# 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 —

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 <a href="www.iso.org/directives">www.iso.org/directives</a>).

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

The main changes are as follows:

- in 3.1, 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, 3.11 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";
- 9.1 titled "General" and its content has been added;
- the title of 9.2 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)";
- 9.4 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";
- 10.1 "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 Annex C, a table footnote has been added to coarse angora or rabbit;
- two references have been added in the bibliography.

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 <a href="https://www.iso.org/members.html">www.iso.org/members.html</a>.

#### 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 Annex B.

# 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

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 <a href="https://www.iso.org/obp">https://www.iso.org/obp</a>
- IEC Electropedia: available at <a href="https://www.electropedia.org/">https://www.electropedia.org/</a>

#### 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

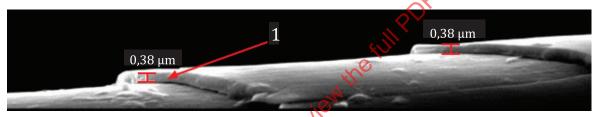
number of scales (3.4) along the fibre axis per unit length

#### 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 Figure 1.



#### Key

1 distal edge

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.

an va 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.

## **Reagents and materials**

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

## 6 Apparatus

- **Scanning electron microscope**, comprised of a vacuum system, electronic optical system, signal collecting and imaging system, display system, and measurement software.
- **Sputter coater,** with a gold and or other metal cathode. 6.2
- Microtome and razor blade, scalpel or double blades. 6.3
- 6.4 **Glass plate**, measuring approximately 150 mm × 150 mm.
- 6.5 Tweezers, scissors.
- **Test specimen stub**, aluminium or brass, 13 mm in diameter. 6.6
- Glass tube, 10 mm to 15 mm in diameter. 6.7
- **Stainless-steel rod**, approximately 1 mm in diameter. 6.8

#### 7 Sampling

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

#### 8 Preparation of test specimens

#### 8.1 Number of test specimens

One single set of test specimens is composed by 3 test specimen stubs and at least 600 fibres.

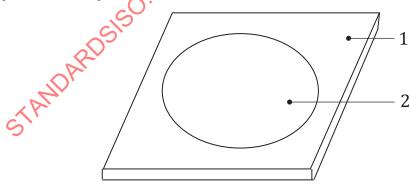
Prepare two sets of test specimens, each comprising 3 stubs, i.e. 6 test specimen stubs in total. Fibre snippets on a single set of test specimen stubs shall be sufficient to ensure at least 600 fibre snippets can be examined, for a total of 1 200 fibres (on 6 stubs) on two sets, whatever the number of operators.

In case of discrepancy on the test results between the two sets, a third set of test specimens (3 stubs and 600 fibres) shall be prepared and tested.

#### 8.2 Preparation method for test specimens

#### 8.2.1 Loose fibre

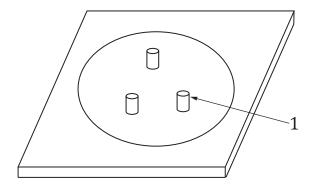
- **8.2.1.1** Put the laboratory sample flat on the test table, pick up approximately 500 mg of fibres randomly on not less than 20 spots with tweezers from the top and bottom sides of the sample. Blend them homogeneously and divide them into 3 equal portions. Sort these drawn fibres into basically parallel fibre bundles.
- **8.2.1.2** Cut each bundle in the middle with a microtome and razor blade, scalpel or double blades to get approximately 0,4 mm long fibre snippets. Cut only once in each of the fibre bundles.
- **8.2.1.3** Collect all fibre snippets in the glass tube and suspend them in 1 ml to 2 ml acetone or ethyl acetate by stirring the mixture with a stainless-steel rod. Pour the suspension onto a glass plate to ensure that the fibre snippets are uniformly distributed on a spot of approximately 10 cm in diameter on the glass plate, as shown in Figure 1.
- **8.2.1.4** Press the double-sided adhesive cape on the mounting stubs and use a razor blade to trim the adhesive tape away from around the mounting stubs. After all the acetone or ethyl acetate in the fibre snippets suspension has evaporated, press the mounting stubs with the adhesive tape end onto the glass plate at the positions shown in <u>Figure 2</u>. Transfer the uniformly mixed fibre snippets to the adhesive tape on the test specimen stub.



#### Key

- 1 glass plate
- 2 fibre suspension

Figure 2 — Fibre suspension on glass plate



#### Key

1 test specimen stub

Figure 3 — Positions of a single set of the three test specimen stubs

**8.2.1.5** If the fibre snippets have aggregated after the evaporation of the acetone or ethyl acetate, they shall be recollected by scraping them off the glass plate with a fazor blade and repeat operation procedures 8.2.1.3 and 8.2.1.4.

#### **8.2.2** Sliver

- **8.2.2.1** Cut the laboratory sliver sample into three sections. Take out an appropriate amount of the fibre bundle in the longitudinal direction from each sliver section.
- **8.2.2.2** Cut in the middle of each fibre bundle to obtain approximately 0,4 mm long fibre snippets with a microtome and razor blade, scalpel or double blades. Cut only once in each fibre bundle.
- **8.2.2.3** Other operation procedures are the same as described in 8.2.1.3 to 8.2.1.5.

#### 8.2.3 Yarn

- **8.2.3.1** Divide the laboratory sample into three equal portions.
- **8.2.3.2** Cut each portion in the middle with a microtome and razor blade, scalpel or double blades to obtain approximately 0,4 mm long fibre snippets. Cut only once in each yarn portion.
- **8.2.3.3** Other operation procedures are the same as described in <u>8.2.1.3</u> to <u>8.2.1.5</u>.

#### 8.2.4 Woven fabrics

- **8.2.4.1** 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  $m_{\rm T}$  and  $m_{\rm W}$ , 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.
- **8.2.4.2** Cut once from the parallel yarn portion in the middle with a microtome and razor blade, scalpel or double blades to obtain approximately 0,4 mm long fibre snippets. Cut only once in each yarn segments.

**8.2.4.3** Other operation procedures are the same as described in 8.2.1.3 to 8.2.1.5.

#### 8.2.5 Knitted fabrics

- **8.2.5.1** 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 0,4 mm long fibre snippets. Cut only once in each yarn portion.
- **8.2.5.2** Other operation procedures are the same as described in 8.2.1.3 to 8.2.1.5.

#### 8.3 Coating of the test specimens

Use the sputter coater to apply a thin layer of gold and/or other metals to the test specimen on test specimen stub.

#### 9 Procedure

#### 9.1 General

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

#### 9.2 Preparation and test on test specimen stubs

- **9.2.1** Place a stub with the test specimen into the test chamber of the SEM. First, view the selected stub at a lower magnification (for example, at  $10\times$ ). Then, selecting from an area near the upper left edge of the stub on the monitor, set the magnification to  $1\ 000\times$ , scan the stub and observe the fibres. The fibre types may be identified according to characteristics of the fibre morphologies (see details in Annex B) of cashmere, sheep's wool and other animal fibres.
- **9.2.2** Return to the lower magnification after identifying all fibres in the selected area. Choose another observation area along vertical or horizontal direction, repeat <u>9.2.1</u> operation until finished, scanning the entire stub before continuing to analyse fibre snippets on another stub.

#### 9.3 Qualitative analysis (purity analysis)

Examine 200 fibres on the first test specimen stub of the first set of test specimens. The following three conditions can happen.

- Case 1: If only one fibre type is found, examine another 200 fibre snippets on the first test specimen stub of the second set of test specimens. If no fibre of a second type is found, the sample is declared as pure.
- Case 2: If two fibre types are found and the amount of one type is lower than 3 % by number (less than 6 fibres of the second type), it is considered as a minor component. Examine 200 further snippets from the first test specimen stub of the second set of test specimens and calculate the percentage by number of the two types of fibres.
- Case 3: If two or more fibre types are found and the fibre mixture is considered to be a blend, perform a quantitative analysis according to 9.4.

#### 9.4 Quantitative analysis

If the sample is found to be a blend, examine 200 further fibres and measure the diameters of the first 25 fibres of each component identified (or all fibres of that component, if less than 25) on each of the remaining stubs of the first set of test specimens. At least a total of 600 fibres shall be identified for a

sample and 50 measurements of fibre diameter are made for each component. The mean fibre diameter of each component is calculated according to diameters measured for the 50 fibres. If the total amount of each component is less than 50, calculate the mean fibre diameter according to the actual number of that fibre component.

Repeat the procedure on the second set of test specimens for a total of  $1\,200$  fibres and 100 measurements of fibre diameter.

This diameter is measured in vacuum condition and is not comparable to diameter measured by other instruments. Therefore, the value shall only be used for calculation of fibre content of each component in Clause 10.

## 10 Calculation and expression of test result

#### 10.1 Calculation of test result

**10.1.1** Calculate the mass fraction of each component with Formula (1) for each set of test specimens. Density of various types of animal fibres shall be as specified in Annex (1)

$$w_i = \frac{N_i \left(D_i^2 + S_i^2\right) \rho_i}{\sum \left[N_i \left(\left(D_i^2 + S_i^2\right) \rho_i\right)\right]} \times 100 \tag{1}$$

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 ( $\mu$ m);

 $\rho_i$  is the density of the component, in grams per cubic centimetre (g/cm<sup>3</sup>).

**10.1.2** Calculate the mass fraction of a fibre component in woven fabric samples composed of different warp and weft yarn compositions with <u>Formula (2)</u> for each set of test specimens.

$$w_i = \frac{w_{iT} \times m_T + w_{iW} \times m_W}{m_T + m_W} \times 100 \tag{2}$$

where

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).

#### 10.2 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 specimen 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

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percentage of angora or rabbit is the sum of percentages of both coarse and normal angora or rabbit hairs.

Test result of fibre content is rounded to one decimal.

# 11 Test report

Test report shall at least include the following information:

- sample description;
- a reference to this document, i.e. ISO 17751-2:2023;
- test results; c)
- any deviations from the procedure; d)
- any unusual features observed;
- the date of the test. f)

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#### Annex A

(normative)

# Drawing of the lot sample and the laboratory sample

#### A.1 Loose fibre

Fifty percent of the total number of packages shall 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  $\times$  10 cm (warp  $\times$  weft). Samples shall be taken from the fabric roll ensuring they are at least 100 mm from the cut edge, and least 150 mm from the selvedge 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 as the laboratory sample and retain the other as a spare sample.

#### A.5 Knitted fabrics

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

# **Annex B**

(informative)

# Surface morphology of common animal fibres

#### **B.1** Cashmere from China

#### **B.1.1** Typical ring-shaped morphology

Consider Figure B.1, Figure B.2, Figure B.3, Figure B.4, Figure B.5, Figure B.6, Figure B.7, Figure B.8, Figure B.9 and Figure B.10 for fibre identification.

This cashmere shows high fibre diameter evenness in its axial direction and good lustre. The fibre scales are regular, most are ring-shaped and a few irregular ring-shaped; few changes can be seen. The scale envelopes the fibre shaft flatly and evenly; scales are thin with smooth surfaces. The distance between adjacent scales is large. The mean scale height is less than  $0.4~\mu m$ . The mean scale frequency is between 54~scales/mm and 64~scales/mm.



Figure B.1 Scales encircle fibre shaft flatly and regularly with regular patterns and smooth surface

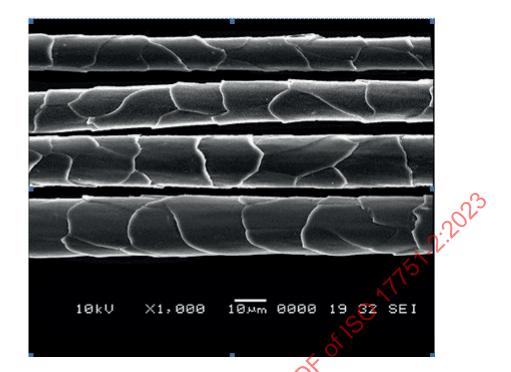


Figure B.2 — Some scale patterns are slightly irregular



Figure B.3 — Scale frequencies from low to high



Figure B.4 — Scale frequencies are slightly high

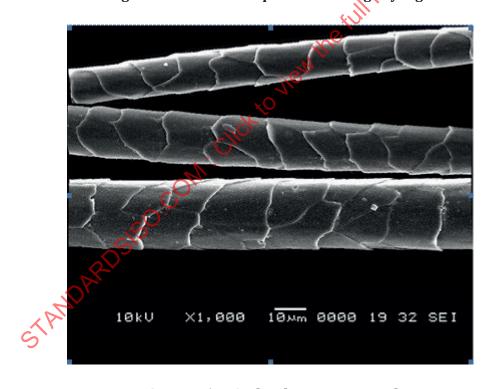


Figure B.5 — Scale edges are serrated

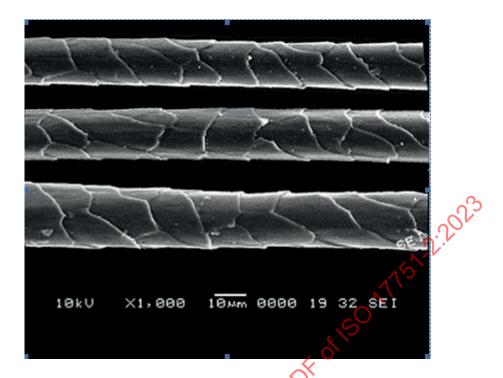


Figure B.6 — Scales are slightly slantwise and thick

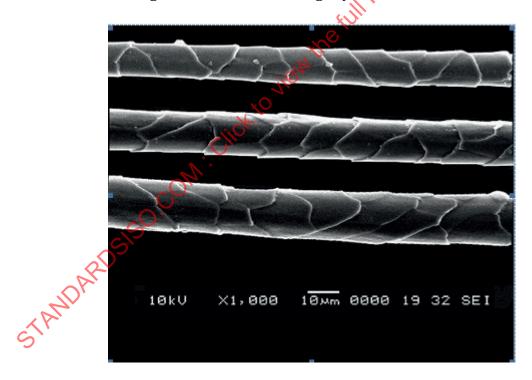


Figure B.7 — High scale frequency with irregular scale edges

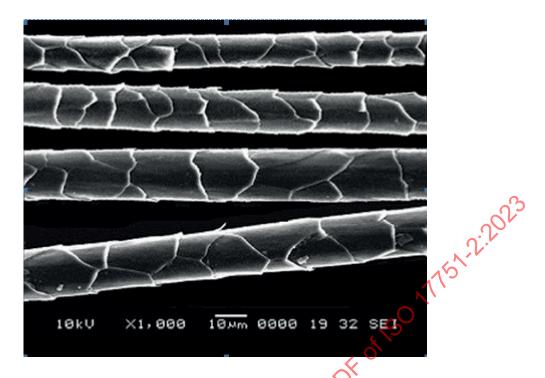


Figure B.8 — Scale edges slightly warpoutward

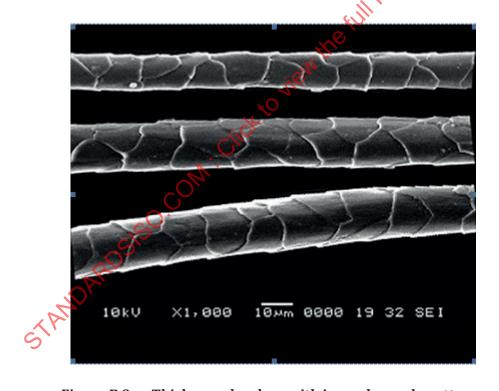


Figure B.9 — Thicker scale edges with irregular scale patterns



Figure B.10 — Thicker scale edges and higher scale frequency

#### **B.1.2** Irregular ring-shaped morphology

Consider Figure B.11, Figure B.12, Figure B.13 and Figure B.14 for fibre identification.

The scale morphology of this type is slightly different from that of a typical ring-shaped morphology. Some scale patterns are not regular, scale edges are not orderly, or scale edges are thick with high scale frequency; however, scales wrap around the fibre shaft flatly and orderly with smooth surfaces and high fibre evenness in its longitudinal direction.

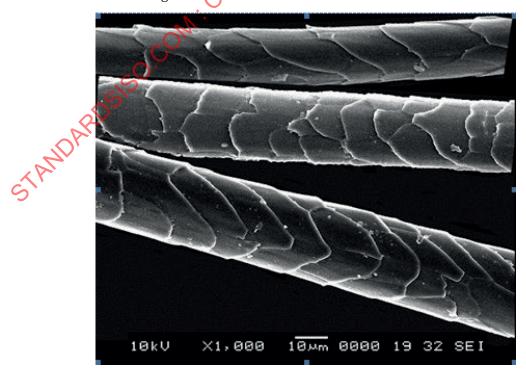


Figure B.11 — Higher fibre frequency with slightly slantwise scales

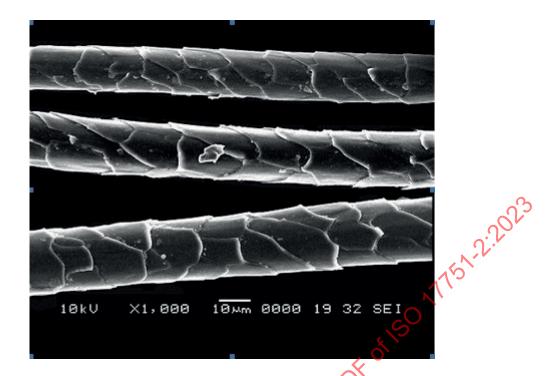


Figure B.12 — Larger scale height and higher scale frequency



Figure B.13 — Irregular scale patterns with scale edges warping outwards



Figure B.14 — Furrows on scale surface with serrated edges

#### **B.1.3** Morphology of variation cashmere fibres

Consider Figure B.15, Figure B.16, Figure B.17 and Figure B.18 for fibre identification.

The term "morphology of variation cashmere fibres" refers to fibre morphologies deviating from those of cashmere fibres and belonging to morphologies which are difficult to distinguish or easy to be misidentified as wools.

If cashmere fibres with such morphologies are encountered in the testing process, the following conditions should be taken into consideration.

- a) When testing pure cashmere samples: To determine whether fibre with such morphology is variation cashmere orblended wool is based on the condition that whether wool is blended into the samples.
  - 1) If wool is not artificially blended into the sample, fibres with variation morphologies should be identified as variation cashmere.
  - 2) If wool is deliberately blended into the sample, fibres with variation morphologies should be identified according to the corresponding characteristics of cashmere and wool including scale structure (scale frequency, scale height, scale patterns), longitudinal fibre evenness, fibre lustre etc. In some technical literature, a scale height of more than 0,5  $\mu$ m is more likely to be associated with wool while a scale height of less than 0,5  $\mu$ m is more likely to be associated with cashmere.
- b) When testing cashmere/wool blend samples. Identify in accordance with the mentioned principle of deliberate adulteration of sheep's wool to decide whether it's cashmere or sheep's wool.



Figure B.15 — Flowerpot-like scale patterns with high scale frequency resembling those characteristics of fine Merino wool

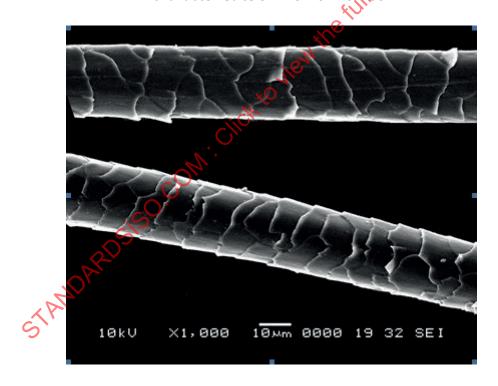


Figure B.16 — Scales are very thin but scale frequency is very high, belongs to transitional type from down cashmere to goat coarse hair

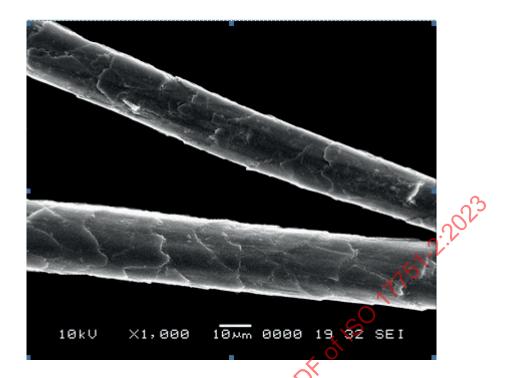


Figure B.17 — Scales shed to varying degrees, scale patterns are blurry

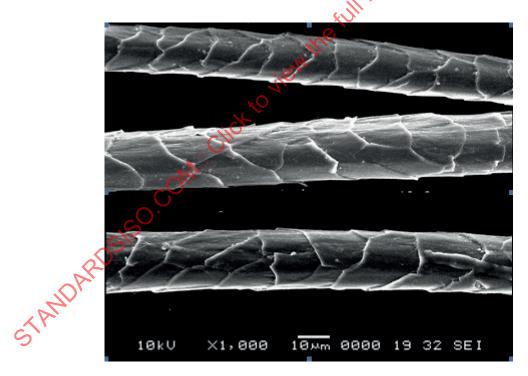


Figure B.18 — Thick scales enclose fibre shaft unevenly with furrows on fibre surface showing bad lustre resembling characteristics of Chinese native sheep's wool

#### **B.2** Cashmere from Mongolia

Consider Figure B.19, Figure B.20, Figure B.21, Figure B.22, Figure B.23, Figure B.24, Figure B.25, Figure B.26, Figure B.27 and Figure B.28 for fibre identification.

Almost all cashmeres from Mongolia are pigmented cashmere showing blurry scale patterns on the whole. Their fibre lustre is not as good as that of the Chinese cashmere. In all the batches of samples

from which these micrographs were taken, it was observed that the scales were partially broken and that scales warp outward. However, Mongolian cashmere shows more consistent fibre morphologies.

The mean scale height of the Mongolian cashmere is  $0.46 \, \mu m$  and the mean scale frequency is  $59.4 \, scales/mm$ .

No further classification of Mongolian cashmere fibre morphology types is made because the scale morphologies are relatively consistent. Micrographs are shown according to fibre morphologies from good to bad.

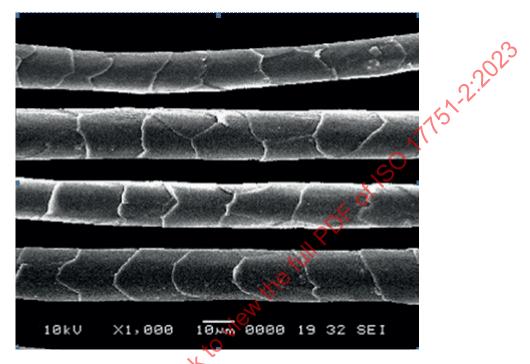


Figure B.19 — Orderly ring-shaped scales with thin and smooth edges, scale frequency is low

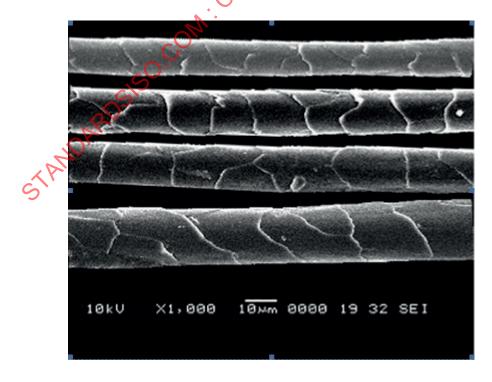


Figure B.20 — Scale edges are somewhat unsmooth



Figure B.21 — High scale frequency with untidy scale edges

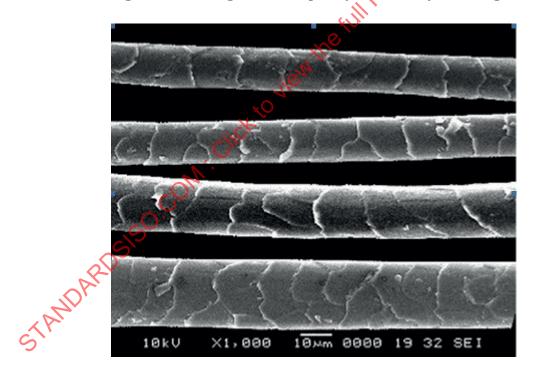


Figure B.22 — High scale frequency and untidy scale edges, fibre surfaces are not smooth



Figure B.23 — Blurry scales with untidy scale edges



 $Figure\ B.24-Blurry\ scales\ with\ untidy\ scale\ edges, fibre\ surfaces\ are\ not\ smooth$ 

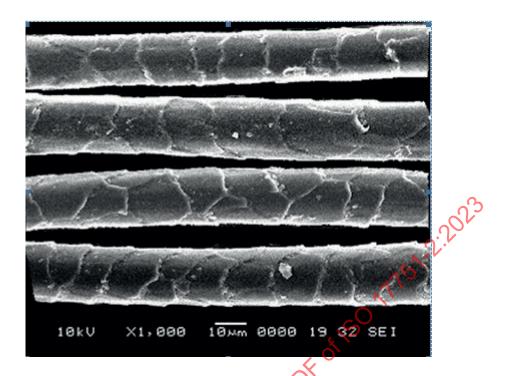


Figure B.25 — Blurry and unclear scales with untidy and thicker scale edges

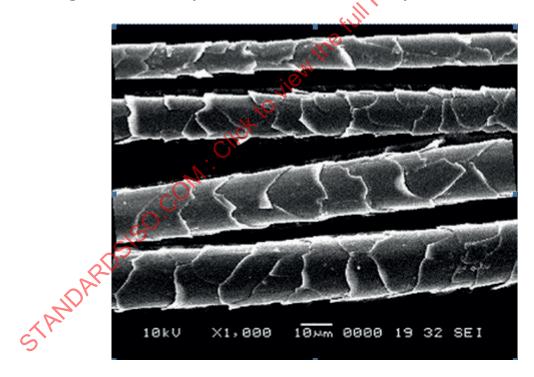


Figure B.26 — High scale frequency, disorder and bent scale edges

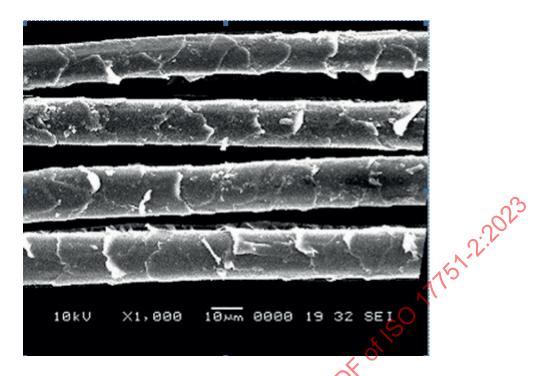


Figure B.27 — Damaged, stripped and warped scales

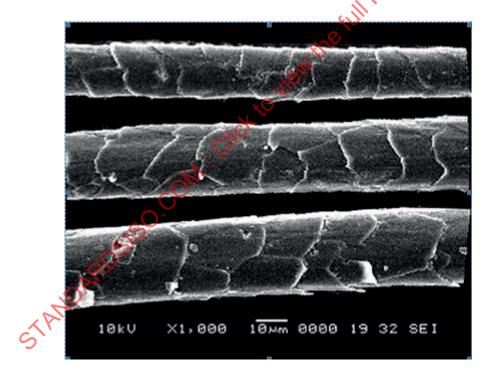


Figure B.28 — Rough scale surface and indistinctive ring-shaped scales with disorder scale margins. Scales are block-shaped on the whole

#### **B.3** Cashmere from Iran and Afghanistan

Consider Figure B.29, Figure B.30, Figure B.31, Figure B.32, Figure B.33, Figure B.34, Figure B.35, Figure B.36, Figure B.37, Figure B.38, Figure B.39 and Figure B.40 for fibre identification.

Cashmere from Iran and Afghanistan are basically pigmented cashmere with various colours. Scale morphologies of cashmere from such origins are better in comparison with those of cashmeres from

Mongolia. Cashmere from Iran and Afghanistan can be determined from the aspect of mean fibre diameter for the entire testing lot of materials. This is because mean fibre diameter of brown and grey cashmere from China is lower than that of white cashmere, normally less than 15  $\mu$ m. The mean fibre diameter of cashmere from Iran and Afghanistan is higher than 16  $\mu$ m. However, if these cashmeres are blended into cashmere from China, it's not easy to distinguish them.

The mean scale height of cashmere from Iran and Afghanistan is 0,43  $\mu$ m and the mean scale frequency is 62,6 scales/mm. As for the cashmere from Iran and Afghanistan, no classification according to fibre scale morphology types is made because the scale morphologies are relatively consistent. Micrographs are shown according to the fibre morphologies from good to bad.

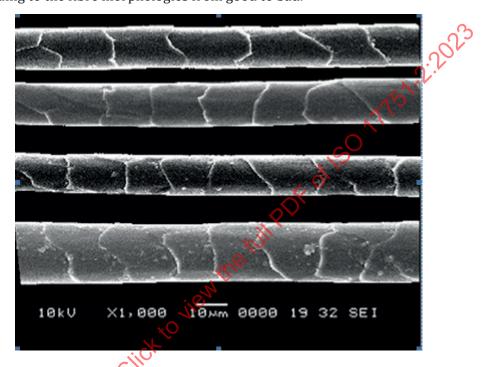


Figure B.29 — Regular ring-shaped scales with smooth and thin edges, scale frequency is low

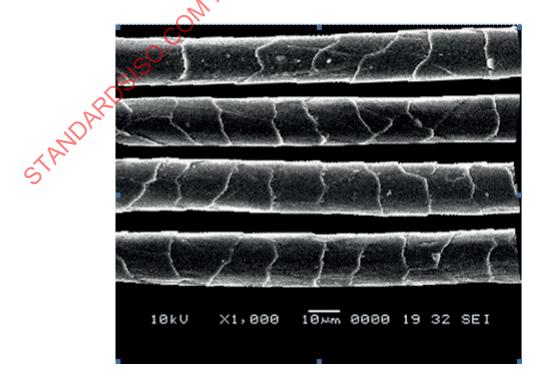


Figure B.30 — Regular ring-shaped scales with smooth edges

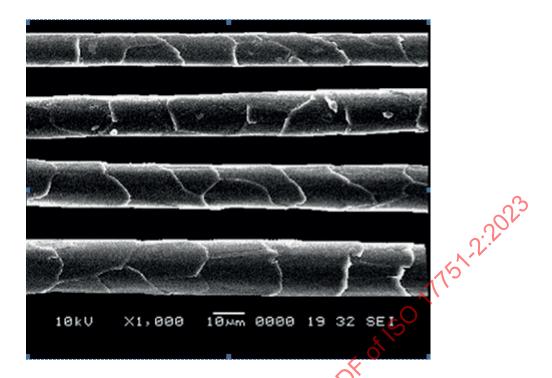


Figure B.31 — Slightly irregular scales with slightly low scale frequency

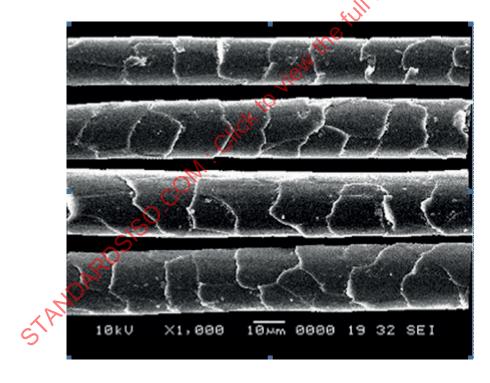


Figure B.32 — Slightly unsmooth scale surfaces with relatively high scale frequency

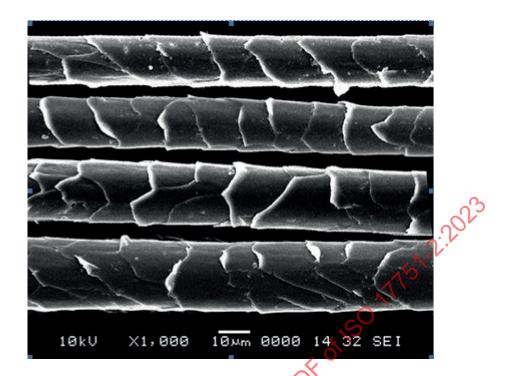


Figure B.33 — Slightly warping irregular scale edges

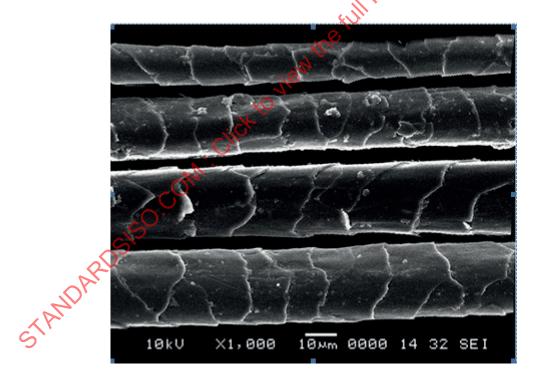


Figure B.34 — Unsmooth scale surface with thick scale edges



Figure B.35 — High scale frequency and slightly unsmooth scale surface

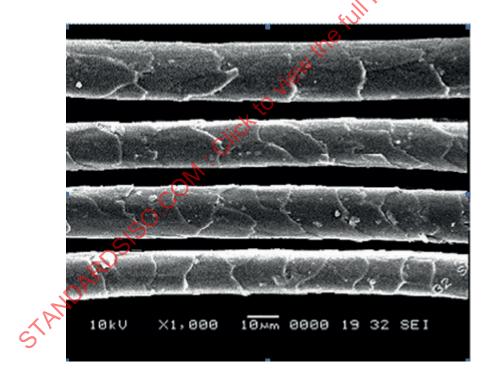


Figure B.36 — Slightly irregularly-arranged scale



Figure B.37 — Blurry and unclear scales

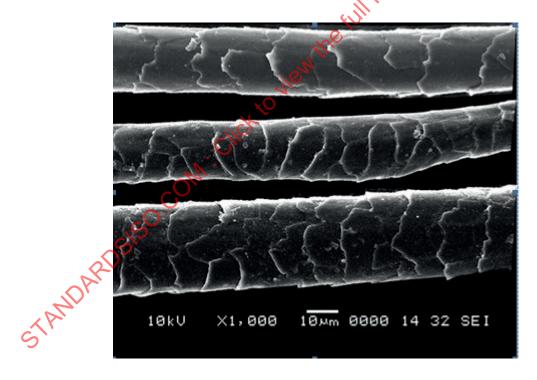


Figure B.38 — Serrated scale edges with high scale frequency

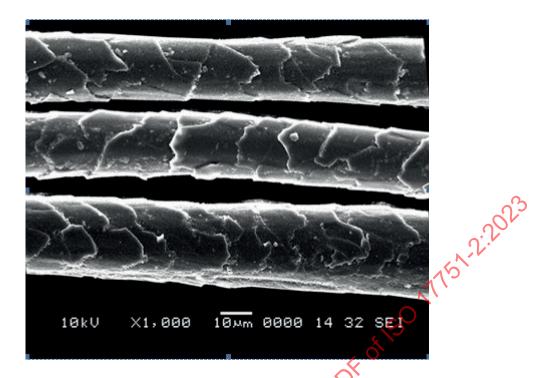


Figure B.39 — Blurry scales with unsmooth and irregular scale edges

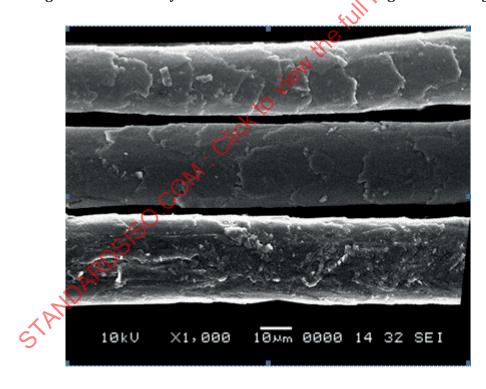


Figure B.40 — Fibres with shed scales

# B.4 Sheep's wool and modified sheep's wool

#### **B.4.1** Chinese native fine sheep's wool

Heterogeneous sheep's wools taken from Chinese native sheep without crossbreeding deviates largely in fibre diameter; coarse wool and fine wool can be separated using cashmere dehairing equipment and mean fibre diameter of fine wool, thus sorted, is between 19  $\mu$ m and 15  $\mu$ m; additional dehairing

leads to lower mean fibre diameter of this type of fine sheep's wool. This type of wool is often used in cashmere adulteration due to its easy access and its similarities in fibre morphology with cashmere.

The mean scale height of commonly used native fine sheep's wool is  $0.6 \mu m$  to  $0.7 \mu m$ , and mean scale frequency is between 59 scales/mm and 65 scales/mm.

Chinese native sheep's wool has a predominant feature of higher fibre evenness in its axial direction and lower scale frequency compared with Australian wool which are similar with features of cashmere, but it is still distinguishable by experienced fibre analysts. First, judge, as a whole, whether there is Chinese native fine wool blended into the sample; Second, when identifying fine wool fibres showing similar scale morphology as those in Group I: distinguish them by the aspects of fibre lustre and scale height if a light micrograph is used, or distinguish them by the aspects of scale surface smoothness and scale height if a scanning electron microscope is used. For fine wool fibres with the same scale morphologies as shown in group II to group V, misidentification rate is comparatively low due to the existence of more different judging elements.

Five groups are classified according to fibre scale morphologies from good to had.

— Group I: Consider <u>Figure B.41</u>, <u>Figure B.42</u> and <u>Figure B.43</u> for fibre identification. Fibre morphologies of native fine sheep's wool in Group I are basically the same as those of cashmere, which fibres show high diameter evenness in its axial direction with regular ring-shaped scales, scales envelop the fibre shaft regularly, but fibre lustre is a little worse, and scale height is higher than that of cashmere. The diameter of wools with such scale morphologies is basically below 15 μm.

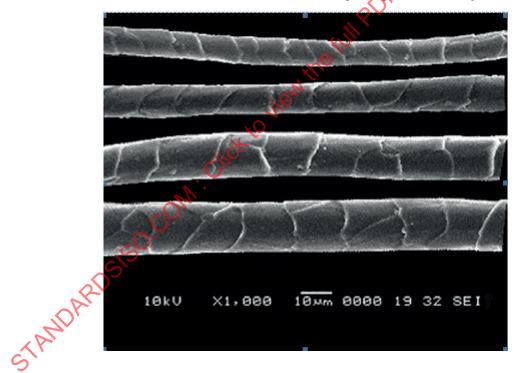


Figure B.41 — Ring-shaped scales encircle fibre shafts



Figure B.42 — Unsmooth fibre surfaces

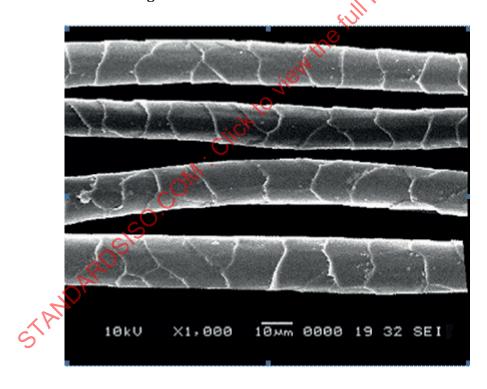


Figure B.43 — Bad fibre lustre

— Group II: Consider <u>Figure B.44</u>, <u>Figure B.45</u> and <u>Figure B.46</u> for fibre identification. Scales display irregular ring-shaped patterns. The fibre diameter is comparatively even in its axial direction. Wool fibres show very obvious differences from cashmere in the aspects such as less smooth scale surfaces, thicker scale heights, and higher scale frequency and larger warping angles of scale edges.

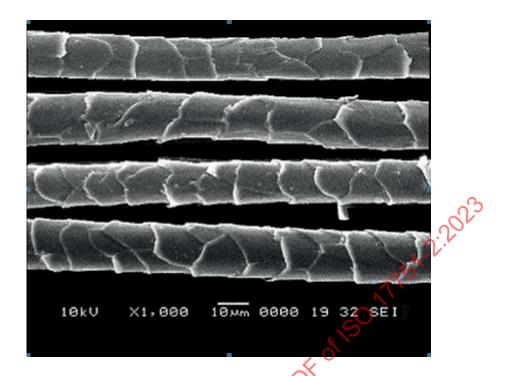


Figure B.44 — Thicker scale edges warping outside

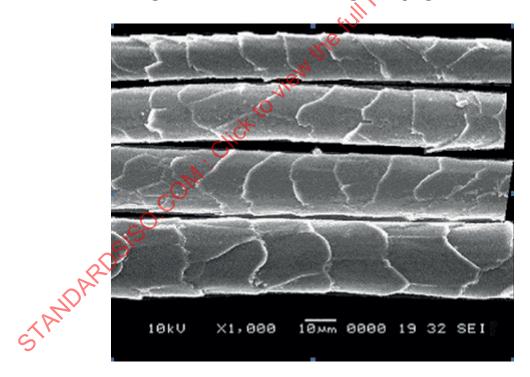


Figure B.45 — Unsmooth and irregular fibre surface

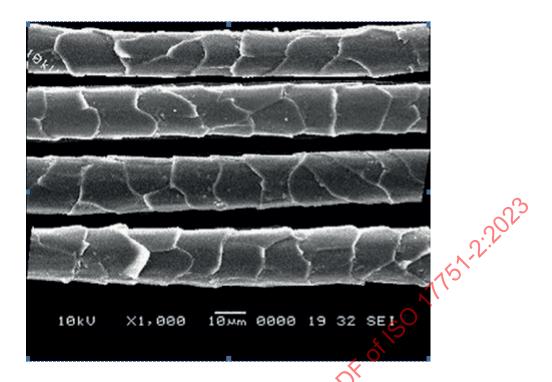


Figure B.46 — Higher scale frequency with thicker scale edges

— Group III: Consider <u>Figure B.47</u>, <u>Figure B.48</u> and <u>Figure B.49</u> for fibre identification. Fibres show higher evenness in its axial direction than the same diameter Australian wool. Fibres exhibit worse lustre. Scale edges have larger warping angles. Fibres show lower scale height, but higher scale frequency than fibres in Group II.

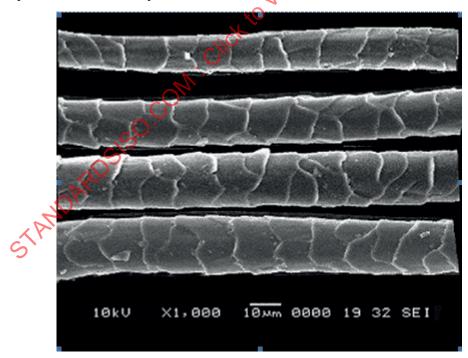


Figure B.47 — High scale frequency with thicker scale edges



Figure B.48 — High scale frequency with irregular scales

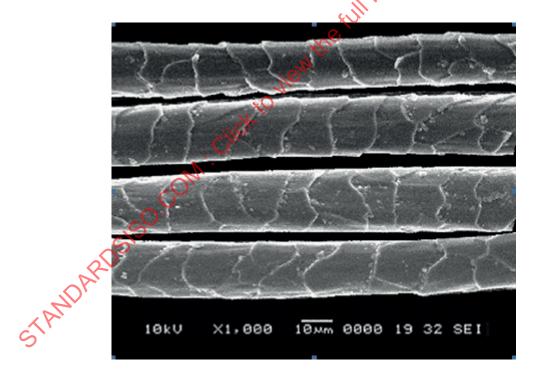


Figure B.49 — Rough scale surfaces

— Group IV: Consider <u>Figure B.50</u>, <u>Figure B.51</u>, <u>Figure B.52</u> and <u>Figure B.53</u> for fibre identification. Fibres show higher evenness in the axial direction than the same diameter Australian wool. Finer wools (with diameter below  $13~\mu m$ ) have low scale frequency with irregular scales. Coarser wools show tile or crack shaped patterns with rough scale surfaces and bad lustre, warping angles of scales are larger, partial scales shed from fibre shaft.

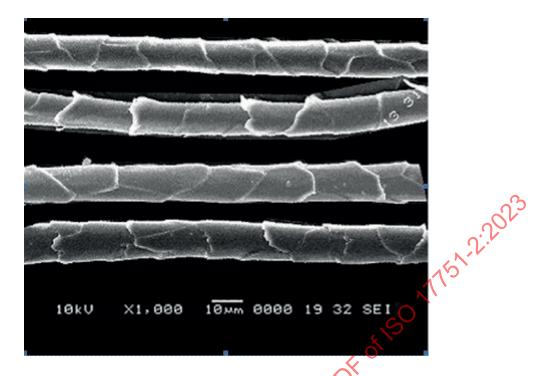


Figure B.50 — Scale frequency is low but scales are irregularly arranged

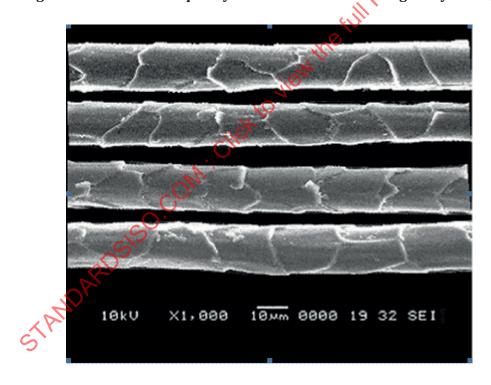


Figure B.51 — Untidy scale edges



Figure B.52 — Irregular scales

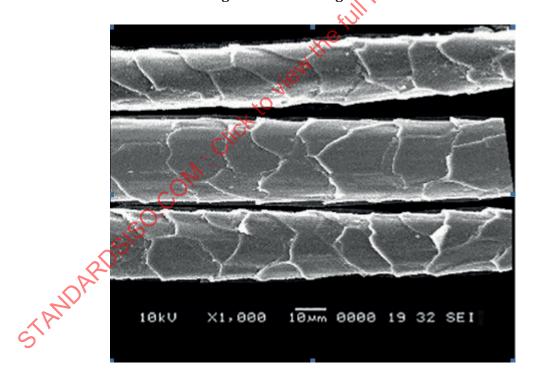


Figure B.53 — Thick scales and large scale frequencies

— Group V: Consider Figure B.54, Figure B.55, Figure B.56, Figure B.57, Figure B.58, Figure B.59 and Figure B.60 for fibre identification. Fibres show higher diameter evenness in the axial direction than the same diameter Australian wool. Scales show large variation on the tile or crack shaped patterns with large scale height. Fibre surfaces are rough with bad lustre. Striations and furrows are obvious and partial scales shed from the fibre shaft.

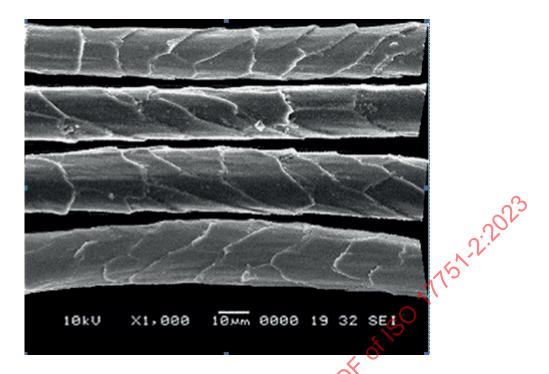


Figure B.54 — Rough surfaces with irregular scales

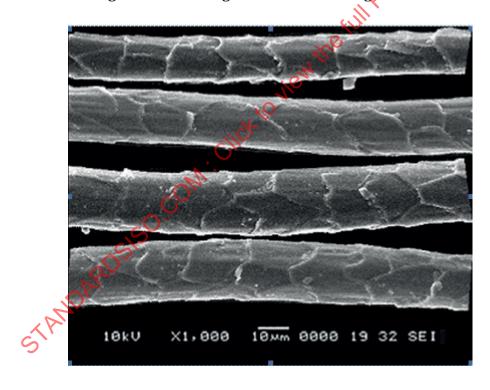


Figure B.55 — Thick scales with unclear scale edges

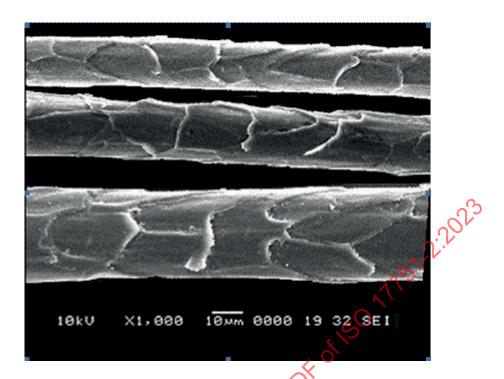


Figure B.56 — Very thick scales, rough surface with furrows

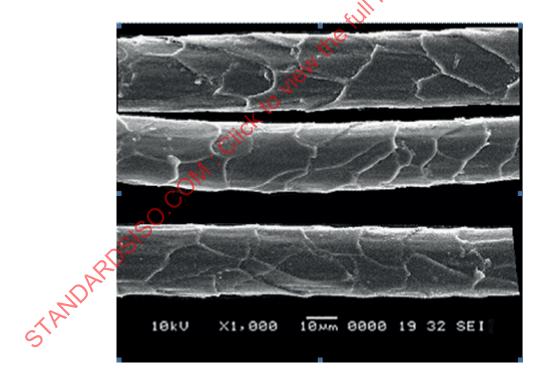


Figure B.57 — Disorderly scales, rough surface with furrows

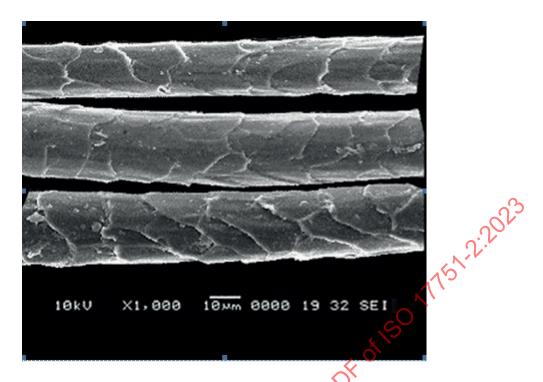


Figure B.58 — Disorderly scales, rough surfaces

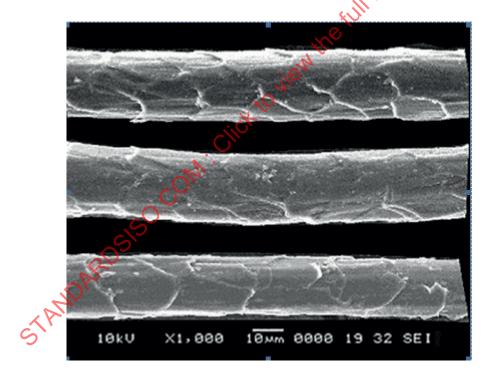


Figure B.59 — Blurry scales

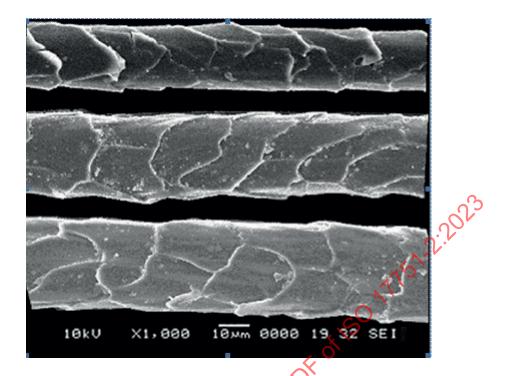


Figure B.60 — Thick scales, high scale frequency, rough fibre surface

## **B.4.2** Australian Merino wool

Consider Figure B.61, Figure B.62 and Figure B.63 for fibre identification.

Merino wool from Australia with a fibre diameter less than 18,5  $\mu m$  has a comparatively high yield. Such wools are often blended with cashmere to produce worsted products taking advantages of its large fibre length, low diameter and low coefficient of variation (CV %) of mean diameter. Typical characteristics of such wools are high fibre scale frequency and low fibre evenness in its axial direction which makes it easier to be distinguished from other wools if blended.

The mean scale height of commonly used superfine Merino wool is 0,60  $\mu$ m to 0,65  $\mu$ m. The mean scale frequency varies from 72 scales/mm to 79 scales/mm. There is little difference on scale morphologies between fine and coarse wools.

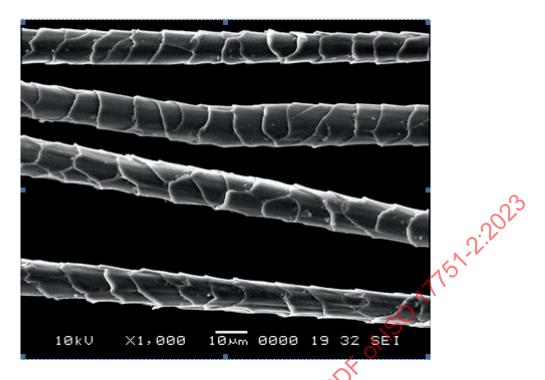


Figure B.61 — Fine wool from Australia

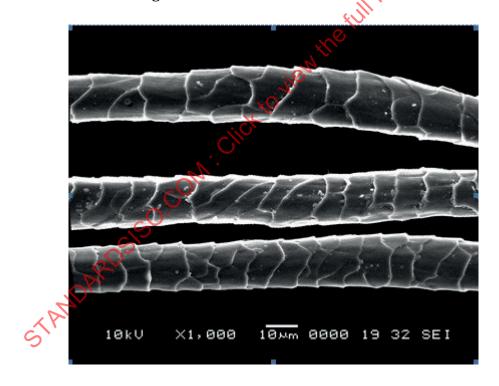


Figure B.62 — Medium wool from Australia