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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.

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.

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

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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.

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

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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

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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.

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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.

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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
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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

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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

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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.

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6 Apparatus and reagents

6.1 Light microscope

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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.

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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.

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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.

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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).

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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.

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