INTERNATIONAL STANDARD

ISO 20705

First edition
2019-12

Textiles — Quantitative microscopical analysis — General principles of testing

Textiles — Analyse quantitative par microscopie — Principes généraux des essais

Reference number
ISO 20705:2019(E)

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Published in Switzerland

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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 documents 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*.

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

This document is used for the quantitative analysis of textiles containing mixtures of fibres which cannot be separated readily by mechanical methods or by chemical methods, as described in the different parts of ISO 1833.

The quantitative microscopical analysis rely on the ability of a fibre analyst to identify and count, by means of a microscope [light microscope (LM) or scanning electron microscope (SEM)], the relative number of fibres of each type in a prepared test specimen (based on fibre apparent diameter of a longitudinal view or fibre section area of a cross view, depending on the fibre types).

Fibre counts lead to the calculation of the percentage in the mixture of the test specimen by number of fibres (based on fibre apparent diameter or fibre section area) and by their respective density. And then, the calculation of the fibre percentage by mass of the laboratory sample is carried out in relation to its structure (loose fibres, yarns, woven fabrics, knitted fabric, etc.).

Textiles — Quantitative microscopical analysis — General principles of testing

1 Scope

This document specifies common methods for the quantitative microscopical analysis of various mixtures of fibres. The methods described are based on the use of a light microscope (LM) or a scanning electronic microscope (SEM), on the measurements of the fibre apparent diameter (preparation of longitudinal views) or on the measurements of fibre section area (preparation of cross views), depending on the section shape of the fibres.

NOTE 1 When the section shape is circular or almost circular, the longitudinal views are appropriate. For the other section shapes, the cross views are adequate and Annex A lists conventional density of fibres to be used for the calculation of the mass percentage of the components. Pictures of section shapes of fibres can be found in ISO/TR 11827.

NOTE 2 Annex B presents statistical data on fibre diameter measurements (longitudinal view) and on fibre area measurements (cross view).

The given procedures apply to fibres in any textile form when mixtures of fibres cannot be separated by manual methods or by chemical methods.

Examples of mixtures of fibres are cashmere and wool, cotton and flax, flax and hemp.

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 1833-1, *Textiles — Quantitative chemical analysis — Part 1: General principles of testing*

3 Terms and definitions

For the purposes of this document, the following term and definition apply.

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

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

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

3.1
test specimen unit
linear portion of a single thread

Note 1 to entry: The length of the test specimen unit depends on the test specimen holder dimension.

Note 2 to entry: This expression is not applicable to test specimen prepared from samples of loose fibre (see 7.1.2) or sliver (see 7.1.3).

4 Principle

A longitudinal view image (respectively, a cross view image) of fibre snippets representative of a test specimen is magnified to an appropriate scale/size under optical light microscope or scanning

electron microscope. All fibre types found in the test specimens are identified by the difference in fibre morphology and are counted, measuring their individual apparent diameter (respectively section area). Including their respective density in the calculation, the percentage of the fibres in the mixture is determined by mass.

If it is practicable to chemically separate the components, the method described in the individual parts of ISO 1833 should be used in preference to the microscopical methods.

5 Apparatus

5.1 Transmitted-light type microscope, shall comprise a light source, a light condenser, a stage, an objective, an ocular with a graduated scale (eyepiece graticule or micron scale). The objective and ocular of this type of microscope shall be capable of providing a magnification of ×150 to ×500.

The stage is movable in two directions at right angles by means of a sliding mechanism capable of successive displacements in approximately 1,0 mm steps.

Alternatively, a projection light microscope (PLM) may be used.

NOTE A description of a PLM can be found in ISO 137.

5.2 Scanning electron microscope, shall comprise the following components: vacuum system, electronic optical system, signal collecting and imaging system, display system.

NOTE A description of a method for calibrating the magnification of images generated by a scanning electron microscope (SEM) using an appropriate reference material can be found in ISO 16700.

5.3 Tools.

5.3.1 Scissors, tweezers, dissecting needle, cleaning fabric, watch-glass, etc.

5.3.2 Slides and cover glasses.

5.3.3 Microtome.

6 Reagents

6.1 Neutral liquid medium, (e.g. liquid paraffin).

6.2 Resin, 2-hydroxyethyl methacrylate.

7 Preparation of the test specimens

7.1 Selection of the test specimens

7.1.1 General

Follow the general procedure described in ISO 1833-1, and then proceed as follows.

Take a laboratory test sample that is representative of the laboratory bulk sample and sufficient to provide all the specimens.

Fabrics may contain yarns of different composition and account should be taken of this fact in the sampling of the fabric.

Treat loose fibres as described in 7.1.2, slivers as described in 7.1.3, yarns as described in 7.1.4, and fabrics as described in 7.1.5.

7.1.2 Loose fibres

Put the laboratory sample flat on the test table. Pick up appropriate amount of fibres randomly on not less than 20 spots with tweezers from top and bottom sides of the sample.

Blend homogeneously and divide into two equal portions.

Sort those drawn fibres into two basically parallel fibre bundles, as the two "loose fibre" test specimens.

7.1.3 Slivers

Cut out two sections from the laboratory sliver sample, so that the length section is greater than the length of the test specimen holder (slide, SEM stub or tube).

Take out appropriate amount of fibre bundle in the longitudinal direction from each sliver section.

7.1.4 Yarns

Cut out two sections from the laboratory yarn sample, so that the length section is greater than the length of the test specimen holder (slide, SEM stub or tube).

For the structure of the yarn, if necessary, destructure each yarn section by subsequently untwisting the yarn and its possible components in order to get test specimen units.

For example, in the case of:

— a single yarn, the test specimen unit is directly obtained;

— a yarn made of two twisted single yarns, untwist the 2-ply yarn section in order to separate the two single yarn sections. And then, two test specimen units are obtained from one initial section (four test specimen units in total);

— a yarn made of two twisted 2-ply yarns, untwist firstly the yarn section in order to separate the two 2-ply yarn sections, then untwist each 2-ply yarn section in order to separate the 2-plies. And then four test specimen units are obtained from one initial section (eight test specimen units in total).

7.1.5 Fabrics

7.1.5.1 Woven fabrics

Unravel warp and weft yarns in order to get couple(s) of representative yarns from two different places of each direction.

For the structure of the woven fabric, destructure the woven fabric by unravelling warp and weft yarns, and then continue the preparation of each yarn sections as described in 7.1.4 in order to get test specimen units.

Cut out sections of the selected yarns from the laboratory woven fabric sample, so that the length section is greater than the length of the test specimen holder (such as slide, SEM stub or tube).

For example, in the case of:

— woven fabric made of single yarn in warp and another single yarn in weft, two single yarns shall be selected in the warp direction (one couple) and two single yarns shall be selected in the weft direction (one couple). And then, four test specimen units are prepared in total;

— woven fabric made of a 2-ply yarn in warp and another 2-ply yarn in weft, two 2-ply yarns shall be selected in the warp direction (one couple of 2-ply yarn) and two 2-ply yarns shall be selected in the

weft direction (one couple of 2-ply yarn). Each 2-ply yarn is prepared as described in 7.2. And then, eight test specimen units are prepared in total.

7.1.5.2 Knitted fabrics

De-knit yarns in order to get couple(s) of representative yarns from two different places.

For the structure of the knitted fabric, destructure the knitted fabric by de-knitting, and then continue the preparation of each yarn sections as described in 7.1.4 in order to get test specimen units.

Cut out sections of the selected yarns from the laboratory knitted fabric sample, so that the length section is greater than the length of the test specimen holder (such as slide, SEM stub or tube).

7.2 Preparation of a test specimen slide (LM) or stub (SEM)

7.2.1 Preparation for longitudinal view for LM

Prepare the test specimen(s) units as specified below. The selection of the test specimens is described in 7.1.

For each separate place, drop appropriate amount of neutral liquid medium (6.1). Cut snippets from the fibre bundle or the test specimen unit and place them on the two separate places of the slide. Disperse the fibre snippets uniformly by stirring with the dissecting needle. Carefully, lower a glass cover of the correct size over the fibre/ neutral liquid medium mixture and avoid air bubbles.

If the thickness of the preparation prevents the diameter measurements, discard the slide and prepare another one.

Prepare at least two slides.

If it is required to get more accurate results, more than one thousand fibres need to be counted. Considering that a yarn can have 100 to 120 fibres in a section, it could lead to prepare at least 10 slides.

7.2.2 Preparation for longitudinal view for SEM

Prepare the test specimen(s) units as specified below. The selection of the test specimens is described in 7.1.

Cut snippets from the fibre bundle or the test specimen unit and place them on the SEM stub. Prepare at least two stubs.

7.2.3 Preparation for cross view for LM or SEM

Prepare the test specimen(s) units as specified below. The selection of the test specimen or test specimen unit is described in 7.1.

Double the fibre bundle several times or fold the test specimen unit several times in order to fill the test tube, before filling it with the resin (6.2).

NOTE In order to reduce the time of the SEM stub preparation, different test specimen units can be placed on the same stub provided that they remain distinguishable.

Using the relevant procedure for the microtome type (5.3.3), prepare at least two cross sections from the fibre bundle embedded in the tube.

8 Procedures

8.1 General

Identification of the fibre type can be carried out based on ISO/TR 11827.

The total of all the fibres measured for the test specimen shall be at least 600.

8.2 LM procedure

8.2.1 Longitudinal view

Place the slide on the microscope stage, cover glass towards the objective. After the fibres have settled, the slide is examined in different fields. Begin the examination by moving the slide until a corner of the cover slip is focused. Then traverse the slide 1,0 mm (to B) then along a targeted fibre in the transverse direction, thus bringing the first area into view on the screen.

Traverse the slide in 1,0 mm steps, using the sliding mechanism described in 5.1, and analyse other fibres in each field as before. Continue traversing until the edge of the cover glass C is reached. Cross-traverse the slide 1,0 mm distance and continue with a second traverse and then a third, etc. following the A B C D E F G, etc. pattern (see Figure 1) until the observations have been done.

Measure the diameter of each targeted fibre after its observation and count the number of fibres. Record these results.

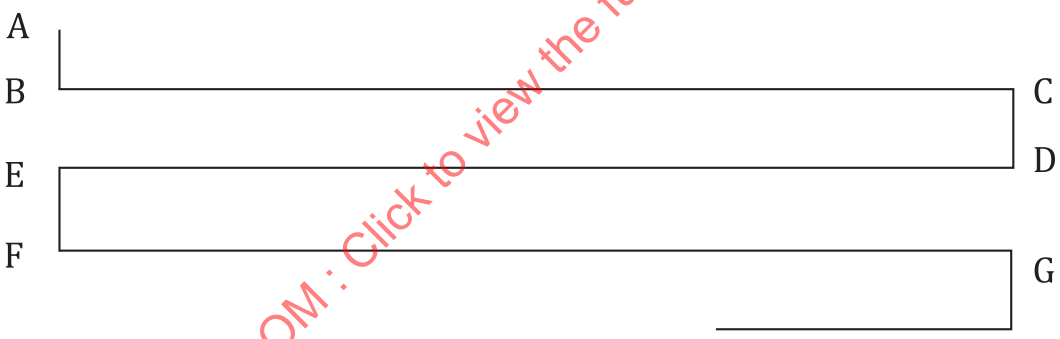

Figure 1 — Examination of the test specimen

8.2.2 Cross view

Place the slide on the microscope stage.

Focus to examine in different fields based on low magnification in order to target some fibres, and then set up higher magnification to get details of the targeted fibres.

Repeat again the same operation on several spots until the observations have been done.

Measure the area of each targeted fibre after its observation and count the number of fibres. Record these results.

8.3 SEM procedure

8.3.1 Longitudinal view

Place the stub inside the SEM. Focus to examine in different fields. Begin the examination by moving the stub to A. Then traverse the stub (to B) then along a targeted fibre in the transverse direction, thus bringing the first area into view on the screen.

Traverse the stub in several steps and analyse other fibres in each field as before, with a step width adapted to the stub. Continue traversing until C is reached. Cross-traverse the stub and continue with a second traverse and then a third, etc. following the A B C D E F G etc. pattern (see Figure 1) until the number of measurements (width, as a fibre diameter estimation D_i) has been reached.

Measure the diameter of each targeted fibre after its observation and count the number of fibres. Record these results.

8.3.2 Cross view

Place the stub inside the SEM. Focus to examine in different fields based on low magnification in order to target some fibres, and then set up higher magnification to get details of the targeted fibres.

Repeat again the same operation on several spots until the number of measurements (fibres area A_i) has been reached.

Measure the area of each targeted fibre after its observation and count the number of fibres. Record these results.

9 Calculation and expression of the results

9.1 Calculation based on fibre diameter measurements (Longitudinal view)

Calculate the content of each fibre component *i* as percent by mass using Formula (1).

$$P_i = \frac{N_i D_i^2 \rho_i}{\sum\left[N_i D_i^2 \rho_i \right]} \times 100 \quad (1)$$

where

P_i is the percentage by mass of component *i*, %;

N_i is the number of fibres counted for component *i*;

D_i is the mean fibre diameter of component *i*, in micron (µm);

ρ_i is the density of component i, in gram per cubic centimetre (g/cm^3), from Annex A, Table A.1.

Take the mean value of calculations of the two tests as the test result. If the difference between two tests is larger than 3 %, the third specimen shall be tested, in such case, the mean value of the three tests is taken as the test result.

Test result of fibre content is rounded to one decimal.

9.2 Calculation based on fibre area measurements (Cross view)

Calculate the content of each fibre component *i* as percent by mass using the Formula (2).

$$P_i = \frac{N_i A_i \rho_i}{\sum [N_i A_i \rho_i]} \times 100 \quad (2)$$

where

P_i is the percentage by mass of component *i*, %;

N_i is the number of fibres counted for component *i*;

A_i is the mean fibre area of component *i*, in square-micron (μm^2);

ρ_i is the density of component *i*, in gram per cubic centimetre (g/cm^3), from Annex A, Table A.1.

Take the mean value of calculations of the two tests as the test result; if the difference between two tests is larger than 3 %, the third specimen shall be tested, in such case, the mean value of the three tests is taken as the test result.

Test result of fibre content is rounded to one decimal.

9.3 Calculating the percentage by mass of fibre component in woven fabric sample

Calculate the content of each fibre component in woven fabric sample as percent by mass using Formula (3).

$$P_i = \frac{P_{iT} \times W_T + P_{iW} \times W_W}{W_T + W_W} \times 100 \quad (3)$$

where

P_i is the percentage by mass of some component in woven fabric sample, %;

P_{iT} is the percentage by mass of some component in warp yarns of woven fabric sample, %;

W_T is the mass of warp yarns in woven fabric sample;

P_{iW} is the percentage by mass of some component in weft yarns of woven fabric sample, %;

W_W is the mass of weft yarns in woven fabric sample.

10 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 20705:2019;

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

c) the identification of the sample (e.g. lot number, article number);

d) the method of sampling;

e) the number of test specimens;

f) the type of apparatus: LM or SEM;

g) the species of identified fibres;

h) the total number of measurements;

i) for each fibre species, the mean value of fibre diameter (longitudinal view) or of fibre area (cross view), the number of counted fibres and the applied fibre density;

j) the percentage of each fibre component;

k) any deviation from the given procedure.

Annex A
(normative)

Fibre density (Conventional)

Table A.1 lists conventional fibre density, in g/cm^3, to be used for the calculation of the mass percentage of the components, see Formulae (1) and (2).

NOTE This list derivates from ISO/TR 11827:2012, Table G.1.

Table A.1 — Density of the fibres

Fibre		Density (g/cm^3)
Elastodiene		0,95
Hemp		1,51
Cotton		1,51
Jute		1,51
Wool		1,34
Linen (Flax)		1,51
Ramie		1,51
Silk		1,32
Acetate		1,33
Viscose / Modal / Lyocell		1,51
Acrylic		1,18
Modacrylic		1,30
Chlorofibre		1,40
Elastane		1,20
Polyamide	“6”:	1,12
	“6,6”:	1,14
	“11”:	1,04
Polyester		1,38
Polypropylene		0,91
Polyethylene		0,95
Glass		2,60
Aramid	“para”:	1,45
	“meta”:	1,38

Annex B
(informative)

Statistical data

B.1 Based on fibre diameter measurements (Longitudinal view)

A knitted fabric sample made of wool and lyocell has been tested in 2016. Five laboratories have been involved. All carried out the testing using light microscopy (LM). Four sets of results have been taken into consideration for the statistical data (as the data set of one laboratory has been discarded for a problem during the measurements).

For the calculation of the percentage by mass, the following fibre density has been used for each fibre: 1,34 g/cm^3 for wool and 1,51 g/cm^3 for lyocell. The sets of initial data are presented in Table B.1.

The statistical data are based on ISO 13528:2015, Annex C, using robust algorithm A (mean, standard deviation and uncertainty type u_x) and robust algorithm S (repeatability and reproducibility). The statistical data, calculated only on the mass percentage of wool, are presented in Table B.2.

Table B.1 — Sets of initial data

		Lab. A		Lab. B		Lab. C		Lab. D	
		Test specimen unit		Test specimen unit		Test specimen unit		Test specimen unit	
		# 1	# 2	# 1	# 2	# 1	# 2	# 1	# 2
Wool	Number of counted fibres	670	719	268	nd	2 316	1 508	148	168
	Mean fibre diameter (µm)	19,0	18,6	19,4	nd	19,0	18,7	20,3	20,2
	Mass percentage (%)	50,1	49,5	48,0	nd	49,7	49,2	50,9	53,6
Lyocell	Number of counted fibres	799	872	332	nd	2851	1849	185	184
	Mean fibre diameter (µm)	16,4	16,0	17,1	nd	16,2	16,2	16,8	16,9
	Mass percentage (%)	49,9	50,5	52,0	nd	50,3	50,8	49,1	46,4

Table B.2 — Statistical data according to ISO 13528:2015, Annex C

Using robust algorithm A		Using robust algorithm S	
Mean/Mass percentage (%)	49,9	Repeatability, std dev [limit]	1,3 [3,5]
Standard deviation	2,0	Reproducibility, std dev [limit]	2,2 [6,0]
Uncertainty type u_x	1,25		

B.2 Based on fibre area measurements (Cross view)

B.2.1 Consideration on the measurement of the fibre area

Pictures of fibre cross view (based on SEM) were provided in 2018 to 7 laboratory participants. The laboratory participants were requested to use the scale provided with the pictures and to express the areas in µm^2. It was pointed out that one laboratory used the "paper method" to measure areas when the others used various software.

For each fibre area: the 7 measurements were used to calculate the ("robust") mean, the ("robust") standard deviation, the uncertainty of the measurement u_x (according to the algorithm A, ISO 13528:2015, C.3.1); and then, the differences were calculated.

An extract of measurements on glass fibres (areas in µm^2) is shown in Figure B.1 and Table B.3.

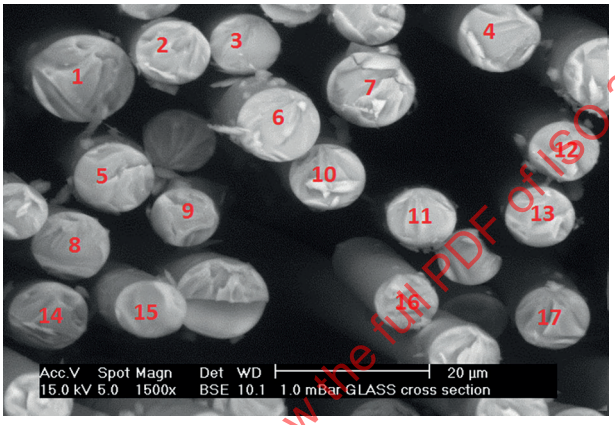

Figure B.1 — Cross view of glass fibres

Table B.3 — Results related to Figure B.1

Fibre n°	Lab A	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	$x^* =$	$s^* =$	$u_X =$
1	137,9	115,8	123,1	121,2	118,2	131	119,8	**124**	8	4
2	74,7	63,1	65,6	65,3	62,5	69	63,5	**66**	4	2
3	67,2	58,9	60,7	59,4	55,5	62	56,1	**60**	4	2
4	86,6	77,8	80,2	78,7	74,1	83	76,1	**80**	5	2
5	86,6	77,9	75,8	76,9	73,2	80	74,5	**77**	4	2
6	99,4	85,2	81,9	85,8	82,6	90	82,2	**86**	5	2
7	103,9	90	93,4	90,2	87,1	97	89	**93**	6	3
8	86,6	69,8	73,3	73,4	69,7	77	68,8	**73**	5	2
9	67,2	54,3	56	56,6	53,2	57	53,6	**56**	3	1
10	78,5	68,5	67,3	68,9	65,6	73	68,6	**70**	4	2
1	70,9	55,4	58	58,2	55,3	61	56,2	**58**	3	2
12	67,2	57,4	57,1	58,5	55,6	61	56,1	**58**	3	1
13	67,2	55	58,7	58,1	56,1	61	58,3	**59**	3	2
14	86,6	71,1	78,4	74,2	73	78	66,8	**75**	7	3
15	63,6	56,3	61,3	56,9	55,1	61	56,2	**59**	4	2
16	56,7	48,2	51,2	49,6	47,2	51	48	**50**	3	1
17	78,5	63,7	68,7	65,6	63	69	64,2	**67**	4	2

An extract of measurements on polypropylene fibres is shown in Figure B.2 and Table B.4.

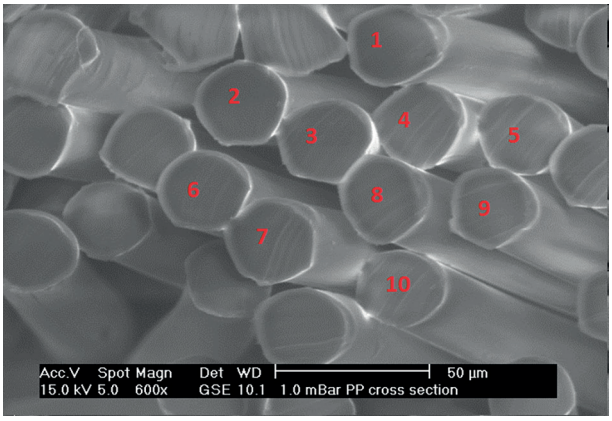

Figure B.2 — Cross view of polypropylene fibres

Table B.4 — Results related to Figure B.2

Fibre n°	Lab A	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	$x^* =$	$s^* =$	$u_X =$
1	713,7	576,6	699,7	609,4	606	744	612,1	**652**	74	35
2	756,9	594,9	673,2	651,2	646,5	714	639,1	**668**	60	28
3	775,1	636,8	682	644,7	639,5	716	659,3	**675**	48	23
4	704,8	573,5	645,9	619,2	607,7	683	591,2	**632**	55	26
5	672,3	573,1	634,8	637,3	591,7	698	597,9	**629**	51	24
6	725	601,2	656,2	627,2	608,1	694	598,3	**644**	56	27
7	738,2	634	681,3	666,8	623,8	701	616,4	**666**	51	24
8	771,8	586,9	673,9	643,7	588,2	703	569,7	**648**	83	39
9	697,7	594,8	639,2	587,8	561,6	635	534,3	**607**	62	29
10	677,1	569,4	617,1	591,7	560,4	640	522,6	**597**	59	28

An extract of measurements on polyester fibres is shown in Figure B.3 and Table B.5.

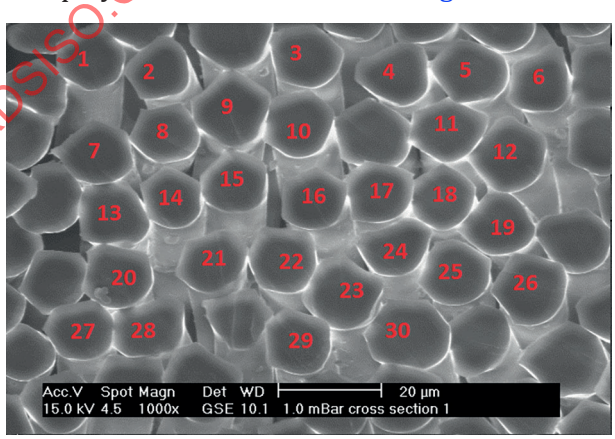

Figure B.3 — Cross view of polyester fibres

Table B.5 — Results related to Figure B.3

Fibre n°	Lab A	Lab B	Lab C	Lab D	Lab E	Lab F	Lab G	$x^* =$	$s^* =$	$u_X =$
1	151	122,1	134	126,7	124,1	138	126,5	**131**	10	5
2	134,8	108,3	170,7	114,7	112,2	125	114,6	**122**	15	7
3	162,3	133,8	147	138,6	130,5	150	135	**142**	12	6
4	157,1	130,3	145,7	132	126,7	141	127,3	**137**	12	6
5	167,6	131,5	151,8	137,1	134,2	150	136,7	**144**	13	6
6	141,7	118,9	131,8	122,2	117,1	135	118,2	**126**	11	5
7	158,8	128,9	147	136,4	131	150	138,8	**142**	12	6
8	141,4	112,6	127,8	120,7	116,4	127	115,8	**122**	10	5
9	185	153,8	173,1	159,7	156,4	178	159,5	**167**	14	6
10	167,9	135,3	142,7	139,9	134	149	133,7	**141**	9	4
11	166,1	128,4	142,2	139,9	134,6	149	136,7	**141**	11	5
12	184,9	148,2	169,1	160,5	151,5	172	153,7	**163**	15	7
13	166,3	128,6	142,7	136,3	133,5	151	133,6	**141**	13	6
14	132,6	103,2	117,4	107,7	105,3	119	110,4	**113**	10	5
15	147,6	114,4	133,4	125,7	118,2	138	126,7	**129**	13	6
16	148,2	119,4	137,9	123,3	116,3	133	121	**128**	13	6
17	153,4	127,8	129,7	127,9	118,8	138	124,5	**130**	10	5
18	140,6	116,3	124,9	116,7	115,3	129	119	**122**	9	4
19	149,3	120,6	139,5	128,9	126,9	143	126	**133**	12	6
20	160,3	125,1	144,6	138	129,5	148	134,7	**140**	14	6
21	147,8	120,1	147,3	123,5	120,9	138	123,5	**132**	14	7
22	167,7	128,6	148,6	142,7	134	152	140,7	**145**	14	6
23	191,3	152,3	160,1	157,4	154,3	174	160,7	**163**	12	6
24	137,9	117,9	124,6	116,9	116,1	130	119,6	**123**	8	4
25	160,2	132,7	141,7	139,4	131,8	149	136,5	**141**	10	5
26	184,9	149	169,4	155,7	149,9	168	156	**161**	14	6
27	148	127,3	129,4	125,7	118,9	135	123,4	**129**	9	4
28	158	140,1	138,2	138,9	130	147	129,1	**140**	11	5
29	165,6	130,4	141,4	136,1	128,5	145	135,6	**139**	10	5
30	212,5	171,9	187,2	177,3	172,9	195	184,2	**185**	14	7