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Textiles — Qualitative and quantitative analysis of some cellulose fibres (lyocell, cupro) and their blends —

Part 2: Blend quantification using light microscopy method

Textiles — Analyses qualitative et quantitative de certaines fibres cellulosiques (lyocell, cupro) et leurs mélanges —

Partie 2: Quantification du mélange par une méthode de microscopie optique

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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Contents

Page

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.*

A list of all parts in the ISO 21915 series can be found on the ISO website.

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

Introduction

The qualitative and quantitative determination of fibres is important for the distribution of textile products. In many countries, it is legally obligatory for producers to attach information on the type of fibres used and their mixing ratio to textile products.

Therefore, it is desirable that qualitative methods of all fibres used in textile products and quantitative methods in the case where fibres are mixed (all combinations that can be assumed) exist as test standards.

Cupro and lyocell described in this document are regenerated fibres made from plants and can be said to be materials that contribute to a sustainable society in that raw materials are not derived from petroleum.

However, cupro and lyocell are difficult to qualify. Because the characteristics of appearance, chemical resistance, infrared spectroscopy (IR) spectrum, etc. are almost the same, the qualitative property according to ISO/TR 11827 and the quantification by the ISO 1833 series cannot be performed in some cases. That is, even if we know that unknown fibre is a cupro or lyocell, we cannot identify which one is.

Therefore, it is difficult to distinguish cupro or lyocell if the cupro or lyocell exists in the textile product or the possibility that cupro and lyocell are mixed completely cannot be denied.

ISO 21915 is composed of three parts. ISO 21915-1 specifies the identification method of cupro and lyocell by scanning electron microscope and infrared spectrum analysis. Those may be the time-consuming methods to use the composition analysis. This document and ISO 21915-3 specify methods for the composition analysis. The method to be used is determined by the instrument availability and experience.

Textiles — Qualitative and quantitative analysis of some cellulose fibres (lyocell, cupro) and their blends —

Part 2:
Blend quantification using light microscopy method

1 Scope

This document specifies the quantitative analysis of cupro and lyocell mixtures using the microscopical analysis as described in ISO 20705 after re-dyeing cupro and lyocell mixtures.

This testing method is applied only for cupro and lyocell, or those blends. If other fibres are present, those are identified using the test method of ISO/TR 11827 and removed using the relevant parts of the ISO 1833 series.

This method is not applicable for the fibre surface is damaged during the process (e.g. chemically or physically).

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 (all parts), *Textiles — Quantitative chemical analysis*

ISO 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 20705:2019, *Textiles — Quantitative microscopical analysis — General principles of testing*

ISO 21915-1, *Textiles — Qualitative and quantitative analysis of some cellulose fibres (lyocell, cupro) and their blends — Part 1: Fibre identification using scanning electron microscope and spectral analysis methods*

3 Terms and definitions

For the purposes of this document, the following terms and definitions 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
cupro
cellulose fibre obtained by the cuprammonium process

[SOURCE: ISO 2076:2013, 4.1]

3.2
lyocell
cellulose fibre obtained by an *organic solvent* (3.3) *spinning process* (3.4)

[SOURCE: ISO 2076:2013, 4.2]

3.3
organic solvent
mixture of organic chemicals and water

3.4
solvent spinning
dissolving and spinning without the formation of a derivative

3.5
re-dyeing
process of decolouring and dyeing again fibres

Note 1 to entry: Identification of cupro and lyocell is based on the difference of their dye affinity. Usually, an alkali treatment is applied after the decolouration.

Note 2 to entry: If the fibre mixtures have not been dyed, decolouration stage may be omitted.

4 Principle

Decolour the testing sample consisting of cupro and lyocell or either cupro or lyocell, then dye again the decoloured sample by using the specified stain solution and the condition. Observe the re-dyed sample by the microscopical analysis described in ISO 20705 on longitudinal views of fibre. Count the numbers of cupro or lyocell based on the colour shade difference.

5 Reagents

The reagents to be used shall be as follows.

5.1 Water, grade 3 quality as specified in ISO 3696.

5.2 Sodium hydrosulphite solution 5 %.

Boil 50 ml of water (5.1) and add 2,5 g of sodium hydrosulphite.

SAFETY PRECAUTIONS — The harmful effects of this regent shall be borne in mind, and full precautions shall be taking during use.

5.3 Sodium hydroxide solution of 1,8 %.

5.4 Hydrochloric acid solution of approximately 2 %, diluted by water (5.1).

5.5 Stain solution

— dyestuff (C.I. Direct Blue 71 or C.I. Direct Blue 78): 1 g;

— sodium sulfate: 1 g;

— make up to 100 ml by adding water (5.1).

Information of other dyestuff is shown in Annex A.

6 Apparatus

6.1 Water bath, capable of heating up to 40 °C ± 2 °C.

6.2 Thermometer, capable of measurement of 40 °C.

6.3 Specimen cutting tool.

6.4 Microtome, capable of cutting fibre from 0,4 mm to 0,8 mm in length and is composed of fibre holder, fibre fixing metal fittings and fibre pusher for setting of fibre to the specified length, or this microtome with similar functions.

6.5 Razor blade.

6.6 Light microscope and tools, as specified in ISO 20705:2019, 5.1 and Clause 6.

7 Procedure

7.1 Prior identification

Fibres present in the sample are identified according to ISO/TR 11827, and other fibres shall be removed using the relevant parts of ISO 1833 series as stated in ISO 21915-1.

7.2 Preparation of test specimen

Prepare the specimen with a mass of 1 g and cut to approximately 1 cm × 1 cm or unravel to yarn.

7.3 Decolouring

Decolour the specimen if the sample is dyed as follows.

7.3.1 Dip the specimen in water (5.1) to wet, then extract water by absorbent paper.

7.3.2 Soak the specimen into the boiled 5 % sodium hydrosulphite solution (5.2).

7.3.3 Stir the solution until the specimen is decolourised.

NOTE A stirring duration of about 1 min has been found suitable.

7.3.4 Wash the specimen by water (5.1) and then extract water by absorbent paper.

7.4 Pre-treatment for re-dyeing

The pre-treatment shall be performed to improve the dyeability as the following procedure.

7.4.1 Dip the specimen in water (5.1) to wet, then extract water by absorbent paper.

7.4.2 Add the specimen into 50 ml of 1,8 % sodium hydroxide solution (5.3) and stir for 15 min.

7.4.3 Take out the specimen from 7.4.2 and transfer the specimen to another glass beaker with about 25 ml of approximately 2 % dilute hydrochloric acid solution (5.4), then,

7.4.4 Wash the specimen, then extract water by absorbent paper and dry up naturally.

7.5 Re-dyeing

7.5.1 Prepare the specimen a mass of 0,5 g from the pre-treated specimen.

7.5.2 Dip the specimen in water (5.1) to wet, then extract water by absorbent paper.

7.5.3 Prepare a beaker and pour 15 ml of the stain solution (5.5) into the beaker for dyeing.

7.5.4 Prepare another beaker and pour 15 ml of water into the beaker to measure the liquid temperature.

7.5.5 Place the beakers of 7.5.3 and 7.5.4 into the water bath (6.1) and dip a bar thermometer (6.2) in the beaker with water 7.5.4.

7.5.6 Adjust the temperature of water in the beaker to 40 °C ± 2 °C by the water bath.

7.5.7 Place the specimen into the beaker with the stain solution and dye the specimen for 5 min.

7.5.8 After dyeing, remove the specimen and place into another beaker with 1 l of water (5.1) with (25 °C ± 3 °C), and wash the specimen for 1 min, and repeat this process total 5 times. Then extract water by absorbent paper and dry up naturally.

The effect of the re-dyeing temperature is shown in Annex B.

7.6 Measurement of fibres

7.6.1 Preparation for counting

7.6.1.1 Prepare the re-dyed specimens for longitudinal view on light microscope (6.6) as described in ISO 20705:2019, Clause 8.

7.6.1.2 Cut the specimen a length of 0,6 mm ± 0,2 mm.

7.6.2 Measurement

7.6.2.1 Count the number of cupro fibre or lyocell fibre by the differentiation of the colour shade according to ISO 20705:2019, 9.2.1, up to a total of at least 1 000 fibres.

7.6.2.2 Measure the fibre diameters for all identified fibres at least 100 for each.

7.6.2.3 Calculate the mass percentage of the fibres according to ISO 20705:2019, 10.1, with the conventional fibre density of 1,51 g/cm^3 for both cupro and lyocell, as listed in ISO 20705.

In case the distinction of the colour shade difference between lyocell and cupro is inconclusive, 100 % lyocell and 100 % cupro are dyed separately by the same condition of the specimens and observed separately as the reference.

NOTE The samples of 100 % lyocell and 100 % cupro are obtained from test applicants or ISO/TC 38 secretariat.

8 Accuracy

The interlaboratory test results are shown in Annex C. A total of 8 laboratories participated in this trial and all data are shown in the Annex C. The coefficient determination was R^2 = 0,999 4 as shown in Figure C.8.

9 Test report

The test report shall include the following information:

a) a reference to this document, i.e. ISO 21915-2:2020;

b) all details necessary to identify the product analysed (such as manufacturer, product type, batch or date of manufacture, as required);

c) the result obtained, expressed as the composition of cupro and lyocell nearest 0,1;

d) any deviation from the given procedure;

e) any unusual features observed;

f) the date of the test.

Annex A
(informative)

Observation of fibres by the re-dyeing method

A.1 General

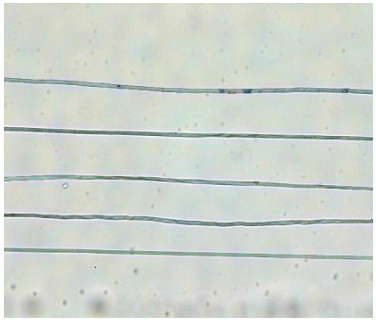
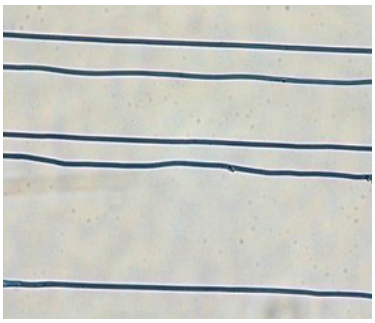
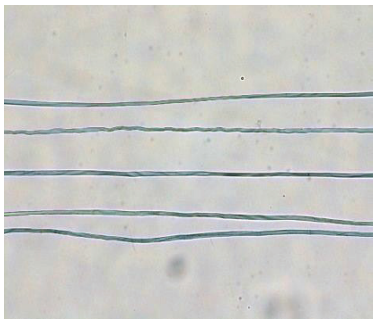
When the cupro and lyocell were re-dyed by specific dyestuffs, the appearance of the colour shade is observed and explained in this annex.

A.2 Observation of colour difference

A.2.1 Longitudinal view

The colour shade obtained by the re-dyeing method is shown in Table A.1 for longitudinal view. Two dyestuffs are shown where the shade of cupro is darker than the shade of lyocell.

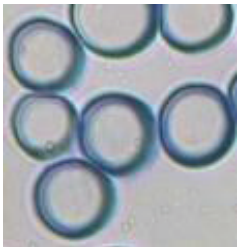
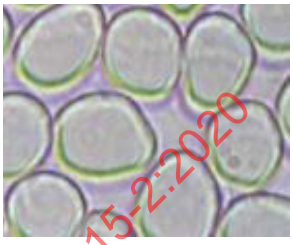
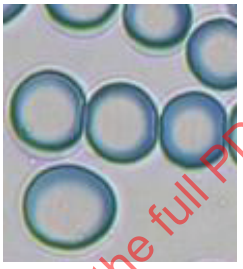
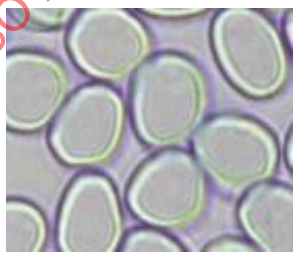
Table A.1 — Longitudinal view of cupro and lyocell

Light microscopy: Longitudinal view			
Fibres		Cupro	Lyocell
Dyestuff	**C.I. Direct Blue 71**		
	C.I. Direct Blue 78		

A.2.2 Cross-sectional view

The cross-sectional views of the fibres are shown in Table A.2. The skin layer of cupro fibre is dyed intensively and lyocell fibres are not dyed clearly. This effect causes the colour shade difference of the longitudinal view.

Table A.2 — Cross-sectional view of cupro and lyocell

Light microscopy: Cross view			
Fibres		**Cupro**	**Lyocell**
Dyestuff	**C.I. Direct Blue 71**	 Cupro	 Lyocell
	C.I. Direct Blue 78	 Cupro	 Lyocell

A.3 Example of application of this testing method

A.3.1 Sample

One fabric sample with colour designated as the composition of cupro and lyocell was purchased in market.

A.3.2 Test procedure

The sample is cut to 10 specimens as described in (see 7.2) and decoloured as described in (see 7.3). The 5 specimens are re-dyed (see 7.4) by using dyestuffs C.I. Direct Blue 71 and another 5 specimens are dyed by using C.I. Direct Blue 78.

A.3.3 Test results

The test results are shown in Table A.3. Both dyestuffs give the equivalent results without significant difference in accuracy for two dyestuffs.

NOTE As fibre diameters for both lyocell and cupro were similar, the mass percentages were calculated based on the fibre counts only.

Table A.3 — Test results for five specimens

		Composition testing result (%)							
Dyestuff		**C.I. Direct Blue 71**				**C.I. Direct Blue 78**			
Fibre specimen		**Cupro**		**Lyocell**		**Cupro**		**Lyocell**	
1	*n* = 2	29,6	28,2	70,4	71,8	27,9	30,8	72,1	69,2
	Ave	28,9		71,1		29,4		70,6	
2	*n* = 2	27,3	30,1	72,7	69,9	33,5	31,1	66,5	68,9
	Ave	28,7		71,3		32,3		67,7	
3	*n* = 2	27,9	29,9	72,1	70,1	31,3	32,9	68,7	67,1
	Ave	28,9		71,1		32,1		67,9	
4	*n* = 2	26,3	29,1	73,7	70,9	29,4	32,2	70,6	67,8
	Ave	27,7		72,3		30,8		69,2	
5	*n* = 2	28,8	26,6	71,2	73,4	29,3	31,3	70,7	68,7
	Ave	27,7		72,3		30,3		69,7	
Ave		28,4		71,6		31,0		69,0	
Standard deviation		0,6				1,2			
Measurement uncertainty		1,9				1,6			

Annex B
(informative)

Determination of re-dyeing temperature

B.1 General

As the major factor of re-dyeing condition to affect to colour shade, the re-dyeing temperature was tested and observed relating to the colour shade.

B.2 Dyestuff

The following dyestuffs were used.

— C.I. Direct Blue 71

— C.I. Direct Blue 78

B.3 Fibre sample

Cupro 100 % fibre and lyocell 100 % fibre were prepared.

B.4 Re-dyeing temperature used for test

35 °C, 38 °C, 40 °C, 42 °C and 45 °C were used for the test.

B.5 Observation of shade difference

The test results are shown in Table B.1. The temperature of dyeing was determined as 40 °C ± 2 °C from this result.

Table B.1 — Observation of shade difference of dyed fibres

Dyeing temperature	C.I. Direct Blue 71		C.I. Direct Blue 78	
	Cupro	Lyocell	Cupro	Lyocell
35 °C	Poor	Poor	Poor	Poor
38 °C	Good	Good	Good	Good
40 °C	Best	Best	Best	Best
42 °C	Good	Good	Good	Good
45 °C	Poor	Poor	Poor	Poor

Annex C
(informative)

Interlaboratory test result

C.1 General

The interlaboratory test was performed from January to February of 2018 with 8 laboratories and the result is shown below.

C.2 Test sample

The test samples were prepared and sent by the organizer to the laboratories. The details of sample are shown in Table C.1.

Table C.1 — Test samples

Sample	Declared fibre composition	Number of laboratories tested
A (yarn)	cupro (Cu) 80 %/lyocell (Ly) 20 %	4
B (yarn)	cupro 40 %/lyocell 60 %	8
C (yarn)	lyocell 100 %	7
D (yarn)	cupro 60 %/lyocell 40 %	8
E (yarn)	cupro 100 %	8
F (yarn)	cupro 20 %/lyocell 80 %	4
G (fabric)	cupro 30 %/lyocell 70 %	6

C.3 Dyestuffs used

The colour index of dyestuff used in respective laboratories is shown in Table C.2.

Table C.2 — Colour index used

Lab	Colour index of dyestuff used
Lab 1	C.I. Direct Blue 78
Lab 2	C.I. Direct Blue 71
Lab 3	C.I. Direct Blue 78
Lab 4–1	C.I. Direct Blue 71
Lab 4–2	C.I. Direct Blue 78
Lab 5	C.I. Direct Blue 71
Lab 6	C.I. Direct Blue 78
Lab 7	C.I. Direct Blue 78
Lab 8	—

C.4 Test results

C.4.1 Test result for Sample A

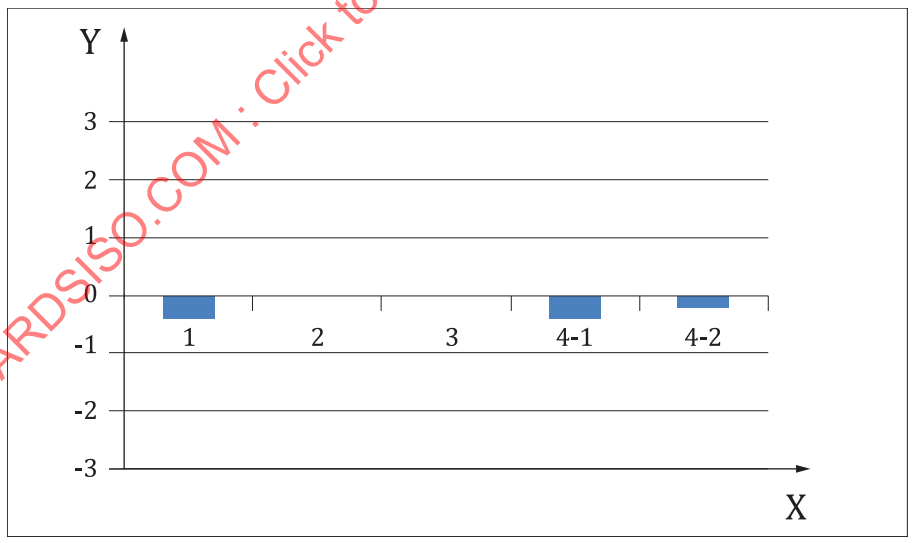
Test results are shown in Table C.3, in Table C.4, in Figure C.1 and in Table C.5.

Table C.3 — Test results for Sample A

Sample		Declared fibre composition	Number	Lab 1	Lab 2	Lab 3	Lab 4–1	Lab 4–2	Lab 5	Lab 6	Lab 7	Lab 8	Average
A	Cupro	80	1	78,3	80,1	81,4	79,4	78,6	—	—	—	—	79,6
			2	79,5	80,0	78,9	78,1	80,3	—	—	—	—	79,4
			Average	78,9	80,0	80,1	78,7	79,4	—	—	—	—	79,5
	Lyocell	20	1	21,7	19,9	18,6	20,6	21,4	—	—	—	—	20,4
			2	20,5	20,0	21,1	21,9	19,7	—	—	—	—	20,6
			Average	21,1	20,0	19,9	21,3	20,6	—	—	—	—	20,5
Z'-score				−0,4	0,0	0,0	−0,4	−0,2	—	—	—	—	—

Table C.4 — Repeatability and reproducibility for Sample A

		Cu	Ly
Mean value		79,5	20,5
Repeatability	S_r	1,11	
	CV_r	1,39 %	5,40 %
Reproducibility	S_R	1,01	
	CV_R	1,27 %	4,93 %



Key

X laboratory number

Y Z'-score

Figure C.1 — Z'-score for Sample A

Table C.5 — Summary of Z'-score

	Number of laboratories
$\|Z'\| \leq 2$	5
$2 < \|Z'\| \leq 3$	0
$3 \leq \|Z'\|$	0

C.4.2 Test result for Sample B

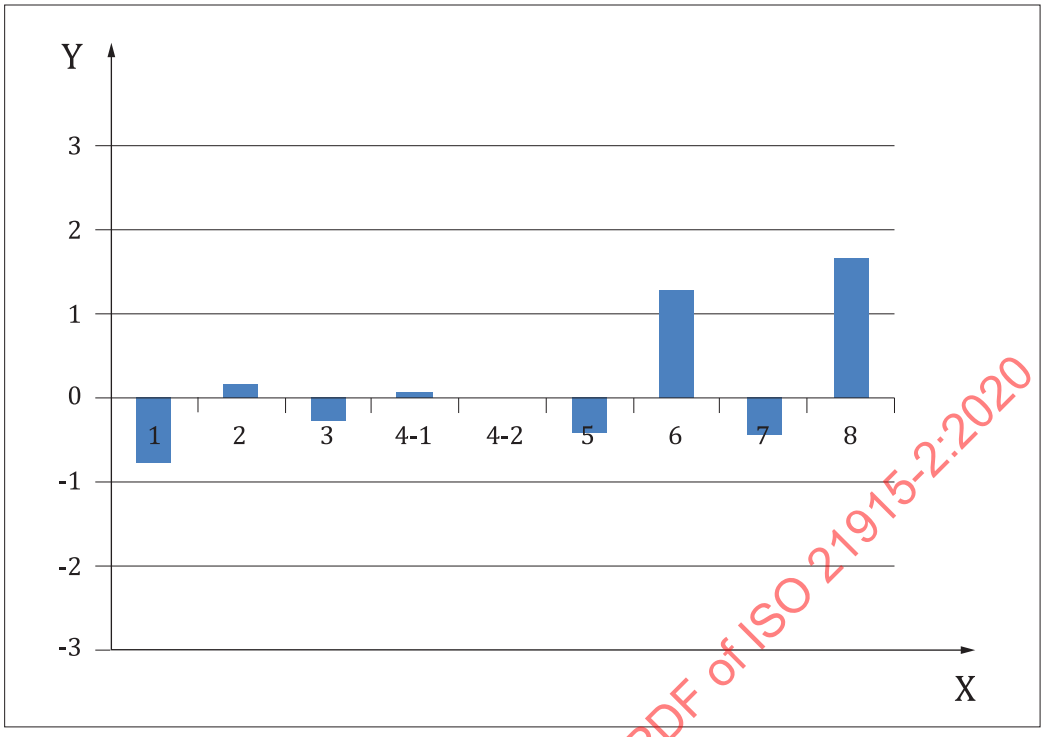
Test results are shown in Table C.6, in Table C.7, in Figure C.2 and in Table C.8.

Table C.6 — Interlaboratory test results of Sample B

Sample		Declared fibre composition	Number	Lab 1	Lab 2	Lab 3	Lab 4–1	Lab 4–2	Lab 5	Lab 6	Lab 7	Lab 8	Average
B	Cupro	40	1	35,3	41,3	37,2	41,5	41,8	37,4	45,9	36,2	46,3	40,3
			2	37,1	40,2	40,2	39,2	38,5	38,6	46,9	39,5	50,3	41,2
			Average	36,2	40,7	38,7	40,3	40,1	38,0	46,4	37,9	48,3	40,7
	Lyocell	60	1	64,7	58,7	62,8	58,5	58,2	62,6	54,1	63,8	53,7	59,7
			2	62,9	59,8	59,8	60,8	61,5	61,4	53,1	60,5	49,7	58,8
			Average	63,8	59,3	61,3	59,7	59,9	62,0	53,6	62,1	51,7	59,3
Z'-score				−0,8	0,2	−0,3	0,1	0,0	−0,4	1,3	−0,4	1,7	—

Table C.7 — Repeatability and reproducibility for Sample B

		Cu	Ly
Mean value		40,7	59,3
Repeatability	S_r	1,81	
	CV_r	4,44 %	3,05 %
Reproducibility	S_R	4,23	
	CV_R	10,39 %	7,15 %



Key

X laboratory number

Y Z'-score

Figure C.2 — Z'-score for Sample B

Table C.8 — Summary of Z'-score

	Number of laboratories
$\|Z'\| \leq 2$	9
$2 < \|Z'\| \leq 3$	0
$3 \leq \|Z'\|$	0

C.4.3 Result for sample C

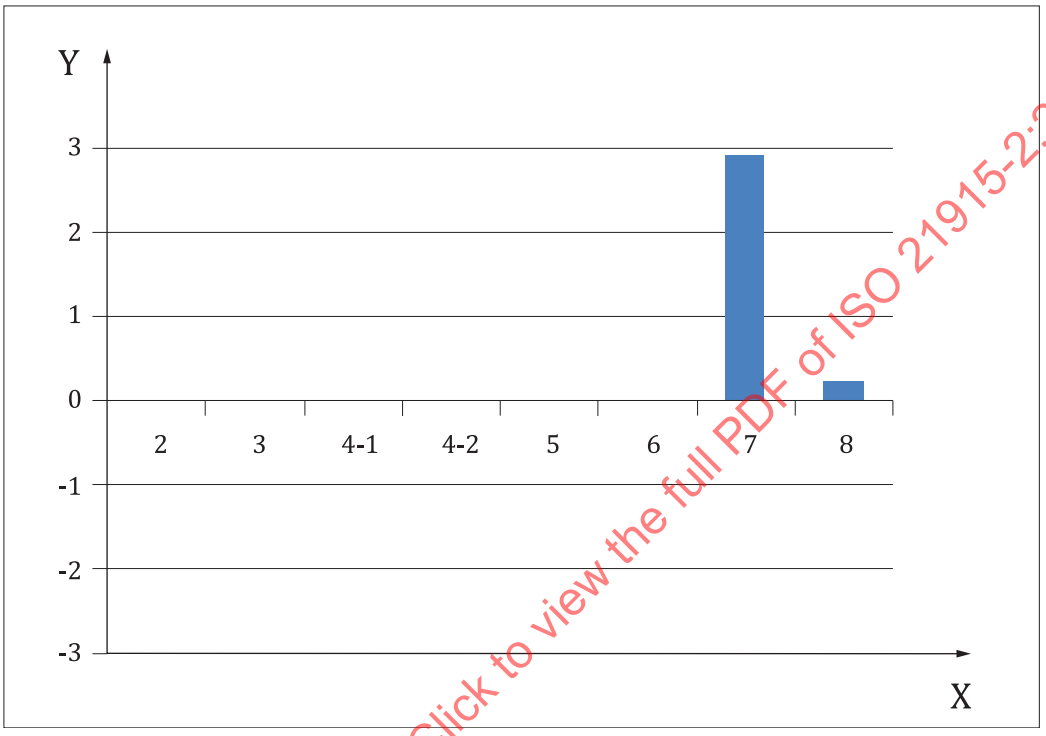
Test results are shown in Table C.9, in Table C.10, in Figure C.3 and in Table C.11.

Table C.9 — Interlaboratory test results of Sample C

Sample		Declared fibre composition	Number	Lab 1	Lab 2	Lab 3	Lab 4–1	Lab 4–2	Lab 5	Lab 6	Lab 7	Lab 8	Average
C	Cupro	0	1	—	0,0	0,0	0,0	0,0	0,0	0,0	10,5	0,0	1,3
			2	—	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,8	0,1
			Average	—	0,0	0,0	0,0	0,0	0,0	0,0	5,3	0,4	0,7
	Lyocell	100	1	—	100	100	100	100	100	100	89,5	100	98,7
			2	—	100	100	100	100	100	100	100	99,2	99,9
			Average	—	100	100	100	100	100	100	94,7	99,6	99,3
Z'-score				—	0,0	0,0	0,0	0,0	0,0	0,0	2,9	0,2	—

Table C.10 — Repeatability and reproducibility for Sample C

		Cu	Ly
Mean value		0,1	99,9
Repeatability	S_r	0,21	
	CV_r	—	0,21 %
Reproducibility	S_R	0,21	
	CV_R	—	0,21 %



Key

X laboratory number

Y Z'-score

Figure C.3 — Z'-score for sample C

Table C.11 — Summary of Z'-score

	Number of Laboratories
$\|Z'\| \leq 2$	7
$2 < \|Z'\| \leq 3$	1
$3 \leq \|Z'\|$	0

C.4.4 Result for sample D

Test results are shown in Table C.12, in Table C.13, in Figure C.4 and in Table C.14.

Table C.12 — Interlaboratory test results of Sample D

Sample		Declared fibre composition	Number	Lab 1	Lab 2	Lab 3	Lab 4–1	Lab 4–2	Lab 5	Lab 6	Lab 7	Lab 8	Average
D	Cupro	60	1	52,8	56,6	61,8	60,3	60,4	60,2	68,5	49,9	49,2	57,7
			2	52,5	55,1	57,6	57,9	61,3	57,1	71,1	51,3	60,9	58,3
			Average	52,6	55,8	59,7	59,1	60,8	58,9	69,8	50,6	55,0	58,0
	Lyocell	40	1	47,2	43,4	38,2	39,7	39,6	39,8	31,5	50,1	50,8	42,3
			2	47,5	44,9	42,4	42,1	38,7	42,9	28,9	48,7	39,1	41,7
			Average	47,4	44,2	40,3	40,9	39,2	41,1	30,2	49,4	45,0	42,0
Z'-score				–1,2	–0,7	0,0	–0,1	0,1	–0,2	1,6	–1,5	–0,8	—

Table C.13 — Repeatability and reproducibility for Sample D

		Cu	Ly
Mean value		58,0	42,0
Repeatability	S_r	3,18	
	CV_r	5,48 %	7,57 %
Reproducibility	S_R	6,01	
	CV_R	10,36 %	14,32 %

Key

X laboratory number

Y Z'-score

Figure C.4 — Z'-score for Sample D

Table C.14 — Summary of Z'-score

	Number of laboratories
$\|Z'\| \leq 2$	9
$2 < \|Z'\| \leq 3$	0
$3 \leq \|Z'\|$	0

C.4.5 Result for Sample E

Test results are shown in Table C.15, in Table C.16, in Figure C.5 and in Table C.17.

Table C.15 — Interlaboratory test results of Sample E

Sample		Declared fibre composition	Number	Lab 1	Lab 2	Lab 3	Lab 4–1	Lab 4–2	Lab 5	Lab 6	Lab 7	Lab 8	Average
E	Cupro	100	1	—	100	100	100	100	100	100	100	100	100
			2	—	100	100	100	100	100	100	100	99,3	99,9
			Average	—	100	100	100	100	100	100	100	99,7	100
	Lyocell	0	1	—	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,0
			2	—	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,7	0,1
			Average	—	0,0	0,0	0,0	0,0	0,0	0,0	0,0	0,3	0,0
Z'-score				—	0,0	0,0	0,0	0,0	0,0	0,0	0,0	−0,3	—

Table C.16 — Repeatability and reproducibility for Sample E

		Cu	Ly
Mean value		100,0	0,0
Repeatability	S_r	0,18	
	CV_r	0,18 %	—
Reproducibility	S_R	0,17	
	CV_R	0,18 %	—