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Coffee and coffee products — Determination of particle size of ground roasted coffee — Horizontal sieving motion method using circular brushes

Café et dérivés du café — Détermination de la taille des grains de café torréfié moulu — Méthode de tamisage horizontal à l'aide de brosses circulaires

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Foreword

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This document was prepared by Technical Committee ISO/TC 34, *Food products*, Subcommittee SC 15, *Coffee*.

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Coffee and coffee products — Determination of particle size of ground roasted coffee — Horizontal sieving motion method using circular brushes

1 Scope

This document specifies a method for carrying out particle-size distribution analysis of roasted ground coffee by horizontal sieving motion method using circular brushes to minimize the effects of obstruction, agglomeration and adhesion. It specifies general principles to follow concerning apparatus, procedure and presentation of results.

This document is applicable to particle sizes ranging from approximately 150 µm to 2 mm.

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 565, *Test sieves — Metal wire cloth, perforated metal plate and electroformed sheet — Nominal sizes of openings*

ISO 2395, *Test sieves and test sieving — Vocabulary*

ISO 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 9276-1, *Representation of results of particle size analysis — Part 1: Graphical representation*

ISO 9276-2, *Representation of results of particle size analysis — Part 2: Calculation of average particle sizes/diameters and moments from particle size distributions*

ISO 9276-3, *Representation of results of particle size analysis — Part 3: Adjustment of an experimental curve to a reference model*

ISO 9276-4, *Representation of results of particle size analysis — Part 4: Characterization of a classification process*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 2395 apply.

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

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

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

4 Principle

Separate a sample of roasted ground coffee by horizontal sieving machines using circular brushes on each test sieve to obtain reliable analysis.

Annex B compares the results of the horizontal sieving method (with or without brushes) to the results of the laser diffraction method.

5 Apparatus

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

5.1 Horizontal sieve shaker or tap sieve shaker.

The equipment shall be provided with a timer that counts to at least 10 min, as well as all the necessary accessories for its proper and safe functioning.

5.2 Test sieves.

The sieves given in Table 1 shall be used in accordance with ISO 565 and ISO 3310-1. It is not necessary to use sieves smaller than 150 µm (experimental evidence for this is given in Annex C).

Test sieving shall be carried out with a single test sieve or with a series of test sieves with different nominal aperture sizes. A lid and receiver pan should be included in both cases, where appropriate. The number of sieves used in the test should be sufficient to give the requisite information about the material and to avoid excessive wear or blinding.

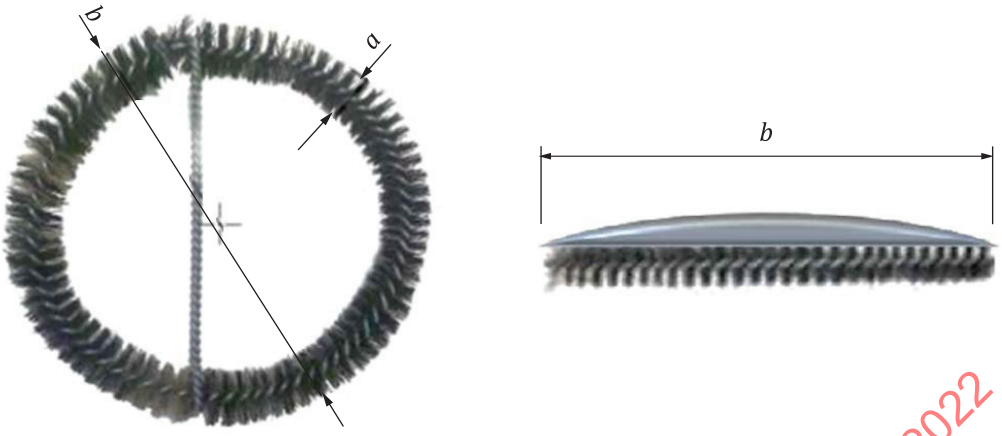
Table 1 — Sieves' opening in accordance with ISO 565 and ISO 3310-1

Series R 20/3 µm	Series R 40/3 µm
1 400	1 180
1 000	850
710	600
500	425
355	300
250	212
180	150

5.3 Balance, capable of weighing to an accuracy of 0,1 g.

5.4 Special circular brush, with a convex or flat cap and a mass not greater than 25 g in order to avoid possible fractionation of the material by the brush. For detailed information on the brushes and their manufacturing, see Annex E.

A special circular brush with a cap is illustrated in Figure 1.



Key

a bristle brush diameter: 13 mm to 17 mm

b circular brush diameter: 80 mm to 100 mm

Figure 1 — Special circular brush with a cap

6 Sample preparation

6.1 General

The sampling method used should be such that the sample taken for sieving is truly representative of sample material from which is has been drawn.

6.2 Division of the sample

The original sample is often too large for direct use in a sieve test: it shall therefore be reduced. In reducing the sample, it is just as important to ensure that the final quantity (test sample) taken for sieving is truly representative of the original sample as it is to ensure that the original sample was representative of the material.

NOTE A precision divider can be used for this purpose.

6.3 Test sample

Take a test sample of a division process, between 40 g to 50 g.

7 Procedure

7.1 Weigh the receiver, making sure it is completely clean.

7.2 Weigh each sieve with one brush (5.4) (this is the initial mass) and assemble in increasing order of mesh opening on top of the receiver.

7.3 Weigh the test portion of sample obtained from 6.2 to the nearest 0,1 g.

7.4 Place the sample in the top sieve and cover with a lid.

7.5 Place the set of sieves in the equipment (5.1), tighten and sieve the sample for 10 min.

7.6 Separate each sieve and weigh with its corresponding special circular brush to the nearest 0,1 g (this is the final mass).

7.7 Calculate the difference between the final mass and the initial mass per sieve.

7.8 Carry out at least two tests.

8 Precision of the method

8.1 Interlaboratory test

Details of the interlaboratory test to determine the precision of the method are summarized in Annex A.

The repeatability and reproducibility limits, as defined in ISO 5725-1, were calculated in accordance with ISO 5725-6.

The model of RRBS, as defined in ISO 9276-3:2008, Table 1, was used to determine the particle size ($x_{50,3}$) of the coffee samples.

8.2 Repeatability

The absolute difference between two independent single test results, obtained using the same method on identical test material in the same laboratory by the same operator using the same equipment within a short interval of time, may not exceed the repeatability limits, *r*, given in Table A.1 in more than 5 % of cases (i.e. repeatability at a 95 % probability).

8.3 Reproducibility

The absolute difference between two single test results, obtained using the same method on identical test material in different laboratories with different operators using different equipment, may not exceed the reproducibility limits, *R*, given in Table A.1 in more than 5 % of cases (i.e. reproducibility at a 95 % probability).

9 Test report

Report results in accordance with ISO 9276-1, ISO 9276-2, ISO 9276-3 or ISO 9276-4.

The test report shall contain at least the following information:

a) any information necessary for the identification of the sample (type, origin and designation of the sample);

b) the date and type of sampling procedure (if known);

c) the date of sample receipt;

d) the date of test;

e) the test results and the units in which they have been expressed as well as statistical information on the accuracy of these results, if required;

f) any special observations made during testing;

g) any operations of the procedure not specified in the method or regarded as optional, which might have affected the results.

Annex D gives a representation of results of particle size analysis using Rosin-Rammler-Bennett-Sperling (RRBS) to determine the particle size of a coffee sample. Refer to ISO 9276-3 for any other models.

NOTE It is possible to use commercial software for this purpose, provided it is validated.

Annex A
(informative)

Precision data of the validation study on the particle sizes of ground roasted coffee for horizontal sieving motion method using circular brushes

An interlaboratory test was carried out with each laboratory performing three determinations, and which gave the statistical information summarized in Table A.1. This information was evaluated in accordance with ISO 5725-2. RRBS was used to determine the particle size ($x_{50,3}$) of the coffee samples.

For samples "coarse" and "medium", the set of Series R 40/3 was used (see Table 1). For the sample "fine", a set of only four sieves up to 600 µm was chosen.

Table A.1 — Precision data of interlaboratory test (results expressed in µm)

Coffee grind size	Coarse	Medium	Fine
Number of laboratories	5	5	5
Mean ($x_{50,3}$)	1 117	762	302
Standard deviation of repeatability, S_r	47	11	7
Coefficient of variation of repeatability, $C_{V,r}$, %	4,2 %	1,5 %	2,4 %
Repeatability limit ($2{,}8 \times S_r$)	132	32	20
Standard deviation of reproducibility, S_R	76	35	44
Coefficient of variation of reproducibility, $C_{V,r}$, %	6,8 %	4,6 %	14,5 %
Reproducibility limit ($2{,}8 \times S_R$)	212	99	122

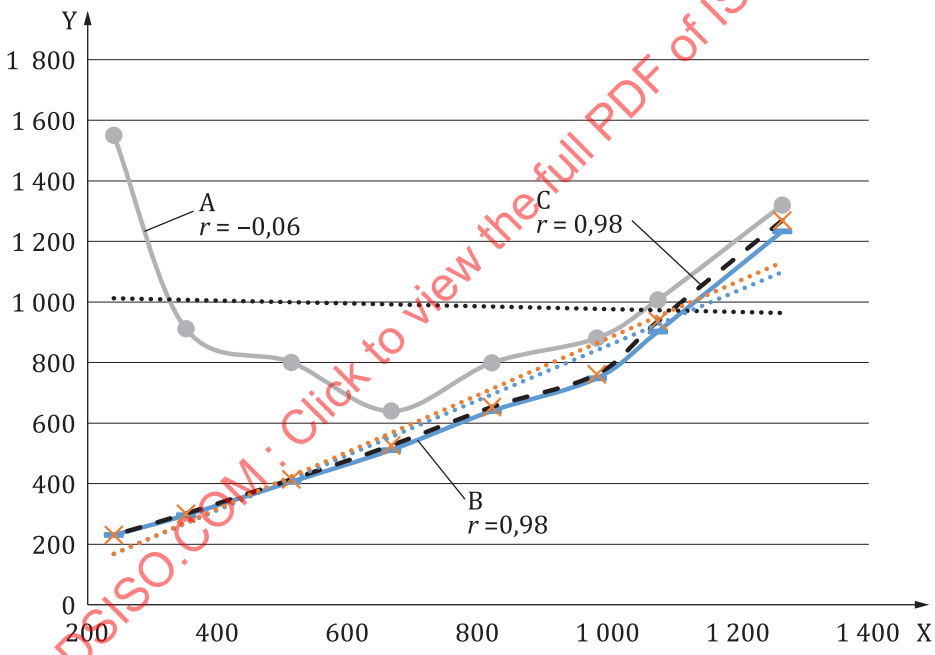
Annex B
(informative)

Reliability of the horizontal sieving method using brushes

In order to establish the reliability of the horizontal sieving method using the brushes, samples within a 240 µm to 1 200 µm range of roasted and ground coffee were tested (with and without brushes), and results were compared with those of the laser diffraction dry method.

Figure B.1 shows that the finer the particle is, the less correlation there is between the result of horizontal sieving motion method without the brushes (r = −0,06) and the laser diffraction method.

When the brushes are used in horizontal sieving either with or without tapping motion, the result of both have a high correlation (r = 0,98) with the laser diffraction method, in the range of 200 µm to 1 400 µm.



Key

X laser diffraction method (µm)
Y horizontal sieving motion method (µm)
A horizontal sieving motion method
B horizontal sieving motion method with brushes
C horizontal sieving motion method with taps and brushes

Figure B.1 — Horizontal sieving motion method versus laser diffraction method

Annex C
(informative)

Sieve size

A sample of very fine ground roasted coffee (obtained by espresso grinder adjusted to its finest setting) was used to establish the smallest mesh size to use with the brushes. The test was carried out using a sample of 40,1 g, a set of brushes (one per sieve) and a set of sieves (see Table C.1), which were sifted for a period of 10 min by a horizontal motion machine.

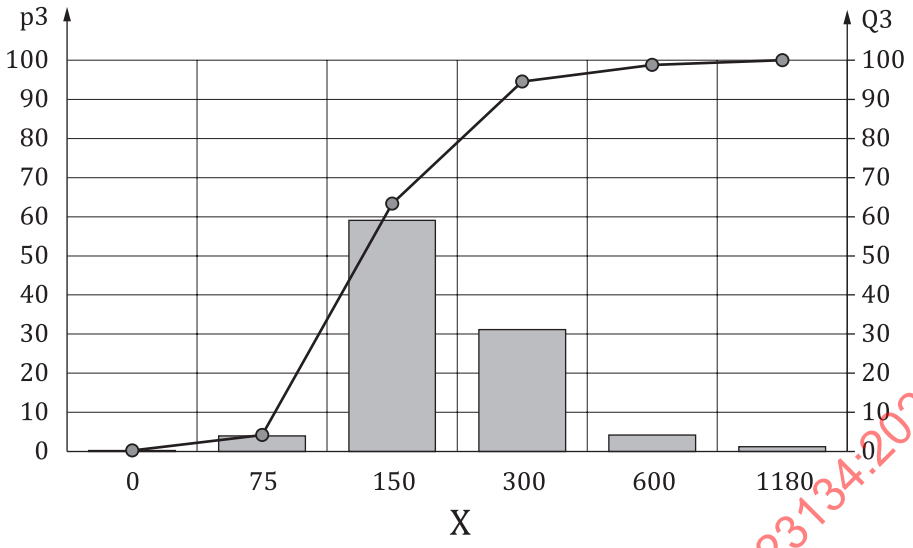
As shown in Table C.1 and in Figure C.1, approximately 95,5 % of the sample has a particle size greater than 150 µm, which indicates only approximately 4,5 % of the sample passed through the150 µm sieve.

According to this result, it is possible to conclude that test sieves with a smaller opening than 150 µm are not necessary for particle size analysis of any grounded roast coffee sample by the horizontal sieving motion method with brushes.

Table C.1 — Fine ground coffee sample cumulative percentage

Sieve, x[a]	**Size class**	**p3**[b]	**Q3**[c]	Δm[d]	**Cumulative** $\Sigma\Delta m$[e]
µm	µm	%	%	g	g
0	< 75	0,25	0,25	0,1	0,1
75	75 to 150	3,99	4,24	1,6	1,7
150	150 to 300	59,10	63,34	23,7	25,4
300	300 to 600	31,17	94,51	12,5	37,9
600	600 to 1 180	4,24	98,75	1,7	39,6
1 180	> 1 180	1,25	100,00	0,5	40,1

[a] Sieves' opening, series R 40/3.
[b] Percentage retained on each sieve (fraction).
[c] Percentage of cumulative distribution.
[d] Mass retained in each sieve.
[e] Cumulative mass.



Key

X sieves' opening, series R 40/3 (µm)

p3 percentage retained on each sieve (fraction) (%)

Q3 percentage of cumulative distribution (%)

Figure C.1 — Percentage of cumulative distribution and percentage retained on each sieve (fraction) of data in Table C.1

Annex D
(informative)

Determination of average particle size calculated by mathematical model

The particle size of a sample of ground roasted coffee is analysed in duplicate, and the data are recorded as given in Tables D.1 and D.2, where the mass measured for each sieve is shown in bold.

The results are applied to a mathematical model known as a linearized equation of RRBS, as shown by Formula (D.1):

$$\ln(-\ln(1-Q)) = n\mathrm{Ln}\, x - n\mathrm{Ln}\, d' \tag{D.1}$$

where

Q is the cumulative distribution;

x is the particle size;

n is the slope;

d' is the intercept parameter of RRSB distribution.

Table D.1 — Data for the calculation of average particle size

Sieve, x µm	**Initial mass,** m_I g	**Final mass,** m_F g	$(m_I - m_F)$ Δm g	$\sum \Delta m$ g	P3 %	<Q3 %	ln(x) µm	ln(ln(1/(1–Q3/100))) %
Receiver pan	**249,46**	**251,12**	1,66	1,66	4,15	0,00		
212	**332,54**	**334**	1,46	3,12	3,65	4,15	5,356 59	−3,160 94
300	**519,28**	**523,04**	3,76	6,88	9,40	7,80	5,703 78	−2,510 72
425	**364,01**	**368,45**	4,44	11,32	11,10	17,20	6,052 09	−1,667 37
600	**386,33**	**398,85**	12,52	23,84	31,30	28,30	6,396 93	−1,100 58
850	**375,02**	**387,73**	12,71	36,55	31,78	59,60	6,745 24	−0,098 34
1 180	**496,28**	**499,73**	3,45	40,00	8,63	91,37	7,073 27	0,896 29

Table D.2 — Data for the calculation of average particle size (duplicate)

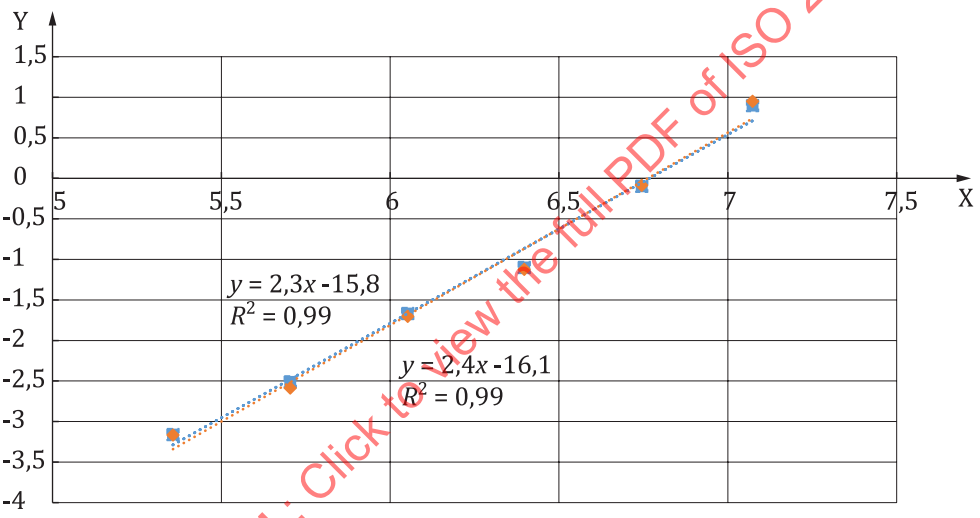
Sieve, x µm	**Initial mass,** m_I g	**Final mass,** m_F g	$(m_I - m_F)$ Δm g	$\sum \Delta m$ g	P3 %	<Q3 %	ln(x) µm	ln(ln(1/(1–Q3/100))) %
Receiver pan	**249,45**	**251,10**	1,65	1,65	4,13	0,00		
212	**332,53**	**333,78**	1,25	3,90	3,13	4,13	5,356 59	−3,167 12
300	**519,32**	**523,09**	3,77	6,67	9,42	4,25	5,703 78	−2,586 77
425	**364,04**	**368,47**	4,43	11,10	11,08	16,68	6,052 09	−1,701 43
600	**386,36**	**399,26**	12,90	24,00	32,25	27,75	6,396 93	−1,123 81
850	**375,02**	**388,01**	12,99	36,99	32,48	60,00	6,745 24	−0,087 42
1 180	**496,29**	**499,30**	3,01	40,00	7,52	92,48	7,073 27	0,950 48

After performing the lineal regression and determining the values of n, d' and r, the average diameter of the distribution is calculated (× 50 %) which is equivalent to calculating a median.

In this example, the values of 743 µm and 741 µm are obtained (see Table D.3). In addition, the fit of the test data to the mathematical reference model RRSB is shown in Figure D.1.

Table D.3 — Calculation of average particle sizes by mathematical model

Sample	Test 1	Test 2
Diameter (× 50 %) (mm)	743	741
Slope (n)	2,3	2,4
Intercept (b)	−15,8	−16,1
Intercept of RRSB (d')	870	865
Correlation coefficient (r)	0,995	0,994



Key

X ln(x)

Y ln(ln(1/(1−Q3[%]/100)))

Figure D.1 — Adjusting the test data to a mathematical reference model RRSB

Annex E
(informative)

Specifications and manufacturing of circular brushes

E.1 General

Considering that the circular brushes are a special accessory, if they are not found on the market, they shall be manufactured in accordance with this annex.

E.2 Specifications and materials

Specifications and materials:

— convex or flat plastic cap with a diameter of 80 mm to 100 mm;

— tube brush with the following characteristics:

  — 13 mm to 17 mm diameter (*a*);

  — light-coloured soft nylon bristles of thickness # 22 or # 23;

  — bristled section (C) with a length between 19 cm and 27 cm, depending on the diameter of the plastic cap;

  — at least 35 cm length including handle (B+C).

See Figure E.1, Figure E.2 and Table E.1 for the dimensions of the cap and the tube brush.

The values of dimensions *a* and *b* should be within the ranges given in Table E.1. The value of C' is calculated with b' as shown in Table E.1.

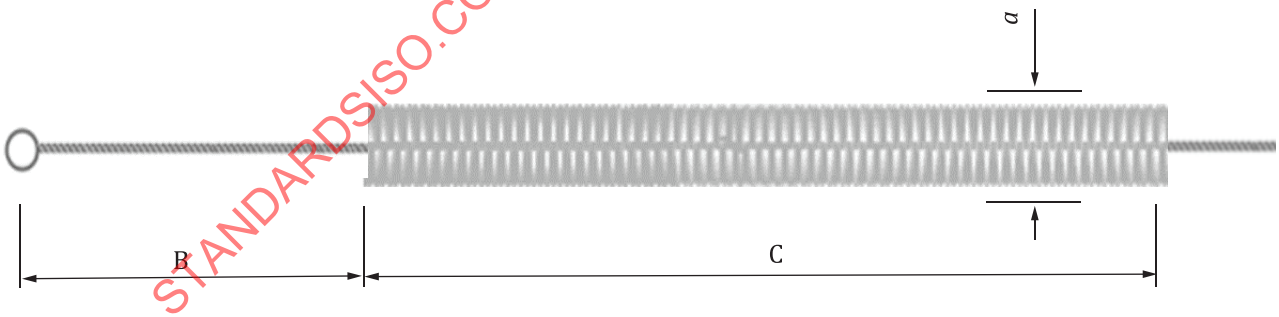


Figure E.1 — Dimensions of the brush

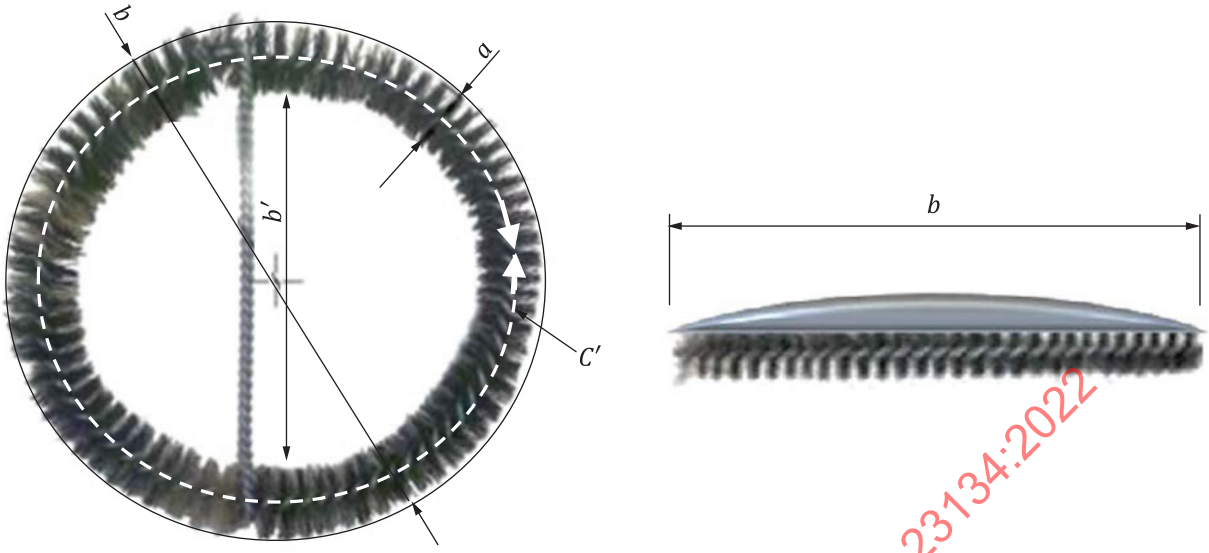


Figure E.2 — Dimensions of the tube brush and cap

Table E.1 — Dimensions of the tube brush and the cap

a	13 mm to 17 mm
b	80 mm to 100 mm
b'	$b' = b - a$
C'	$C' = 2\pi(b'/2)$

E.3 Manufacturing

Bend the wire at 90° at the bristled end of the brush, see Figure E.3.

Figure E.3