification to $\times$ 1 000 and focus the image. Scan the stub either horizontally or vertically. During scanning, identify each fibre appearing on the monitor and count the number of fibres encountered from each fibre source. If necessary, higher magnification settings can be used ($\times$ 3 000 to $\times$ 10 000).

If required by the composition of the material (case 3 below), diameter measurements of fibres from each kind of source shall be made.

In the case of severely damaged fibres, examine the fibre snippet along its length to facilitate analysis. In order to avoid examining the same fibre twice, move the point of focus back to its original position on the fibre and return the magnification to $\times$ 1 000 before continuing the analysis. When the opposite edge of the mounting stub is reached, move the scanning range by at least twice the length of a snippet ($>$ 1,2 mm) and reverse the direction of scanning of the mounting stub.

8.2.2 Fibre identification

The height of the distal edge of the cuticle scale, a crucial characteristic of fibre type, is determined on the fibre surface at a magnification that makes it possible to decide whether the scale edges are “high” (0,55 µm for wool) or “low” ($<$ 0,55 µm for speciality fibres).

For fibres where the distinction is not obvious, examine several cuticle scale edges at high magnification settings, such as $\times$ 3 000 to $\times$ 10 000, until confident of the identification. Observations of further fibre surface characteristics, such as scale frequency and scale patterns, may be necessary to assist the operator with some fibre identifications. Degraded wool is an example of a fibre type that would be likely to require additional fibre observations.

Annex A shows the typical appearance of the various types of fibre when viewed by SEM.

8.2.3 Qualitative analysis

Examine 150 fibres on the first stub to ascertain whether one or more than one type of fibre is present.

8.2.3.1 Case 1: Single animal fibre source

If only one fibre type is found, examine 150 fibre snippets on each of the next two stubs. If no fibre of a second type is found in the total of 450 fibres examined, the sample is declared as pure.

8.2.3.2 Case 2: Multiple animal fibre sources

If two fibre types are found on the first stub during the examination of the 150 fibres on this stub and the proportion (by number) of one type is less than 3 % (i.e. $\leqslant$ 4 fibres out of 150), it is considered as a minor component. Examine 150 fibres on each of the next two stubs. Based on the 450 observations, calculate the percentage N_x (by number) of the minor component and the corresponding percentage N_y (by number) of the speciality fibre.

EXAMPLE Examination of 450 fibres gave the following results:

Number of wool fibres N_w: 12

Number of mohair fibres N_s: 438

Thus the percentage (by number) of wool fibres is 2,7 % and that of mohair fibres is 97,3 %.

8.2.3.3 Case 3

If two fibre types are found and the proportion (by number) of the less frequent type is greater than 3 % of the total number (more than 12 fibres out of 450), the fibre mixture is considered to be a blend. Perform a quantitative analysis in accordance with 8.2.4.

8.2.4 Quantitative analysis of a two-component blend

If the sample is found to be a blend (case 3), examine 150 fibres from each of a total of seven stubs for identity, and measure the diameters of the first 20 fibres of each component identified (or all fibres of that component, if less than 20) on each stub. A total of 1 050 fibres are thus identified in the sample and measurements of fibre diameters are made for each component.

8.2.5 Fibre diameter measurement

With the aid of the computer software of modern scanning electron microscopes, set the two cross-cursors at both outer sides of each fibre to be measured. Read the shortest distance between them (i.e. the diameter of the fibre being displayed on the monitor) and record it. It is important to note that, due to the vacuum in SEM, these diameters are those for the dry state of the fibres.

NOTE For standard atmospheric conditions as specified in ISO 139, add 5 % or 10 % to the average fibre diameter to adjust for diameter increase with moisture uptake.

When suitable computer software is not available, the apparent diameter of the fibres can be determined from the monitor image with vernier callipers. Since the calibration factor between a vernier reading and the real fibre diameter influences the components in a mixture in the same way, the readings can be used directly for the calculation of component mass percentages. When the real diameters of the components in a vacuum are to be measured, an inner calibration standard, i.e. a standard sized grid, shall be used.

Some trading systems categorize wool, wool top and speciality hair (cashmere, vicuna, camel, alpaca and llama) by grades with specific diameter ranges that distinguish wool and wool top from cashmere and other speciality hair fibre sources (see AATCC Test Method 20A, Tables III and IV).

8.2.6 Calculation of mass percentages

Introduce the measured data, i.e. the mean diameter, the number and the density of each type of fibre (from LM or SEM) into the formula to calculate the contents (mass percentages) of the fibres.

For example, for a case 3 sample, calculate the wool fibre content w_{w}, expressed as a mass percentage, using the following formula:

$$w_{\mathrm{w}} = \frac{n_{\mathrm{w}} (\bar{d}_{\mathrm{w}}^2 + s_{\mathrm{w}}^2)\bar{\rho}_{\mathrm{w}}}{n_{\mathrm{w}} (\bar{d}_{\mathrm{w}}^2 + s_{\mathrm{w}}^2)\bar{\rho}_{\mathrm{w}} + n_{\mathrm{s}} (\bar{d}_{\mathrm{s}}^2 + s_{\mathrm{s}}^2)\bar{\rho}_{\mathrm{s}}} \times 100$$

where

n_{w} is the number of wool fibres;

n_{s} is the number of speciality fibres;

$\bar{d}_{\mathrm{w}}$ is the mean diameter of the wool fibres;

$\bar{d}_{\mathrm{s}}$ is the mean diameter of the speciality fibres;

s_{w} is the standard deviation for $\bar{d}_{\mathrm{w}}$;

s_{s} is the standard deviation for $\bar{d}_{\mathrm{s}}$;

$\bar{\rho}_{\mathrm{w}}$ is the mean density of the wool fibres (see Table 1);

$\bar{\rho}_{\mathrm{s}}$ is the mean density of the speciality fibres (see Table 1).

The mass percentage of the speciality fibres (e.g. cashmere, mohair) is then given by

$$w_{\mathrm{s}} = 100 - w_{\mathrm{w}}$$

Table 1 — Recommended values of mean density

Name	Mean density, g/cm^3	LM	SEM
Wool	1,31	X	X
Cashmere	1,31	X	X
Camel	1,31	X	
Yak	1,31	X	X
Mohair	1,31	X	X
Alpaca	1,30	X	
Rabbit	1,15	X	
NOTE The use of the formula given implies the assumption of circular cross-sections for the component fibres, so the mean density of some animal fibres is not suitable.			

8.2.7 Precision and accuracy

As a measure of precision and accuracy, the confidence ranges q for the mass percentages are calculated at the 95 % confidence level by applying the rules of the Gaussian error progression. The confidence ranges for the two components are equal. Determining $w_{\mathrm{w}} \pm q$ gives the confidence limits, which are the limits within which the true composition of the sample will be found with a probability of 95 %.

Tables C.1 and C.2 in Annex C give values for the confidence ranges for a variety of blends of wool with speciality fibres, for which the components are chosen so as also to cover extremes in the range of practically relevant materials.

Various investigations and round-robin trials have shown that analyses can consistently be conducted within the confidence limits of the method. No bias has been observed. The 95 % confidence limits are thus considered to cover unsystematic as well as minor, though unspecific, systematic errors, and are thus taken to represent the precision as well as the accuracy of the method.

This statement refers only to samples that represent intimate blends of fibres. This condition is expected to be valid for yarns and is met through the preparation procedure for test specimens from small hand samples of loose fibre material that are generally provided for analysis. No information is available on variations between samples taken from a larger bulk of raw material.

9 Test report

The test report shall include the following information:

a) a reference to this International Standard;

b) the nature of the sample (e.g. fibre, yarn, swatch of fabric, coat);

c) identification of the sample (e.g. lot No., article No.);

d) the method of sampling;

e) the number of test specimens;

f) the type of apparatus: LM or SEM;

g) the number of fibres identified and counted for each component;

h) any deviation from the given procedure.

For two-component analysis (by number), include

i) the percentage (by number) of each fibre component in the sample.

For quantitative analysis, include

j) the number of fibres whose diameter was measured for each component;

k) the content of each fibre component in the sample, expressed as a mass percentage rounded to the nearest whole number;

l) the confidence ranges for the mass percentages, rounded to the nearest whole number.

Additional information for quantitative analysis may include

m) the mean fibre diameter and related standard deviation for each fibre component, in micrometres rounded to one decimal place;

n) the coefficient of variation of the mean fibre diameter of each fibre component, expressed as a percentage rounded to the nearest whole number.

Annex A
(informative)

Scale structures of cashmere and wool fibres

A.1 Scale structure of cashmere

Figure A.1 illustrates the scale structure of cashmere. The trunk is surrounded by one or two scales, which have fine and clear edge lines and smooth surfaces. The angle between trunk and scale is small. The large exposed scale results in low scale density. The density is about 60 scales per millimetre of fibre. The scale is thin and its mean thickness is generally about 0,4 µm or less (see Figure A.2).

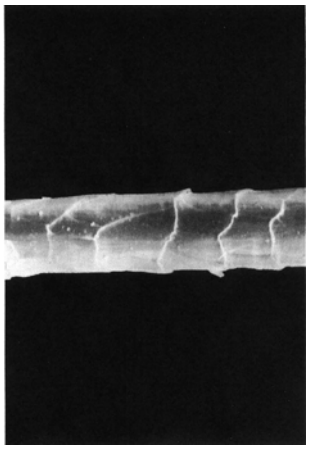

Figure A.1 — Scale structure of cashmere (×1 000)

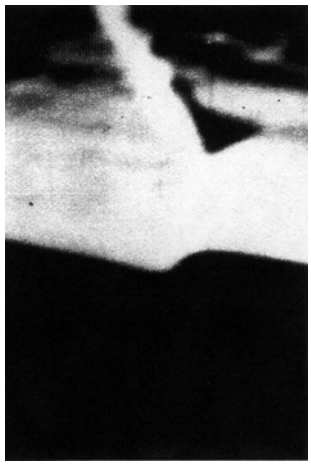

Figure A.2 — Scale thickness of cashmere (×15 000)

A.2 Scale structure of wool

Wool is classified by its diameter into coarse, medium and fine wool. Figures A.3 to A.5 illustrate the scale structure of wool. The mean density of wool scale is greater than that of cashmere. There are about 90 scales per millimetre and the mean thickness is generally about 0,8 µm (see Figure A.6).

Figure A.3 illustrates the scale structure of coarse wool. The scales are in the shape of flakes which are irregular or approximately square. The exposed part of the scale is large, or the greater part is exposed. The scales are connected up to each other and their surfaces are rough and coarse. The scale edge is broadened and indistinct.

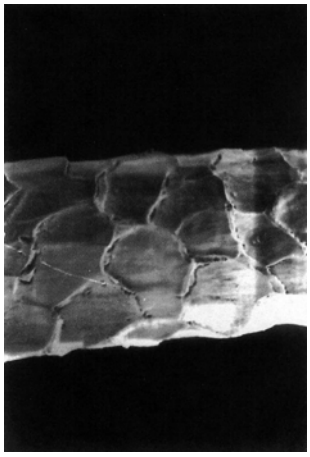

Figure A.3 — Coarse wool (×1 000)

Figure A.4 illustrates the scale structure of medium wool. The scales are in the shape of flakes which are approximately rectangular. The scales overlap or are connected up to each other. The area of exposed scale is larger than for fine wool. The surface is rough and coarse. The scale edge is broadened and indistinct.

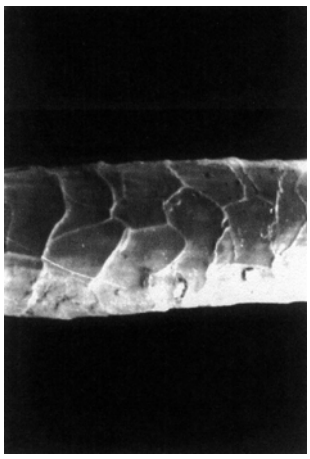

Figure A.4 — Medium wool (×1 000)

Figure A.5 illustrates the scale structure of fine wool. Almost all parts of the trunk are surrounded by ring-shaped scales. The scale edge is broadened and indistinct. The angle between scale and trunk is large, resulting in many saw teeth extending out from the trunk. The surface of the scales is rough and coarse. The size of the exposed part of the scale is smaller than that of cashmere.

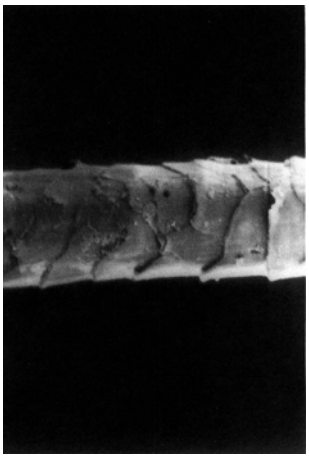

Figure A.5 — Fine wool (×1 000)

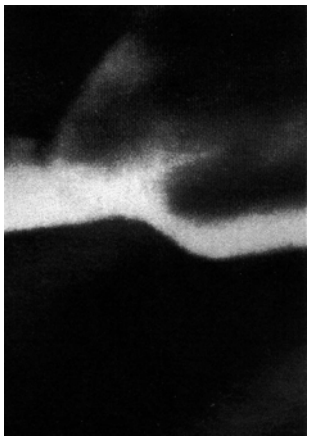

Figure A.6 — Scale structure of wool (×15 000)

A.3 Reference photographs

Figures A.7 to A.14 are for reference purposes.

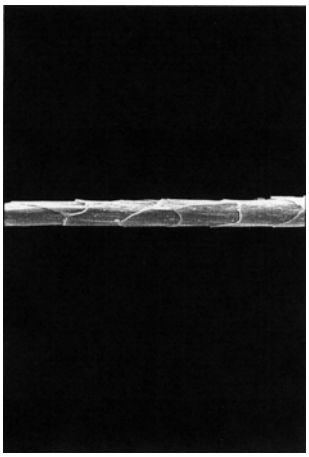

Figure A.7 — Scale structure of Tibetan antelope (shahtoosh) hair (×1 000)

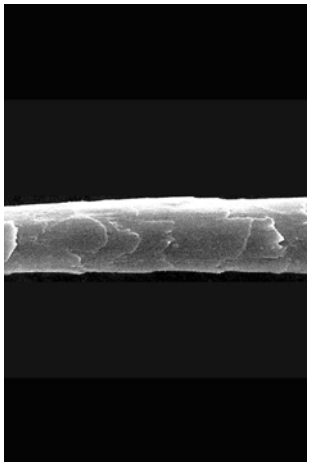

Figure A.8 — Scale structure of yak hair (×1 000)

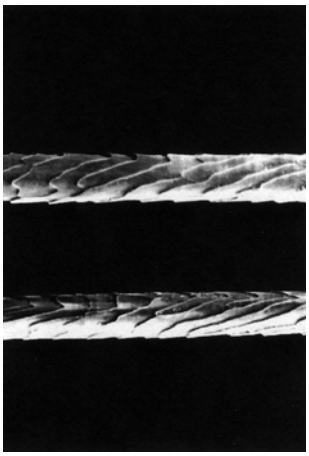

Figure A.9 — Scale structure of rabbit hair (×1 000)

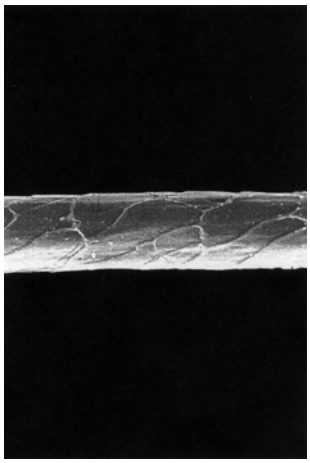

Figure A.10 — Scale structure of camel hair (photo 1) (×1 000)

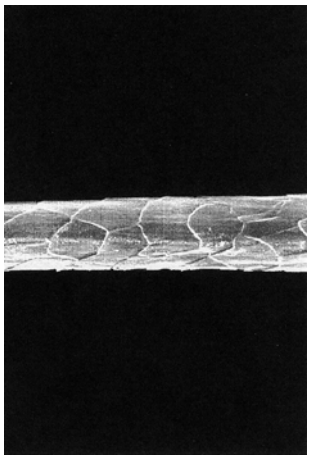

Figure A.11 — Scale structure of camel hair (photo 2) (×1 000)

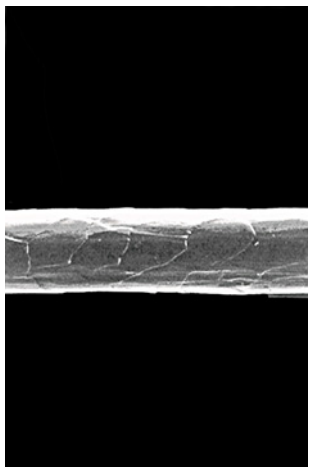

Figure A.12 — Scale structure of mohair (×1 000)

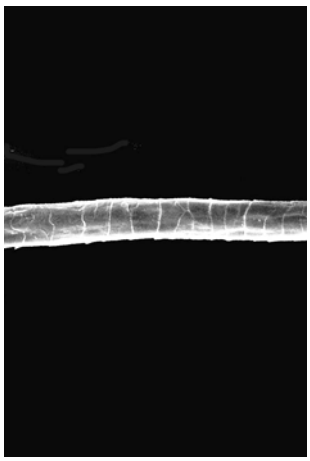

Figure A.13 — Scale structure of vicuna hair (×1 000)

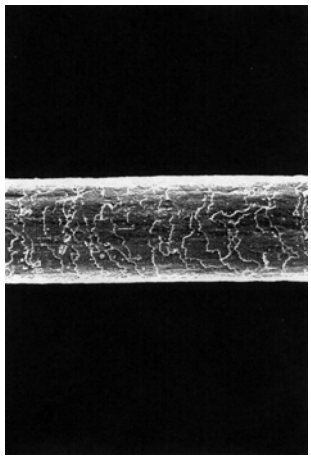

Figure A.14 — Scale structure of alpaca hair (×1 000)

Annex B
(informative)

Sampling and preparation procedures

B.1 Loose fibre

10 g of the fibre should be taken out at random from the top, bottom, right, left and central parts of each package sampled. 50 % of the total number of packages should be sampled. After blending them homogeneously, the samples should be divided into two portions. One portion is used as the test specimen and the other is retained as a spare.

The test specimen is divided into two equal portions and one (selected at random from the two) is rejected. After mixing the retained portion to ensure it is homogenized, it is divided again into two equal portions in the same way and one portion (selected at random) is rejected. Continue the subdivision procedure until about 50 fibres remain as the final portion.

The remaining fibres are washed with petroleum ether or another similar organic solvent.

When the specimen has reached equilibrium in the laboratory atmosphere, the fibres are cut on a glass plate into short lengths between 0,4 mm and 0,8 mm long.

B.2 Yarn

Cut a 5 cm length of yarn from selected packages in the sample. Cut each length of yarn into at least 20 pieces. Cut each piece in the middle to divide it into two portions. One portion is retained as the test specimen, the other is retained as a spare.

De-twist the yarns in the test specimen and then cut them into short lengths between 0,4 mm and 0,8 mm long.

B.3 Woven fabrics

For large fabrics, take three specimens, each measuring 5 cm $\times$ 5 cm, from places which are 10 cm from the edges of the fabric. Unravel each specimen into separate groups of warp and weft yarns. Then divide the warp yarn and the weft yarn separately into two portions. One portion of each is retained as the test specimen, the other is kept as a spare.

For small fabrics, remove the warp yarns at the edge of the fabric until the filling yarns are exposed for at least 1 cm of their length. Cut off the exposed weft yarns. Divide the weft yarns into two equal portions. One is retained as the test specimen, the other is kept as a spare. Use an analogous procedure to obtain a warp yarn specimen.

After de-twisting, cut the fibres of the warp and weft specimens separately into short lengths between 0,4 mm and 0,8 mm long. The warp and weft specimens should be tested, and the results calculated, separately.

For single-colour fabrics of simple, uniform structure, take warp and weft yarns in the same proportion as they occur in the fabric.

B.4 Knitted fabrics

Loosen knitted fabrics into yarns and cut off 2 m lengths. Cut each 2 m length into at least 20 pieces at least 5 cm in length at random. Divide the pieces into two portions. One portion is retained as the test specimen, the other is retained as a spare.

After the yarns in the specimen have been de-twisted, proceed as described in B.2.

For single-colour fabrics of simple, uniform structure, yarns should be unravelled randomly throughout the sample.

NOTE This procedure cannot be used with warp-knitted fabrics.

Annex C
(informative)

Precision and accuracy

C.1 Light microscopy

There are no known precision or accuracy data for LM.

C.2 Scanning electron microscopy

C.2.1 Calculation of the 95 % confidence range

As well as the values calculated for the mass percentage of wool, w_w, and of speciality fibres, w_s, additional parameters required for the analysis are:

n_{dw}: the number of wool fibres for which the diameter was determined;

n_{ds}: the number of speciality fibres for which the diameter was determined.

Calculate the confidence range, q, in percent, for the mass percentages of the components from the equation

$$q = 1{,}98\sqrt{(I + J + K) \times 10^{-4}} \times 100$$

where the terms I, J and K are calculated from the following equations:

$$I = DEF \times 5 \times 10^{-3}$$

$$J = \frac{G^2 s_w^2}{n_{dw}}$$

$$K = \frac{H^2 s_s^2}{n_{ds}}$$

$$D = n_w + n_s$$

$$E = \frac{A^2 B^2}{C^2}$$

$$F = \frac{n_w n_s}{C^2} \times 2 \times 10^6$$

$$G = BF\bar{d}_w \times 10^{-4}$$

$$H = -AF\bar{d}_s \times 10^{-4}$$

$$A = \bar{d}_w^2 + s_w^2$$

$$B = \bar{d}_s^2 + s_s^2$$

$$C = n_w A + n_s B$$

C.2.2 Confidence range values for blend types of practical relevance

Using the equation given above, the confidence ranges have been calculated for a variety of two-component blends of fibre types specified in Tables C.1 and C.2. The fibres were chosen so as to also cover extreme types of blend, as observed in practice.

Table C.1 — Various types of wool and speciality fibre used for the calculation of the 95 % confidence range of their two-component blends

Fibre type	Designation	Mean diameter µm	CV %
Wool	W19	19	20
	W23	23	25
	W30	30	30
Speciality fibre	S16	16	20
	S23	23	25
	S35	35	30

Table C.2 — Values of the 95 % confidence range calculated for various blends of wool and speciality fibre

Blend type	Ratio of wool/speciality fibre in blend, as mass percentages				
	10:90	30:70	50:50	70:30	90:10
W19/S16	2,3	3,8	4,1	3,4	1,9
W19/S23	1,9	3,6	4,3	3,9	2,4
W19/S35	1,7	3,8	4,9	5,0	3,4
W23/S16	2,7	4,2	4,4	3,6	1,8
W23/S23	2,2	3,9	4,4	3,9	2,2
W23/S35	1,8	3,9	4,8	4,6	2,9
W30/S16	3,5	5,0	4,9	3,8	1,7
W30/S23	2,6	4,4	4,7	3,9	2,0
W30/S35	2,1	4,1	4,9	4,4	2,5

The variability of the confidence range, as given by its standard deviation, has been determined in round-robin trials and was found, in terms of the coefficient of variation (CV), to be about and below 10 % for blends in the 50:50 range and about and below 20 % for blends with a minor component.

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