INTERNATIONAL STANDARD

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Second edition
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Textiles — Quantitative analysis of cashmere, wool, other specialty animal fibres and their blends —

Part 1: Light microscopy method

Textiles — Analyse quantitative du cachemire, de la laine, d'autres fibres animales spéciales et de leurs mélanges —

Partie 1: Méthode de microscopie optique

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Foreword

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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 document 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).

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This document was prepared by Technical Committee ISO/TC 38, *Textiles,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 248, *Textile and textile products,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 17751-1:2016), which has been technically revised.

The main changes are as follows:

— in 3.1, a note to entry of different types of speciality animal fibres has been added;

— in 3.5, a note to entry of a micrograph has been added as Figure 1 to indicate the distal edge;

— the title of Clause 5 has been changed to “Reagents” and the reagent used is listed;

— Clause 6, “Apparatus”, has been added and the apparatus are listed with corresponding subclause numbers; subsequent clause and subclause numbers are changed accordingly;

— in 6.1 and 6.2, requirement on stage micrometer for calibration of magnification has been added;

— in 6.4, two alternative apparatus for scalpel and double blades have been added;

— Clause 7, “Sampling”, has been added and its content is rephrased to match with the property adjustment of Annex A;

— Clause 8, “Conditioning”, has been added;

— Clause 9 has been added as “Preparation of test specimens”;

— in 9.1, the amount of test specimens has been increased, together with the requirement for a third set of test specimens to be tested in case of discrepancy on the 2 test results;

— the title of 9.2 has been changed from “Preparation of the test specimens” to “Preparation method for test specimens”;

— in 9.2.1.3, some necessary complementary operations on specimen preparation have been added;

— in 9.2.4.1, missing information on marking of masses of warp and weft yarns and on laboratory sample has been supplemented;

— in 9.3, the title has been changed from “Decolouring of the laboratory sample” to “Pre-treatment of the laboratory sample”, and the Soxhlet extraction description has been adjusted into this subclause. The requirement of reporting of pre-treatment, if applied, has been added in both 9.3.1 and 9.3.2;

— Clause 10 has been renamed as “Procedure”;

— 10.1, “General”, and its content has been added, the subsequent subclauses have been renumbered;

— in 10.3.1.1, the description has been rewritten to elaborate operation procedures and qualitative test descriptions have been added;

— the title of Clause 11 has been changed from “Calculation of test result” to “Calculation and expression of test result”;

— 11.1 and 11.2 and their subclause titles have been added, respectively;

— a new Clause 12, Test report, has been added;

— the status of Annex A has been changed from informative to normative;

— in Annex D, density of some fibres has been modified and the density of coarse rabbit has been added;

— in Annex D, a footnote has been added to coarse rabbit.

A list of all parts in the ISO 17751 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user’s national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Cashmere is a high-value speciality animal fibre, but cashmere and other animal wool fibres such as sheep's wool, yak, camel, etc. exhibit great similarities in their physical and chemical properties, so that their fibre blends are difficult to distinguish from each other by both mechanical and chemical methods. In addition, these fibres show similar scale structures. It is very difficult to accurately determine the fibre content of such fibre blends by current testing means.

Research on the accurate identification of cashmere fibres has been a long undertaking. At present, the most widely used and reliable techniques include the light microscopy (LM) method and the scanning electron microscopy (SEM) method.

— The advantage of LM method is that the internal medullation and pigmentation of fibres can be observed; the disadvantage is that some subtle surface structures cannot be clearly displayed. A decolouring process needs to be carried out on dark samples for testing, while improper decolouring process can affect the judgment of fibre analyst.

— The SEM method shows complementary characteristics to those of LM method, so some types of fibres need to be identified by scanning electron microscope.

The LM and SEM methods need be used together to identify some difficult-to-identify samples in order to utilize the advantages of both methods.

It has been proven in practice that the accuracy of a fibre analysis is highly related to the ample experience, full understanding, and extreme familiarity of the fibre analyst to the surface morphology of various types of animal fibres. In addition to the textual descriptions, micrographs of different types of animal fibres are given in Annex C.

Textiles — Quantitative analysis of cashmere, wool, other specialty animal fibres and their blends —

Part 1: Light microscopy method

1 Scope

This document specifies a method for the identification, qualitative and quantitative analysis of cashmere, wool, other speciality animal fibres, and their blends using light microscopy (LM).

It is applicable to loose fibres, intermediate-products, and final products of cashmere, wool, other speciality animal fibres, and their blends.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 139, *Textiles — Standard atmospheres for conditioning and testing*

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1
speciality animal fibre
any type of keratin fibre taken from speciality animals (hairs) other than sheep

Note 1 to entry: Speciality animal fibres include cashmere, camel, yak, mohair, angora, rabbit, alpaca etc.

3.2
light microscope
optical instrument used to produce magnified images utilizing a visible light source

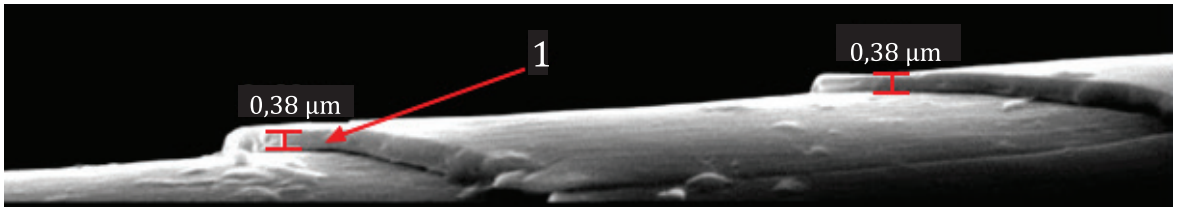
Note 1 to entry: Types of microscopes suitable for fibre identification include projection microscopes and visual microscopic image analysers. Transmitted-light type microscopes with direct graduated scale equipped on optical lens are also applicable.

3.3
scale
cuticle covering the surface of animal fibres

3.4
scale frequency
number of *scales* (3.3) along the fibre axis per unit length

3.5
scale height
height of the cuticle at the *scale's* (3.3) distal edge

Note 1 to entry: The distal edge is shown in Figure 1.



Key
1 distal edge

Figure 1 — Distal edge

3.6
surface morphology
fibre surface morphology
sum of the physical properties/attributes characterizing the fibre surface

Note 1 to entry: The fibre surface morphology includes scale frequency, scale height, patterns of scale edge, scale surface smoothness, fibre evenness along its axis, transparency under light microscope, etc.

3.7
lot sample
portion representative of the same type and same lot of material drawn according to the requirements from which it is taken

3.8
laboratory sample
portion drawn from a *lot sample* (3.7) according to the requirements for preparing test specimens

3.9
test specimen
portion taken from fibre snippets randomly cut from a *laboratory sample* (3.8) for measurement purposes

3.10
warping angle
angle of the free edge of the *scale* (3.3) deviating from the parallel edges of the fibre

4 Principle

A longitudinal view image of fibre snippets representative of a test specimen is magnified to an appropriate scale/size under optical microscope. All the fibre types found in the test specimen are identified by comparing them with known fibre surface morphologies for different types of animal fibres.

For each fibre type, the number and the diameter of the fibre snippets are counted and measured. The mass fraction is calculated from the data for the number of fibre snippets counted, mean value and standard deviation of the snippet diameter and the true density of each fibre type.

5 Reagents

5.1 Liquid paraffin, with a refractive index between 1,43 and 1,53.

6 Apparatus

6.1 Projection microscope, comprised of a light source, a light condenser, a stage, an objective, an ocular and a circular transparent viewing screen or non-transparent projection table with a graduated scale in millimetres. The objective and ocular shall be capable of providing at least a magnification of 500× at the screen. A stage micrometer shall be equipped to calibrate the magnification.

6.2 Visual microscopic image analyser, comprised of a microscope, a camera, a computer, a data acquisition card, exclusive analysing software and a display. The objective and ocular of the microscope shall be capable of providing at least a magnification of 500×. A stage micrometer shall be equipped to calibrate the magnification.

6.3 Transmitted-light type microscope, comprised of a light source, a light condenser, a stage, an objective, an ocular with a graduated scale. The objective and ocular of this type of microscope shall be capable of providing a magnification of 400× to 500×.

6.4 Microtome and razor blade, scalpel or double blades.

6.5 Scissors, tweezers, cleaning fabric, watch-glass, etc.

6.6 Slides and cover glasses.

6.7 Wedge scale, with divisions of 500× magnification. A moveable linear ruler-type scale finely graduated in millimetre may also be used.

7 Sampling

Lot samples and laboratory samples shall be drawn in accordance with the sampling methods described in Annex A.

8 Conditioning

The laboratory sample shall be conditioned for at least 4 h under the standard atmospheres as defined in ISO 139.

9 Preparation of test specimens

9.1 Number of test specimens

Prepare two sets of test specimens (see 9.2.1.3).

Fibres shall be sufficient to ensure a total of at least 1 000 fibres to be identified, whatever the number of operators.

In case of discrepancy on the test results between the two sets, a third set of test specimen shall be prepared and tested.

9.2 Preparation method for test specimens

9.2.1 Loose fibre

9.2.1.1 Put the laboratory sample flat on the test table, pick up approximately 500 mg of fibres randomly on not less than 20 spots with tweezers from the top and bottom sides of the sample. Blend them homogeneously and divide them into 3 equal portions. Sort these drawn fibres into basically parallel fibre bundles.

9.2.1.2 Cut each fibre bundle in the middle with a microtome and razor blade, scalpel or double blades to get approximately 0,6 mm long fibre snippets. Cut only once in each of the fibre bundles.

9.2.1.3 Place all the fibre snippets on the watch glass, drop appropriate amount of liquid paraffin, stir with tweezers to make the suspended snippet liquid distribute uniformly on the watch glass, then take an appropriate amount of this test specimen blend and put on the slide, cover with a cover glass. Remove redundant sticky media blends to ensure no such media blends are squeezed out after the cover glass is put on the slide to avoid fibre snippet loss. To facilitate test, the test specimen can be prepared on one slide with two cover glasses on it, however, ensure that there are at least 500 fibre snippets under each cover glass. Or other test specimen preparation mode is acceptable ensuring that at least 1 000 fibre snippets can be tested.

9.2.2 Sliver

9.2.2.1 Cut the laboratory sliver sample into three sections. Take out an appropriate amount of fibre bundle in the longitudinal direction from each sliver section.

9.2.2.2 Cut in the middle of each fibre bundle to obtain approximately 0,6 mm long fibre snippets with microtome and razor blade, scalpel or double blades. Cut only once in each fibre bundle.

9.2.2.3 Other operation procedures are the same as described in 9.2.1.3.

9.2.3 Yarn

9.2.3.1 Divide the laboratory sample into 3 equal portions.

9.2.3.2 Cut each portion in the middle with a microtome and razor blade, scalpel or double blades to obtain approximately 0,6 mm long fibre snippets. Cut only once in each yarn portion.

9.2.3.3 Other operation procedures are the same as described in 9.2.1.3.

9.2.4 Woven fabrics

9.2.4.1 If the warp and weft yarn share the same composition, all yarn segments unravelled from a rectangular sample of a complete pattern may be cut to obtain an appropriate test specimen. For those fabric samples composed of different compositions of warp and weft yarns, unravel the warp and weft yarns separately, weigh them and record their masses as m_T and m_W, respectively. If the fabrics have a definite repetition in the pattern, unravel at least the integral multiple of a complete pattern. The unravelled warp and weft yarn bundles are kept as warp and weft yarn samples respectively as the laboratory sample.

9.2.4.2 Cut from the parallel yarn portion in the middle with a microtome and razor blade, scalpel or double blades to obtain approximately 0,6 mm long fibre snippets. Cut only once in each yarn portion.

9.2.4.3 Other operation procedures are the same as defined in 9.2.1.3.

9.2.5 Knitted fabrics

9.2.5.1 Unravel at least 25 yarn segments from the laboratory sample for woollen knitted fabrics. Unravel at least 50 yarn segments for worsted knitted fabrics. Cut each yarn portion in the middle to obtain approximately 0,6 mm long fibre snippets. Cut only once in each yarn portion.

9.2.5.2 Other operation procedures are the same as described in 9.2.1.3.

9.3 Pre-treatment of the laboratory sample

9.3.1 If, prior to analysis, Soxhlet extraction in light petroleum (boiling point between 40 °C and 60 °C) is carried out to remove excess surface greases or oils, it shall be reported.

9.3.2 If a decolouring process is carried out on those dark laboratory samples for which it is difficult to see the fibre morphology, prepare the test specimens according to requirements in 9.2. The decolouring process shall be reported. Decolouring methods are given in Annex B.

NOTE The decolouring process can lead to fibre diameters measured from the decoloured fibre different from those diameters measured from the original fibres taken from fabric or yarns prior to decolouring.

10 Procedure

10.1 General

When possible, the analysis of the two test specimens should be carried out independently by two operators.

10.2 Setting of magnification with micrometer scale

Put the micrometer with a 0,01 mm scale on the stage. The 20 scales from the micrometre (0,20 mm) projected on the screen shall be precisely magnified to 100 mm which means the magnification is 500×.

10.3 Fibre identification and fibre diameter measurement

10.3.1 Projection microscope with graduated scale in millimetre on the screen

10.3.1.1 Set the magnification according to 10.2, put the slide with fibre snippets to be tested on the stage, adjust the focus under stipulated magnification to the most proper resolution, scan the slide in a raster pattern to ensure that all parts of the slide are covered and avoid the possibility of any fibre being measured twice. Observe in a proper order the various fibres into the view by comparing fibre morphology of fibres in test with those shown in Annex C and by combining with other features of various fibre types, such as fibre axial evenness, fibre lustra, fibre regularity, etc. Qualitatively identify and record fibre type (s) observed during the test.

10.3.1.2 If more than one types of fibres are found in the test specimen slide, observe and measure the diameters of various types of fibres into the view, measure the diameters of at least 100 fibres for cashmere and wool and at least 150 fibres for other speciality animal fibres, record the number of different types of fibres respectively.

If the number of fibres identified reaches 1 000 while the measurement is still being carried out in the middle of the slide, keep moving and counting until the end of the slide. For fibre types in which only a

minor proportion is blended into and the number of fibres measured fails to meet the requirement of number for fibre diameter measurement, measure all fibres of such type found in the specimen slide.

10.3.1.3 For those fibres observed with diameter exceeding 30 µm for angora or rabbit, record as coarse angora or coarse rabbit. Measure the fibre diameter and record the number.

10.3.1.4 If a measurement falls between two divisions, take the lower of the two values.

10.3.1.5 Calculate the mean fibre diameter and standard deviation for a given component according to Formulae (1) and (2), respectively.

$$\bar{d} = \frac{\sum (d \times F)}{\sum F} \tag{1}$$

$$S = \sqrt{\frac{\sum F(d - \bar{d})^2}{\sum F}} \tag{2}$$

where

$\bar{d}$ is the mean fibre diameter of the component, in micrometres (µm);

d is the group diameter, d = (recorded group value + 0,5) x 2, in micrometres (µm);

F is the number of fibres measured with the same diameter;

S is the standard deviation, in micrometres (µm).

10.3.2 Projection microscope used to measure the fibre diameter with wedge scale or a transparent moveable linear-rule-type scale

10.3.2.1 Measurement is made by moving the wedge scale with its length at right angles to the fibre image until a division coincides with one edge of the focused fibre image. The width of the fibre image is read off on the other edge of the wedge scale. When measuring an image whose edges are not in focus together, adjust the focusing so that one edge is in focus when a fine line appears, and the other edge shows a white line. Measure the width from the edge that is in focus to the inside of the white line.

10.3.2.2 If the width of a fibre image coincides with wedge scale division and lies exactly on a millimetre division of *N*, the width of the measured fibre image may be assigned to either data group *N–1* or *N+1* depending on actual conditions, if such cases reoccur, alternately assign them to data group *N–1* and data group *N+1*.

10.3.2.3 Other operation procedures are the same as defined in 10.3.1.1 to 10.3.1.3.

10.3.2.4 The mean fibre diameter and standard deviation of a given component is calculated using Formulae (3) and (4), respectively.

$$\bar{d} = \frac{\sum (A \times F)}{\sum F} \tag{3}$$

$$S = \sqrt{\frac{\sum F\left(A - \bar{d}\right)^2}{\sum F}} \tag{4}$$

where

$\overline{d}$ is the mean fibre diameter of the component, in micrometres (µm);

A is the median, in micrometres (µm);

F is the number of fibres measured;

S is the standard deviation, in micrometres (µm).

10.3.2.5 Fibre diameter measurement operation with rule-type scale and calculation are the same as defined in 10.3.1.

10.3.3 Visual microscopic image analyser

10.3.3.1 Observe various type of fibres into the screen view, measure the fibre diameter when edges of fibre in focus show clear fine lines, move the cursor to the one side of the focused fibre, click the left mouse button, then move the cursor to the other side of the focused fibre. Click the left mouse button again, the fibre diameter value will be automatically recorded after measurement. Test result will be automatically calculated and recorded in the report sheet.

10.3.3.2 Other procedures are the same as defined in 10.3.1.1 to 10.3.1.3.

10.3.4 Transmitted-light type microscope

Proceed as described in 10.3.1 but with measuring using the graduated scale of the ocular.

11 Calculation and expression of test result

11.1 Calculation of test result

11.1.1 Calculate the mass fraction of each component with Formula (5). Density of various types of animal fibres shall be as specified in Annex D.

$$w_i = \frac{N_i\left(D_i^2 + S_i^2\right)\rho_i}{\sum\left[N_i\left((D_i^2 + S_i^2)\rho_i\right)\right]} \times 100 \tag{5}$$

where

w_i is the mass fraction of the component, %;

N_i is the number of fibres counted for the component;

S_i is the standard deviation of mean fibre diameter of the component, in micrometres (µm);

D_i is the mean fibre diameter of the component, in micrometres (µm);

ρ_i is the density of the component, in grams per millilitre (g/cm^3).

11.1.2 Calculate the mass fraction of a fibre component in woven fabric samples composed of different warp and weft yarn compositions with Formula (6).

$$w_i = \frac{w_{iT} \times m_T + w_{iW} \times m_W}{m_T + m_W} \times 100 \tag{6}$$

where

w_i is the mass fraction of the component in woven fabric sample, %;

w_{iT} is the mass fraction of the component in the warp yarns of the woven fabric sample, %;

m_T is the mass of the warp yarns in the woven fabric sample, in grams (g);

w_{iW} is the mass fraction of the component in the weft yarns of the woven fabric sample, %;

m_W is the mass of the weft yarns in the woven fabric sample, in grams (g).

11.2 Expression of test result

Take the mean value of calculations of the two sets of test specimens as the test result. If the difference between the two sets of test specimens is larger than 3,0 %, a third set of test specimens shall be tested. In such a case, the mean value of the three test results is taken as the test result. Fibre content percentage of angora or rabbit is the sum of percentages of both fine and coarse angora or rabbit hairs.

Test result of fibre content is rounded to one decimal.

12 Test report

Test report shall at least include the following information:

a) sample description;

b) a reference to this document, i.e. ISO 17751-1:2023;

c) the method used for fibre identification and fibre diameter measurement;

d) pre-treatment, if any (Soxhlet extraction, decolouration treatment, etc.);

e) mass fraction of each component, expressed in percentage;

f) any deviations from the procedure;

g) any unusual features observed;

h) the date of the test;

Annex A
(normative)

Drawing of the lot sample and the laboratory sample

A.1 Loose fibre

Fifty percent of the total number of packages should be sampled. Take out a bundle of fibres from at least three parts of each package. After blending them homogeneously, divide the sample into two equal portions, one portion randomly selected is retained and the other is rejected.

After mixing the retained portion to ensure it is homogenized, divide it again into two equal portions in the same way. Reject one portion (select at random).

Continue the subdivision procedure until about 20 g of fibres remain; this is the lot sample.

Divide the 20 g of fibre sample into two portions. Use one portion as the laboratory sample and retain the other as a spare sample.

A.2 Sliver

Randomly take altogether four different 30 cm long slivers from four different ball tops or sliver cans. Strip each of the four slivers in its longitudinal direction to form another sliver, which is the laboratory sample. Retain the remaining portions as spare samples.

A.3 Yarn

Take twenty 20 cm long woollen yarn segments from each of five different cones or skeins to obtain 100 woollen yarn segments.

Take twenty 20 cm long worsted yarn segments from each of ten different cones or skeins to obtain 200 worsted yarn segments.

Cut the yarn bundle in the middle to get two portions – use one portion as the laboratory sample and retain the other as a spare sample.

A.4 Woven fabrics

Take three samples, each measuring 5 cm x 10 cm (warp x weft). Samples shall be taken from the fabric roll ensuring they are at least 100 mm from the cut edge, and at least 150 mm from the selvage and shall be spaced along a diagonal of the fabric to allow for representation of different warp and weft yarns. For each sample, mark its warp and weft directions respectively. Cut at least the integral multiple of a complete pattern in the case of fabrics where there is a definite repetition of the pattern. Cut along the weft direction from the middle of each fabric sample and divide it into two portions. Use one portion as the laboratory sample and retain the other as a spare sample.

A.5 Knitted fabrics

Take three samples, each measuring 5 cm × 10 cm (course × wale). Avoid rib sections such as cuff or bottom parts. Cut each sample from the middle along longitudinal direction into two portions – use one as the laboratory sample and retain the other as a spare sample.