

SLOVENSKI STANDARD SIST EN ISO 17751-2:2023

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Tekstilije - Kvantitativna analiza kašmirskih, volnenih, drugih specialnih živalskih vlaken in njihovih mešanic - 2. del: Metoda štetja z elektronskim mikroskopom (ISO 17751-2:2023)

Textiles - Quantitative analysis of cashmere, wool, other specialty animal fibres and their blends - Part 2: Scanning electron microscopy method (ISO 17751-2:2023)

Textilien - Quantitative Analyse von Kaschmir, Wolle, anderen speziellen tierischen Fasern und deren Mischungen - Teil2: Rasterelektronenmikroskopie-Verfahren (ISO 17751-2:2023)

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Textiles - Analyse quantitative du cachemire, de la laine, d'autres fibres animales spéciales et de leurs mélanges - Partie 2: Méthode par microscopie électronique à balayage (ISO 17751-2:2023)

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Textiles - Quantitative analysis of cashmere, wool, other specialty animal fibres and their blends - Part 2: Scanning electron microscopy method (ISO 17751-2:2023)

Textiles - Analyse quantitative du cachemire, de la laine, d'autres fibres animales spéciales et de leurs mélanges - Partie 2: Méthode par microscopie électronique à balayage (ISO 17751-2:2023) Textilien - Quantitative Analyse von Kaschmir, Wolle, anderen speziellen tierischen Fasern und deren Mischungen - Teil2: Rasterelektronenmikroskopie-Verfahren (ISO 17751-2:2023)

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European foreword

This document (EN ISO 17751-2:2023) has been prepared by Technical Committee ISO/TC 38 "Textiles" in collaboration with Technical Committee CEN/TC 248 "Textiles and textile products" the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by March 2024, and conflicting national standards shall be withdrawn at the latest by March 2024.

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INTERNATIONAL STANDARD

ISO 17751-2

Second edition 2023-08

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

Part 2: Scanning electron microscopy method

Textiles — Analyse quantitative du cachemire, de la laine, d'autres fibres animales spéciales et de leurs mélanges — Ste Partie 2: Méthode par microscopie électronique à balayage

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Foreword

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

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO 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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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

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 17751-2:2016), which has been technically revised.

The main changes are as follows:

- in <u>3.1</u>, a note to different types of speciality animal fibres has been added;
- in <u>3.6</u>, a note to entry and a new <u>Figure 1</u> have been added to indicate the distal edge, and subsequent figures have been renumbered;
- a new term, <u>3.11</u> warping angle, has been added;
- a new <u>Clause 5</u> titled "reagents and materials" and its content has been separated from former clause;
- a new <u>Clause 6</u> titled "Apparatus" has been added and its contents have been renumbered, subsequent clause and subclause numbers are changed accordingly;
- <u>Clause 7</u> retitled "Sampling" has been added and its content has been rephrased to match with the property adjustment of <u>Annex A</u>;
- in <u>8.1</u>, the numbers of test specimen sets and test specimen stubs have been increased;
- the title of <u>8.2</u> (former 6.2) has been changed from "Preparation method for test specimens of various types of samples" to "Preparation method for test specimens";

- in <u>8.2.4.1</u>, missing information on marking of masses of warp and weft yarns and laboratory sample has been supplemented;
- the title of <u>Clause 9</u> has been changed to "Procedure";
- <u>9.1</u> titled "General" and its content has been added;
- the title of <u>9.2</u> has been changed from "Test on each test specimen stub" to "Preparation and test on test specimen stubs";
- the title of <u>9.3</u> has been changed from "Qualitative analysis (Purity analysis) and determination of fibre content" to "Qualitative analysis (Purity analysis)";
- <u>9.4</u> titled "Quantitative analysis" has been added, number of fibre snippets to be examined and measured are changed due to the change of number of test specimen stubs;
- the title of <u>Clause 10</u> has been changed from "Calculation of test result" to "Calculation and expression of test result";
- <u>10.1</u> "Calculation of test result" has been added;
- <u>10.2</u> "Expression of test result" has been added;
- <u>Clause 11</u> titled "Test report" and its contents have been added;
- the status of <u>Annex A</u> has been changed from informative to normative;
- in <u>Annex C</u>, density of some fibres has been modified and the density of coarse rabbit has been added;
- in <u>Annex C</u>, a table footnote has been added to coarse angora or rabbit;
- two references have been added in the bibliography. 2023

A list of all parts in the ISO 17751 series can be found on the ISO website. 2-121407a4dab0/sist-

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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 textual descriptions, micrographs of different types of animal fibres are given in <u>Annex B</u>.

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

Part 2: Scanning electron 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 scanning electron microscopy (SEM).

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

2 Normative references

There are no normative references in this document.

3 Terms and definitions and ards.iteh.ai)

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 or rabbit, alpaca, etc.

3.2

scanning electron microscope

intermediate type of microscopic morphology observation instrument between transmitted electron microscope and light microscope which use a focused beam of high-energy electrons to generate a variety of physical information signals

Note 1 to entry: The principle consists of scanning a primary focused electron beam over a whole area of interest on the surface of solid test specimen, and the signal derived from which is then received, amplified and displayed in images for full observation of surface area topography of the test specimen.

Note 2 to entry: The signals obtained by a scanning electron microscope are, e.g. secondary electrons, Auger electrons, characteristic X-ray, etc.

3.3

secondary electron

low-energy extra-nuclear electron released from and by ionization of a metal atom in the 5 nm to 10 nm scanned region of metal layer less than 10 nm thick nearest to the outermost meta-coated surface of a *test specimen* (3.10) under impact of the focused primary electron beam of energy in units of tens of keV

Note 1 to entry: Being surface sensitive because of the small mean free path of the electron to escape from deep within the test specimen and, therefore the signal of which produces the highest-resolution morphological images of the coated surface.

3.4

scale

cuticle covering the surface of animal fibres

3.5

scale frequency

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

3.6

scale height

height of the cuticle at the *scale's* (3.4) distal edge

Note 1 to entry: The distal edge is shown in <u>Figure 1</u>.



Кеу

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1 distal edge//standards.iteh.ai/catalog/standards/sist/73e7a09f-006f-44a7-a152-f21407a4dab0/sist-

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Figure 1 — Distal edge

3.7

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.

3.8

lot sample

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

3.9

laboratory sample

portion drawn from a *lot sample* (3.8) according to the requirements to prepare *test specimens* (3.10)

3.10

test specimen

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

3.11

warping angle

angle of the free edge of the *scale* (3.4) deviating from the parallel edges of the fibre

4 Principle

A longitudinal view image of fibre snippets representative of a test specimen coated with a thin layer of gold and/or other metals is produced by a scanning electron microscope through scanning the side surface of the test specimen with a focused incident beam of high-energy electrons, detecting signals of secondary electrons emitted by the gold atoms excited when hit by the incident electron beam, and combining the beam position with the detected signals which contain information on surface topography of the test specimen.

All 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 diameter of 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 and materials

- **5.1** Acetone, analytical grade.
- **5.2 Ethyl acetate**, analytical grade.
- 5.3 Double-sided adhesive tape.

6 Apparatus

6.1 Scanning electron microscope, comprised of a vacuum system, electronic optical system, signal collecting and imaging system, display system, and measurement software.

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- **6.2 Sputter coater,** with a gold and/or other metal cathode.
- 6.3 Microtome and razor blade, scalpel or double blades.
- **6.4 Glass plate**, measuring approximately 150 mm × 150 mm.
- 6.5 Tweezers, scissors.
- **6.6 Test specimen stub**, aluminium or brass, 13 mm in diameter.
- 6.7 Glass tube, 10 mm to 15 mm in diameter.
- **6.8 Stainless-steel rod**, approximately 1 mm in diameter.

7 Sampling

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