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Textiles — Fibres — Determination of burning behaviour by oxygen index

Textiles — Fibres — Détermination du comportement au feu au moyen de l'indice d'oxygène

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Foreword

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This document was prepared by Technical Committee ISO/TC 38, *Textiles*, Subcommittee SC 23, *Fibres and yarn*.

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Textiles — Fibres — Determination of burning behaviour by oxygen index

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1 Scope

This document specifies a test method for the determination of burning behaviour of textile fibres by oxygen index.

This document is only used for the purpose of testing burning behaviour of textile fibres under conditions of this test, controlling quality of the products, or studying the factors causing the fire of some particular textile fibres. It is not used for assessing fire risk in their actual use.

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 139, *Textiles — Standard atmospheres for conditioning and testing*

ISO 2060, *Textiles — Yarn from packages — Determination of linear density (mass per unit length) by the skein method*

ISO 2061, *Textiles — Determination of twist in yarns — Direct counting method*

ISO 4589-1, *Plastics — Determination of burning behaviour by oxygen index — Part 1: General requirements*

ISO 4589-2:2017, *Plastics — Determination of burning behaviour by oxygen index — Part 2: Ambient-temperature test*

ISO 4880, *Burning behaviour of textiles and textile products — Vocabulary*

ISO 6741-3:1987, *Textiles — Fibres and yarns — Determination of commercial mass of consignments — Part 3: Specimen cleaning procedures*

3 Terms and definitions

For the purpose of this document, the terms and definitions given in ISO 4589-1 and ISO 4880 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/ui

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

4 Principle

A test specimen is fixed vertically in a transparent chimney with upward flowing oxygen mixture gas. The top end of the test specimen is ignited and its burning behaviour is observed. The burning length or burning duration is compared with the given criterion.

The minimum oxygen concentration is estimated by a series of experiments at different volume fractions of oxygen.

5 Apparatus

The usual laboratory apparatus and, in particular, the following shall be used.

5.1 Test chimney, as specified in ISO 4589-2:2017, 5.1.

5.2 Test specimen holder, to support a test specimen vertically, which is fixed on the axial central position of the test chimney.

The clamping position is at least 15 mm away from the nearest point where the test specimen can burn.

It is recommended that the frame of the test specimen holder is smooth to minimize induction of turbulence in the rising flow gas.

5.3 Gas supplies, shall comprise pressurized sources of oxygen and nitrogen or other gases mixed with oxygen.

When using gas flow meter to control the oxygen concentration (volume fraction), gas supplies shall comprise pressurized sources of oxygen and nitrogen, both with a purity not less than 99,99 %.

When using oxygen analyser to control the oxygen concentration (volume fraction), gas supplies shall comprise pressurized sources of oxygen and/or nitrogen not less than 98 % (mass fraction) pure and/or clean air [containing 20,9 % (volume fraction) oxygen], as appropriate.

5.4 Gas control devices, with a suitable gas flow meter or oxygen analyser measures the oxygen concentration (volume fraction) in the gas mixture with an accuracy of ±0,5 %.

When the gas flow rate within the test chimney is (40 ± 2) mm/s at temperature [10 °C, 30 °C], the precision of concentration adjustment is ±0,1 %.

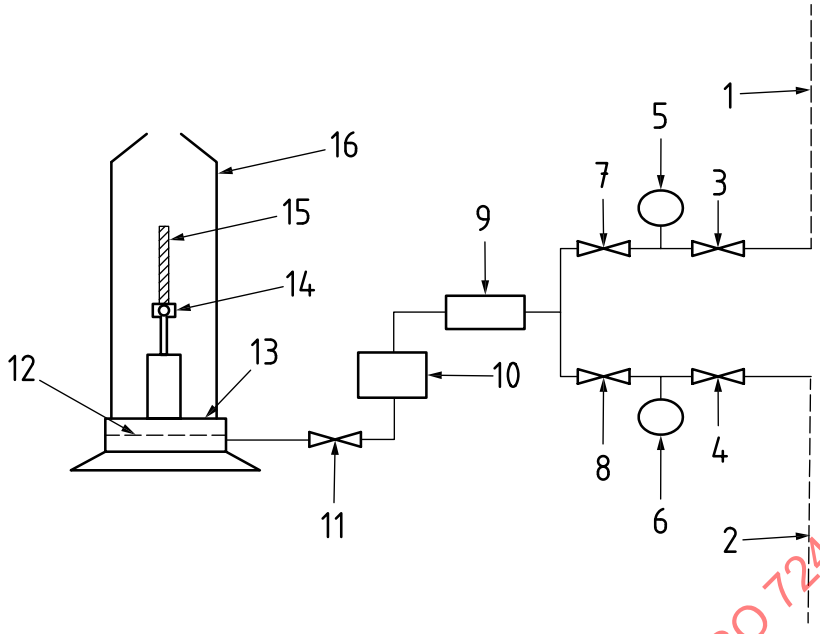
There are needle valves, calibrated orifices, gas pressure regulators, pressure gauges, flow meters on every gas supply line.

It is equipped with a calibrated flow meter to indicate the flow rate of gas through the test chimney is within the required limits.

Equipment shall be regularly calibrated in accordance with ISO 4589-2:2017, Annex A.

A typical test system for oxygen index is shown in Figure 1 or Figure 2.

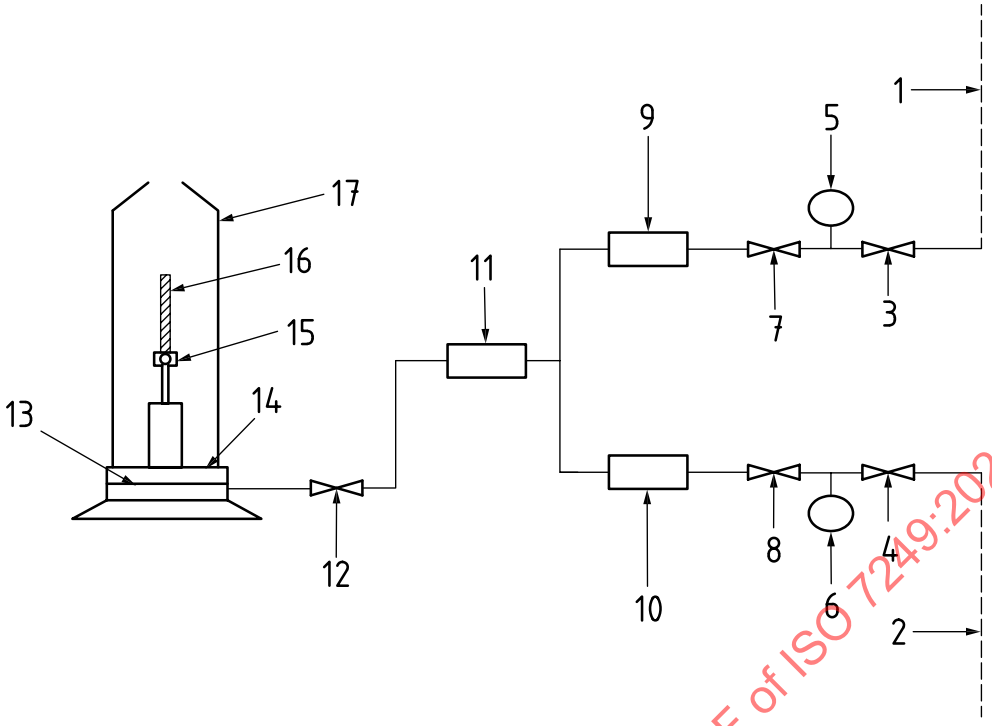
Other apparatus specified in ISO 4589-2:2017, 5.4, may be used for the test provided that equivalent results are obtained.



Key

1	nitrogen supply	9	calibrated mass flow meter
2	oxygen supply	10	oxygen analyser
3	gas pressure regulator of nitrogen	11	calibrated mass flow controller of mixture gas
4	gas pressure regulator of oxygen	12	diffuser
5	gas pressure gauge of nitrogen	13	wire-mesh debris screen
6	gas pressure gauge of oxygen	14	test specimen holder
7	calibrated mass flow controller of nitrogen	15	test specimen
8	calibrated mass flow controller of oxygen	16	test chimney

Figure 1 — A typical apparatus for determination of oxygen index with one calibrated mass flow meter for nitrogen supply and oxygen supply



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1	nitrogen supply	10	calibrated mass flow meter of oxygen
2	oxygen supply	11	calibrated mass flow meter of mixture gas
3	gas pressure regulator of nitrogen	12	calibrated mass flow controller
4	gas pressure regulator of oxygen	13	diffuser
5	gas pressure gauge of nitrogen	14	wire-mesh debris screen
6	gas pressure gauge of oxygen	15	test specimen holder
7	calibrated mass flow controller of nitrogen	16	test specimen
8	calibrated mass flow controller of oxygen	17	test chimney
9	calibrated mass flow meter of nitrogen		

Figure 2 — A typical apparatus for determination of oxygen index with separate calibrated mass flow meters for nitrogen supply and oxygen supply

5.5 Flame igniter, as specified in ISO 4589-2:2017, 5.6.

5.6 Fume extraction system, as specified in ISO 4589-2:2017, 5.8.

5.7 Raw cotton analyser or carding machine for staple fibres, for loosening and carding the staple fibres.

Other method or device may be used to achieve the purpose of loosening and carding.

5.8 Spinning device, for spun yarn with a linear density less than 60 tex.

Simple spinning device may be used, when a small amount of test specimens tested.

A simple spinning device is described in Annex A, whereas other devices with similar functions are also suitable.

5.9 Skein winding reel, having a circumference (1 000 ± 2) mm, as specified in ISO 2060.

Other method or device may be used to obtain the skein of a certain length.

5.10 Yarn twist counter, as specified in ISO 2061.

5.11 Balance, with a resolution of 0,01 g.

5.12 Straight scale, with an accuracy of ±1 mm.

5.13 Timing device, with measuring range at least 5 min, to an accuracy of ±0,2 s.

5.14 Airtight vessel, to store test specimens after conditioning.

6 Preparation of the test specimens

6.1 Test specimens for staple fibres

6.1.1 Card staple fibres by a raw cotton analyser or carding machine for staple fibres (5.7), to open the crimped bundles and for the fibres to be in a single fibre fluffy state.

6.1.2 Take out, randomly and uniformly, 20 g carded fibres as a spinning sample.

6.1.3 Spin the sample by a spinning device (5.8) to spun yarn with a linear density less than 60 tex.

6.1.4 Smoothly reel the spun yarn to a certain length (integer number of turns) on a skein winding reel (5.9) and prepare a skein with mass of (0,30 ± 0,03) g.

When the above test specimen mass is not applicable, test specimen mass will be determined on agreement between the interested parties and shall be stated in the test report.

Remove the skein from the reel. The skein is approximately 500 mm in length and 0,30 g in mass.

6.1.5 Twist the skein by a yarn twister counter (5.10). Twist number is normally between [110, 150] turns.

When the above twist number is not applicable, twist number will be determined on agreement between the interested parties and shall be stated in the test report.

Fold the twisted skein along the middle part into a multifolded yarn and tie two ends together as a test specimen.

Appropriate twist number should be selected to ensuring the multifolded yarn straight and stiff, according to the characteristics of fibres, and number of the twist of single yarn.

6.1.6 Repeat the procedures given in 6.1.1 to 6.1.5, and prepare at least 15 test specimens.

6.1.7 Singeing treatment before conditioning is suggested, when a large amount of hairiness on the surface of test specimens which easily causing flash burning.

6.2 Test specimens for filament yarns

6.2.1 Smoothly reel the filament yarn to a certain length (integer number of turns) on a skein winding reel (5.9) and prepare a skein with mass of (0,30 ± 0,03) g.

When the above test specimen mass is not applicable, test specimen mass will be determined on agreement between the interested parties and shall be stated in the test report.

Remove the skein from the reel. The skein is approximately 500 mm in length and 0,30 g in mass.

6.2.2 Twist the skein by a yarn twister counter (5.10) in same twist direction as the filament yarn. Twist number is normally between [60, 80] turns.

When the above twist number is not applicable, the twist number will be determined on agreement between the interested parties and shall be stated in the test report.

Fold the twisted skein along the middle part into a multifolded yarn and tie two ends together as a test specimen.

The appropriate twist number should be selected to ensuring the multifolded yarn straight and stiff, according to the characteristics of fibres.

6.2.3 Repeat the procedures given in 6.2.1 to 6.2.2, and prepare at least 15 test specimens.

6.2.4 The test specimen is generally not washed. If it is necessary to be washed, it shall be determined on agreement between the interested parties and shall be stated in the test report. Cleaning process is in accordance with ISO 6741-3:1987, Method A1.

6.3 Marking of test specimens

To observe the burning distance of a test specimen, mark the test specimen with transverse lines at 50 mm from the ignition end. If wet inks are used, the marks shall be dried before ignition.

7 Preconditioning and conditioning

7.1 Atmosphere for conditioning

The standard atmosphere shall be as specified in ISO 139.

7.2 Pre-conditioning

When the actual moisture regain exceeds the commercial one, the test specimen shall be pre-conditioned as specified in ISO 139.

7.3 Conditioning

Condition the test specimens in standard atmosphere (7.1) for at least 2 h until equilibrium is attained.

Unless otherwise specified, the test specimen should be considered to be in equilibrium when successive weighing, at intervals of 2 h, shows no progressive change in mass greater than 0,25 %.

7.4 Test specimens storage

Seal test specimens in an airtight vessel (5.14) after conditioning.

8 Procedure for determination of oxygen index (OI)

8.1 Test environment

The atmosphere for testing shall have a temperature of [10 °C, 30 °C] and a relative humidity of [30 %, 70 %].

The apparatus may be installed in a fume hood as to remove smoke or ash after each test. However, the ventilation system shall be closed during burning process to avoid affecting test results.

8.2 Initial volume fraction of oxygen

Select the initial volume fraction of oxygen based on empirical value of similar fibres or ignition of a test specimen in air.

— If the test specimen burns rapidly in air, the initial oxygen concentration is selected to be about 18 %.

— If the test specimen burns slowly or intermittently in air, the initial oxygen concentration is selected to be about 21 %.

— If the test specimen is self-extinguishing after being off the ignition source in air, the initial oxygen concentration is selected to be at least 25 %.

8.3 Test specimen mounting

Ensure the vertical state of the test chimney.

Fix the knotted end of the test specimen in the middle of the holder, while the tip of the test specimen is at least 65 mm from the clamping position.

Mount the holder vertically in the centre of the chimney, so that the tip of the test specimen is at least 100 mm below the top of the chimney and the lowest end of exposed part of the test specimen is at least 100 mm above the bottom of the chimney.

8.4 Gas control unit adjustment

Adjust gas mixing and flow control devices to the desired volume fraction of oxygen. Make the adjusted gas mixture flow through the chimney at a flow rate of (40 ± 2) mm/s.

Before igniting the test specimen, flush the chimney with gas mixture for at least 30 s, as to remove air from the chimney.

Maintain the flow rate during ignition and combustion.

Calculate the flow rate according to ISO 4589-2:2017, A.2.

8.5 Test specimen ignition

After adjusting the flame plume length of igniter to (16 ± 4) mm, put the nozzle into the test chimney.

Apply the lowest part of the flame to the tip surface of the test specimen, without touching the side of the test chimney.

Apply the flame for up to 10 s, while removing once every 5 s to observe whether the test specimen is ignited or not. If the entire tip surface of the test specimen is burning, consider the test specimen as ignited.

Remove the igniter immediately and start measurement of the burning duration and observation of the burning length.

8.6 Burning behaviour observation and recording

8.6.1 If the test specimen is self-extinguishing after ignition, the cumulative burning duration of continuous combustion and smouldering is less than 180 s, and the cumulative burning length of continuous combustion and smouldering is less than 50 mm, the volume fraction of oxygen is too low; record it with an "O" response symbol.

If the cumulative burning duration is more than 180 s, or the cumulative burning length is more than 50 mm, the volume fraction of oxygen is too high; record it with an "X" response symbol.

Read the volume fraction of oxygen according to the indication of the oxygen analyser; or calculate it according to Formula (1).

$$c_O = \frac{V_O}{V_O + V_N} \times 100\,\% \qquad (1)$$

where

c_O is volume fraction of oxygen, expressed in percentage;

V_O is volume of oxygen in the gas mixture, expressed in litres (l);

V_N is volume of nitrogen in the gas mixture, express in litres (l).

8.6.2 Note the burning characteristics of the material, such as melt drip, smoke, soot, char, afterflame, afterglow or others that need to be recorded.

8.6.3 Remove the test specimen and clean the chimney and the igniter. Cool or replace the chimney as to regain a temperature of [10 °C, 30 °C].

If the test specimen is long enough, it may be inverted or cut off burned parts for re-using. However, it can only be used to estimate the approximation of the minimum volume fraction of oxygen required for combustion, and the results cannot be included in the calculation of the oxygen index (OI).

8.7 Volume fractions of oxygen successive selection

Based on the "up-and-down method for small samples"[1)], select the successive volume fraction of oxygen with an arbitrary step size.

During the test, select the volume fraction of oxygen as follows:

a) increase the volume fraction of oxygen, if the response symbol of preceding test specimen is an "O";

b) decrease the volume fraction of oxygen, if the response symbol of preceding test specimen is an "X".

8.8 Preliminary volume fraction of oxygen determination

Repeat the procedures given in 8.3 to 8.7, with any convenient step size, until the difference between two volume fractions of oxygen obtained is ≤1,0 %, and one of the response symbol is an "O" while the other is an "X". Record this volume fraction of oxygen with an "O" response symbol as the preliminary volume fraction of oxygen.

These two opposite response symbols do not necessarily occur successively. Meanwhile, the volume fraction of oxygen with an "O" response symbol is not necessarily lower than that with an "X" response symbol.

1) Dixon W.J. The up-and-down method for small samples, American Statistical Association Journal, December 1965, pp 967-968.

8.9 Volume fraction of oxygen changes

8.9.1 Using the preliminary volume fraction of oxygen obtained in 8.8, test one test specimen by repeating the procedures given in 8.3 to 8.6. Record the volume fraction of oxygen and response symbol.

8.9.2 With the step size of d = 0,2 % for volume fraction of oxygen changes, repeat the procedures given in 8.3 to 8.7 to test further test specimens. Record a series of volume fraction of oxygen values and corresponding response symbols, until the response symbol is different from that obtained in 8.9.1.

8.9.3 Maintaining d = 0,2 %, repeat the procedures given in 8.3 to 8.7 to test four more test specimens. Record the volume fraction of oxygen value and corresponding response symbol of each test specimen.

Denote the volume fraction of oxygen value of last test specimen as c_F.

9 Calculation and expression of results

9.1 Calculation of oxygen index (OI)

Express oxygen index (OI) by volume fraction and calculate according to Formula (2).

$$O_I = c_F + K \times d \tag{2}$$

where

O_I is the oxygen index (OI), express in percentage;

c_F is the volume fraction of oxygen value of last test specimen in 8.9.3, express in percentage;

K is a factor to be obtained from Table 1, dimensionless;

d is the step size for volume fraction of oxygen changes, used and controlled in 8.9, express in percentage.

When reporting OI results, express the value in one decimal place with intermediate results being exactly rounded downwards.

When calculating standard deviation $\hat{\sigma}$, calculate the value to two decimal places.

9.2 Determination of K

The value and sign of K depend on the type of response symbol of test specimens tested in 8.9.

First, arrange the last five response symbols obtained in 8.9.2 to 8.9.3, in their testing sequence.

Then, determine as follows:

a) If the first response symbol is an “X”, refer to column 1 of Table 1 to select the row which corresponding to the last five response symbols.

Afterwards, refer to row a) of Table 1 to select the column which corresponding to the number of “O” response symbols obtained in 8.9.1 to 8.9.2.

The intersection of row and column is K value.

b) If the first response symbol is an “O”, refer to column 6 of Table 1 to select the row which corresponding to the last five response symbols.

Afterwards, refer to row b) of Table 1 to select the column which corresponding to the number of “X” response symbols obtained in 8.9.1 to 8.9.2.

The intersection of row and column is *K* value, but with the sign reversed.

Table 1 — Values of *K* for calculating oxygen index (OI)

1	2	3	4	5	6
Responses for the last five measurements	**a)** 0	00	000	0000	
XOOOO	- 0,55	- 0,55	- 0,55	- 0,55	OXXXX
XOOOX	- 1,25	- 1,25	- 1,25	- 1,25	OXXXO
XOOXO	0,37	0,38	0,38	0,38	OXXOX
XOOXX	- 0,17	- 0,14	- 0,14	- 0,14	OXXOO
XOXOO	0,02	0,04	0,04	0,04	OXOXX
XOXOX	- 0,50	- 0,46	- 0,45	- 0,45	OXOXO
XOXXO	1,17	1,24	1,25	1,25	OXOOX
XOXXX	0,61	0,73	0,76	0,76	OXOOO
XXOOO	- 0,30	- 0,27	- 0,26	- 0,26	OOXXX
XXOOX	- 0,83	- 0,76	- 0,75	- 0,75	OOXXO
XXOXO	0,83	0,94	0,95	0,95	OOXOX
XXOXX	0,30	0,46	0,50	0,50	OOXOO
XXXOO	0,50	0,65	0,68	0,68	OOOXX
XXXOX	- 0,04	0,19	0,24	0,25	OOOXO
XXXXO	1,60	1,92	2,00	2,01	OOOOX
XXXXX	0,89	1,33	1,47	1,50	OOOOO
	b) X	XX	XXX	XXXX	**Responses for the last five measurements**

9.3 Verification of step size of oxygen volume fraction changes

Based on the last six responses, calculate the estimated standard deviation $\hat{\sigma}$ according to Formula (3).

$$\hat{\sigma} = \left[\frac{\sum (c_i - O_I)^2}{n-1} \right]^{\frac{1}{2}} \tag{3}$$

where

$\hat{\sigma}$ is estimated standard deviation;

c_i is volume fraction of oxygen values of last six responses in 8.9.2 to 8.9.3;

O_I is the oxygen index, calculated according to Formula (1);

n is the number of measurements.

NOTE In this method, $n = 6$. For $n < 6$, the precision of the method is reduced. For $n > 6$, other statistical criteria would be selected.

The step size of oxygen volume fraction changes shall satisfy the condition of Formula (4).

$$\frac{2}{3}\hat{\sigma} < d < \frac{3}{2}\hat{\sigma} \tag{4}$$

where

$\hat{\sigma}$ is estimated standard deviation;

d is step size of oxygen volume fraction changes.

If satisfied, oxygen index (OI) calculated according to Formula (1), is valid.

Otherwise:

a) if $d < \frac{2}{3}\hat{\sigma}$, increase d value and repeat the procedures given in 8.9.1 to 8.9.3, until the condition of Formula (4) is satisfied;

b) if $d > \frac{3}{2}\hat{\sigma}$, decrease d value and repeat the procedures given in 8.9.1 to 8.9.3, until the condition of Formula (4) is satisfied.

Unless required by relevant material specification, the general d value is not less than 0,2 %.

9.4 Example of test results

A typical example of test results is described in Annex B.

9.5 Precision

See Annex C.

10 Test report

The test report shall include at least the following information:

a) the International Standard used (including its year of publication);

b) all details necessary for the identification of the sample tested;

c) preparation conditions of test specimens if necessary (test specimen mass if it is not according to 6.1.4 and 6.2.1; twist number if it is not according to 6.1.5 and 6.2.2; cleaning process if test specimens are washed);

d) test conditions (the temperature and relative humidity of the test atmosphere);

e) test results;

f) the burning characteristics of the material (see 8.6.2);

g) any deviation, by agreement or otherwise, from the procedure specified;

h) any unusual features observed;

i) the date of the test;

j) any information needs to be indicated.

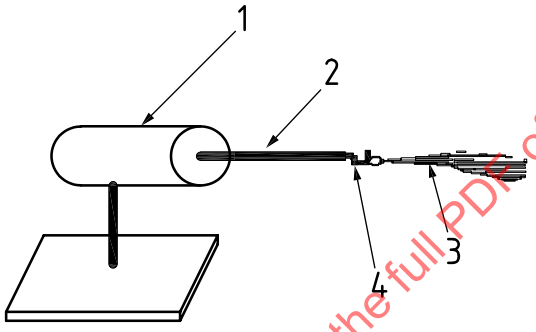
Annex A
(informative)

Example of simple spinning

A.1 Simple spinning device

A.1.1 The main components are a motor, a shaft and a hook. See Figure A.1.

A.1.2 The working principle is that the loose fibre bundle fixed on the hook at one end of the rotating shaft which is driven by the motor, is continuously twisted into yarn under the action of rotation.



Key
1 motor
2 shaft
3 loose fibre bundle
4 hook

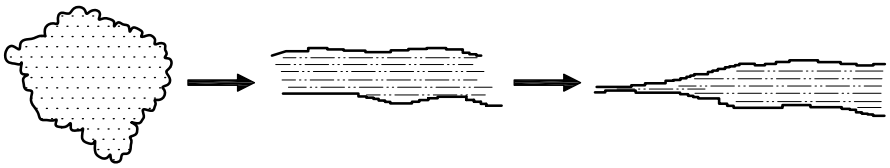
Figure A.1 — Simple spinning device

A.2 Spinning procedure

A.2.1 Take out about 10 g carded staple fibres [see Figure A.2 a)].

Stretch them into a thick strip of fibre bundle about 300 mm in length [see Figure A.2 b)].

Continuously extract a thin strip of fibre bundle about 80 mm in length, from the thick one [see Figure A.2 c)].



a) Carded staple fibre **b) Thick strip of fibre bundle** **c) Thin strip of fibre bundle**

Figure A.2 — Fibre bundle schematic

A.2.2 After slightly hand twisting the thin strip of fibre bundle, pass one end of it around the hook on the spinning device, and merge with another end at point *S* [see Figure A.3 a)]. Fix point *S* by hand.

Set an appropriate rotation speed, and start the spinning device.

With the rotation of the hook, gradually twist the two thin fibre strips in *OS* segment. When the *OS* segment becomes slightly distorted [see Figure A.3 b)], the over-twisting state emerging, turn off the device.

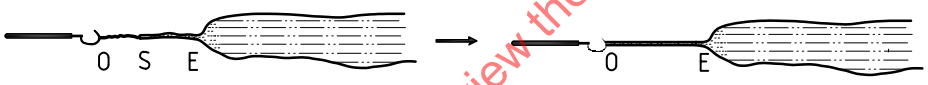
a) Prepare *OS* segment of fibre bundle **b) Prepare *OS* segment of yarn**

Figure A.3 — Preliminary twisting schematic of fibre strips

A.2.3 Still fix point *S* by hand, and stretch the fibre strip backward with another hand. Get a fine fibre strip about 80 mm in length of *SE* segment [see Figure A.4 a)]. Carefully control the pulling speed and strength, to ensure the stretched fine strip continuous and uniform.

After fixing point *E* by hand, loosen the hand fixed at point *S*. Now, the excess twist of *OS* segment begins to spread backward, and *SE* segment is gradually twisted [see Figure A.4 b)].

Pay attention to the twist uniformity of *OE* segment. If it becomes non-uniform, continue to stretch the fine fibre strip and guide the twist to spread backward, or adjust some parts by hand untwisting, to achieve uniform twist.



a) Prepare *OE* segment of fibre bundle **b) Prepare *OE* segment of yarn**

Figure A.4 — Twisting spread schematic of fine fibre strips

A.2.4 Fix point *E* by hand, and start the spinning device.

Continue twisting the *OE* segment until it becomes slightly distorted, and turn off the device.

Repeat the procedures given in A.2.3 to A.2.4, to obtain yarn in sufficient length.

Note that once yarn of about 200 mm in length is spun, wind it on the rotating shaft, pass one end of it around the hook, and continue twisting and spinning.