INTERNATIONAL STANDARD

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Third edition
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Fine-cut tobacco and smoking articles made from it — Methods of sampling, conditioning and analysis —

Part 3: Determination of total particulate matter of smoking articles using a routine analytical smoking machine, preparation for the determination of water and nicotine, and calculation of nicotine-free dry particulate matter

Tabac à rouler et objets confectionnés à partir de ce type de tabac — Méthodes d'échantillonnage, de conditionnement et d'analyse —

Partie 3: Dosage de la matière particulaire totale des objets à fumer au moyen d'une machine à fumer analytique de routine, préparation pour le dosage de l'eau et de la nicotine, et calcul de la matière particulaire anhydre et exempte de nicotine

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO 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).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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 126, *Tobacco and tobacco products*.

This third edition cancels and replaces the second edition (ISO 15592-3:2008), which has been technically revised.

The main changes are as follows:

— the Introduction has been revised;

— the Normative references have been updated;

— a new term "expanded tobacco" has been added;

— requirement for fine-cut smoking articles with 5,2 mm diameter has been deleted;

— Teepol L has been deleted;

— the unit for the average draw resistance has been extended by mm WG (water gauge);

— former Annex A "Summary of report of the CORESTA task force on roll-your-own (fine-cut) tobacco" has been deleted and subsequent annexes have been relabelled;

— the wrapper specifications for wrapper type A have been modified for filler $CaCO_3$, whiteness and opacity (new Annex A);

— wrapper specifications for wrapper type B have been deleted;

— a new Annex B "Expanded tobacco" has been added;

— Annex C "Classification of wrappers" has been deleted;

— Annex D "Fine-cut tobacco stated to be used with specified wrappers" has been deleted;

— Annex E “Effect of incorporation of loose filters” has been deleted;

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— the Bibliography has been updated.

A list of all parts in the ISO 15592 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.

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Introduction

Smokers make smoking articles by enclosing fine-cut tobacco in a suitable wrapper (sometimes incorporating a filter) either by hand or by using a rolling/tubing machine. The scientific literature has shown that the quantity of fine-cut tobacco, the type of wrapper chosen and the size of the articles made vary widely between consumers and between countries[1].

When the article is smoked, the yield of nicotine free dry particulate matter [NFDPM (sometimes referred to as "tar")] and nicotine is determined by the construction of the article. This document has been developed to specify how articles are made in the laboratory, how they are tested. Fine-cut smoking articles made by consumers can therefore differ from the fine-cut smoking articles made for the purpose of testing described in this document.

One smoking article is made using one mass of fine-cut tobacco and one tube made from a specified wrapper (see Annex A).

It should be noted that because the use of fine-cut tobacco is so dependent on the way in which an individual makes a smoking article, a comparison of the smoke yield with the single result from factory-manufactured cigarettes according to ISO 4387 is of limited value.

This document also gives further necessary procedures as follows in Annex B.

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Fine-cut tobacco and smoking articles made from it — Methods of sampling, conditioning and analysis —

Part 3: Determination of total particulate matter of smoking articles using a routine analytical smoking machine, preparation for the determination of water and nicotine, and calculation of nicotine-free dry particulate matter

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1 Scope

This document specifies methods for the determination of total particulate matter and preparation for the subsequent determination of nicotine-free dry particulate matter present in the smoke from articles made from fine-cut tobacco and expanded tobacco, generated and collected using a routine analytical smoking machine.

It specifies the method of making of fine-cut tobacco smoking articles using a tube made from a specified wrapper.

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 3308:2012, *Routine analytical cigarette-smoking machine — Definitions and standard conditions*

ISO 4387, *Cigarettes — Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine*

ISO 6488, *Tobacco and tobacco products — Determination of water content — Karl Fischer method*

ISO 6565, *Tobacco and tobacco products — Draw resistance of cigarettes and pressure drop of filter rods — Standard conditions and measurement*

ISO 10315, *Cigarettes — Determination of nicotine in total particulate matter from the mainstream smoke — Gas-chromatographic method*

ISO 10362-1, *Cigarettes — Determination of water in total particulate matter from the mainstream smoke — Part 1: Gas-chromatographic method*

ISO 15592-1, *Fine-cut tobacco and smoking articles made from it — Methods of sampling, conditioning and analysis — Part 1: Sampling*

ISO 15592-2, *Fine-cut tobacco and smoking articles made from it — Methods of sampling, conditioning and analysis — Part 2: Atmosphere for conditioning and testing*

ISO 16055, *Tobacco and tobacco products — Monitor test piece for smoking machine — Requirements and use*

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3 Terms, definitions and symbols

3.1 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

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

3.1.1
fine-cut tobacco
tobacco produced to be used by consumers for making their own smoking articles

3.1.2
fine-cut smoking article
FCSA
article, suitable for smoking, produced by combining *fine-cut tobacco* (3.1.1) with a *wrapper* (3.1.4)

3.1.3
expanded tobacco
tobacco, which has increased in size by a process of cellular expansion

3.1.4
wrapper
paper specially prepared and supplied in a form suitable for enclosing *fine-cut tobacco* (3.1.1) so as to produce a smoking article

3.1.5
total particulate matter
TPM
portion of the mainstream smoke which is retained in the smoke trap

3.1.6
dry particulate matter
DPM
total particulate matter (3.1.5) after deduction of its water content

3.1.7
nicotine-free dry particulate matter
NFDPM
dry particulate matter (3.1.6) after deduction of its nicotine content

3.1.8
smoking process
use of a smoking machine to smoke *fine-cut smoking articles* (3.1.2) from lighting to final puff

3.1.9
smoking run
specific *smoking process* (3.1.8) to produce such smoke from a sample of *fine-cut smoking articles* (3.1.2) as is necessary for the determination of the smoke components

3.1.10
laboratory fine-cut tobacco sample
sample of *fine-cut tobacco* (3.1.1) intended for laboratory inspection or testing and which is representative of the gross sample or the sub-period sample

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3.1.11
conditioned laboratory fine-cut tobacco sample
sub-sample of *fine-cut tobacco* (3.1.1) selected from the *laboratory fine-cut tobacco sample* (3.1.10) and conditioned prior to making *laboratory fine-cut smoking articles* (3.1.12)

3.1.12
laboratory fine-cut smoking article
fine-cut smoking articles (3.1.2) made from the *conditioned laboratory fine-cut tobacco sample* (3.1.11)

3.1.13
test sample
fine-cut smoking articles (3.1.2) for test taken at random from the *laboratory fine-cut smoking articles* (3.1.12) and which are representative of the laboratory fine-cut smoking articles

3.1.14
test portion
group of *fine-cut smoking articles* (3.1.2) prepared for a single determination of TPM and which is taken at random from the *test sample* (3.1.13)

3.1.15
butt length
length of unburned *fine-cut smoking article* (3.1.2) remaining at the moment when the smoking is stopped

3.1.16
insertion depth
length from the butt end to which a *fine-cut smoking article* (3.1.2) is inserted into the holder

3.1.17
filling power
measure of the volume occupied by a given mass of *fine-cut tobacco* (3.1.1) when a given pressure is applied

3.1.18
firmness
property of a tobacco rod measured through its deformation when subjected to a given load

3.2 Symbols

The following symbols are used in 9.1 and 9.5:

N is the number of FCSAs of a given group to be smoked, resulting from sampling at one point in time or from a sub-period sample;

C is a multiplying factor, value ≥1, to allow for loss due to damage or selection procedures between initial sampling and smoking;

n is the number of replicate determinations of total particulate matter;

q is the number of FCSAs smoked into the same trap;

Q is the total number of FCSAs (*laboratory fine-cut smoking articles*, see 3.1.12).

4 Principle

The fine-cut tobacco is sampled and conditioned prior to manufacturing laboratory fine-cut smoking articles. The laboratory fine-cut smoking articles are made from the conditioned laboratory fine-cut tobacco sample, are conditioned, then smoked by a routine analytical cigarette-smoking machine, with simultaneous collection of the total particulate matter in a glass fibre filter trap. If considered

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necessary, the consistency of the smoking process and subsequent analytical procedures shall be checked by using monitor test pieces specified in ISO 16055. The mass of total particulate matter so collected is determined gravimetrically. The total particulate matter from the trap is extracted for the determination of the water and nicotine contents by gas chromatography.

In laboratories that are not in a position to use gas-chromatographic methods, reference should be made to Reference [6] for the determination of total nicotine alkaloids, and the determination of water in smoke total particulate matter should be performed by the method described in ISO 10362-2. In such cases, values obtained for nicotine and water in smoke total particulate matter may be used with the addition of a note made in the expression of the result.

5 Apparatus

Normal laboratory apparatus and, in particular, the following items.

5.1 Routine analytical cigarette-smoking machine, in accordance with the general requirements of ISO 3308, except that the holder described in ISO 3308:2012, 4.8, shall be replaced by a holder as described in 5.2, referred to as "smoking machine" in this document

5.2 Holder for FCSAs

FCSAs shall be held by means of a latex sleeve holder. The holder shall be impermeable to smoke components and to air. Either the holder or the smoke trap shall be equipped with a device to position a latex sleeve to hold the FCSA. In front of the latex sleeve shall be a positioning ring with a central orifice slightly larger than the diameter of the FCSA to be tested. The latex sleeve and up to the front edge of the positioning ring shall cover 13 mm ± 0,5 mm from the butt end of the FCSA. The device shall enable a source of vacuum to be applied, sufficient to enlarge the latex sleeve until it is in contact with the sleeve bobbin wall to facilitate the easy placing of the FCSA within the sleeve. The vacuum shall be released once the FCSA is in position.

The dimensions of the components of the latex sleeve holder, suitable for use with FCSAs of 7,2 mm diameter, and a schematic illustration of a suitable assembly are given in Figure 1.

5.3 Device for making fine-cut smoking articles (FCSAs)

The device used to make FCSAs shall be constructed so that it first compresses the predetermined quantity of fine-cut tobacco in a controlled manner and then fills a preformed wrapper tube with the compressed fine-cut tobacco portion. The device shall be suitable for the dimensions of the length and diameter of the wrapper tube specified in 7.2.

5.4 Soap bubble flow meter, graduated at 35 ml to an accuracy of ±0,2 ml with a resolution of 0,1 ml.

A calibrated electronic device may also be used to measure puff volume, provided that the calibration is traceable to a primary measurement.

5.5 Apparatus for the determination of puff duration and frequency

The accuracy shall be such as to ensure that a 1 % error in the puff duration can be detected. The timer should be connected directly to the triggering circuits in the smoking machine.

NOTE It is not possible to specify the method of measurement beyond a statement of principle because of the variety of types of suitable timers and smoking machines available.

5.6 Analytical balance, suitable for measuring to the nearest 0,1 mg.

NOTE The weighing of glass fibre filter pad holders can be affected by static electricity, necessitating the use of an antistatic device.

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5.7 Conditioning enclosure, carefully maintained under the conditions specified in ISO 15592-2.

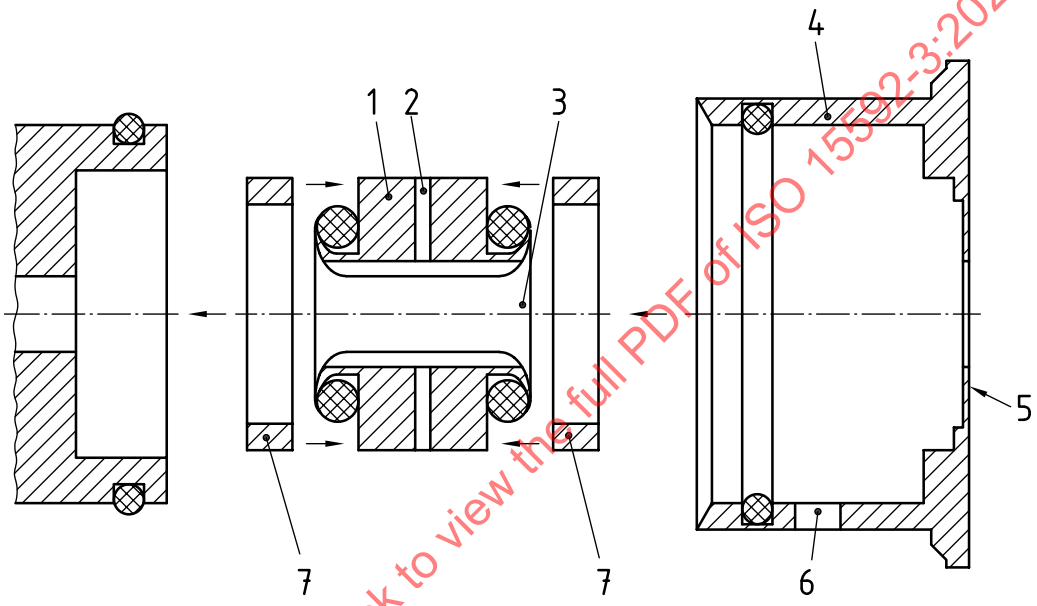
5.8 Length-measuring device, suitable for measuring to the nearest 0,5 mm.

5.9 Apparatus for the determination of diameter, suitable for measuring to the nearest 0,2 mm.

NOTE The tolerance is based on the wrapper that has been slit and measured to the nearest 0,5 mm.

5.10 Smoke trap sealing device, end caps made from a non-hygroscopic and chemically inert material.

5.11 Gloves, cotton or non-talc surgical gloves.



Key

1 bobbin
2 vacuum channel
3 latex sleeve
4 cap
5 positioning ring integrated into cap
6 vacuum channel
7 sealing washer

FCSA holder dimensions FCSA diameter 7,2 mm	
Bobbin	
Internal diameter	9,0 mm
Length	12,2 mm
Latex sleeve before insertion in holder	
Internal diameter	6,0 mm
Length	19,0 mm
Wall thickness	0,35 mm

Figure 1 — FCSA holder

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6 Sampling

A laboratory fine-cut tobacco sample shall be provided by a sampling scheme according to ISO 15592-1. This sample will normally contain fine-cut tobacco taken randomly from the different parts of the population under test.

7 Wrapper used for making FCSAs

7.1 General

Annex A specifies the wrapper (see A.2) which shall be used in order to determine the NFDPM of a laboratory fine-cut tobacco sample.

7.2 Manufacture of standard tubes from the wrapper

The device for making fine-cut smoking articles (see 5.3) relies on the wrapper being supplied in the form of a tube. Therefore, the wrapper specified in Annex A may need to be made into tubes prior to use. Experience has shown that it is suitable to use cylindrical formers of diameter 7,0 mm as appropriate. The standard tube shall have a diameter of (7,2 ± 0,05) mm and a length of (70 ± 0,5) mm.

7.3 Determination of average tube mass

Weigh 10 tubes made in accordance with 7.2 and conditioned in accordance with ISO 15592-2. Determine the average mass of the tube. This mass is required to determine the target average mass of the finished fine-cut smoking articles.

8 Making of the laboratory fine-cut smoking articles

8.1 General

Laboratory fine-cut smoking articles shall be made from a conditioned laboratory fine-cut tobacco sample.

If the fine-cut tobacco blend to be tested contains tobacco of high filling power, the mass of the fine-cut tobacco shall be adjusted appropriately according to Annex B.

It is possible to allow for differences in filling power to control the firmness of the fine-cut smoking articles made from the fine-cut tobacco, for example if the volume occupied by a given mass of fine-cut tobacco is normally A and a sample is found that occupies A + B, then the mass should be multiplied by a factor A/(A + B). If the mass of fine-cut tobacco is adjusted, this shall be mentioned in the test report.

NOTE At the time of publication of this document, there was no internationally agreed definition of filling power or firmness of smoking articles. However, different types of apparatus for measuring each parameter are commercially available and generally used in the tobacco industry.[1)]

The laboratory fine-cut tobacco smoking articles shall be conditioned in accordance with ISO 15592-2.

8.2 Specification of the FCSAs to be made

Each of the laboratory fine-cut smoking articles shall be made by enclosing 750 mg of fine-cut tobacco from the conditioned laboratory fine-cut tobacco sample in a tube specified in 7.2 made from the wrapper (see A.2).

1) Furthermore, there is a standard, ISO/AWI 4906, under preparation.

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8.3 Preparation and selection of the FCSAs for test

8.3.1 Use the making device (5.3) to prepare sufficient fine-cut smoking articles required by the smoking plan and to allow for rejection due to damage or unacceptable quality (like visual defects, e.g. bad shape, uneven filling, etc.). Reject poorly made fine-cut smoking articles accordingly. The end of the FCSA attached to the making device shall be identified for subsequent insertion in the fine-cut smoking article holder (see 9.2.3).

8.3.2 As the physical properties of FCSAs can vary, it is necessary to select the samples for smoking. Selection is done on the basis of mass and shall be carried out as follows: The FCSAs shall be weighed individually after manufacture and those whose mass differs from the target mass of the fine-cut tobacco plus average tube mass by more than 20 mg shall be rejected. The making may be performed by taking an individual weighed fine-cut tobacco portion, making the fine-cut smoking article and then checking the mass, or a number of fine-cut tobacco portions may be weighed in the shortest possible time, fine-cut smoking articles made and then selected by mass.

8.3.3 The mean mass of 22 FCSAs after reconditioning shall be within ±10 mg of the target mass of the fine-cut tobacco plus average tube mass (see 9.4).

8.3.4 If a selection by mass is necessary, the selection shall not be considered as a method of reducing the number of fine-cut smoking articles to be smoked.

8.4 Special precautions

The fine-cut tobacco used to make the FCSAs described in 8.2 is conditioned at (22 ± 2) °C and (75 ± 3) % RH in accordance with ISO 15592-2. The laboratory conditions under which FCSAs are prepared are normally at a different temperature and RH. Consequently, the fine-cut tobacco moisture content can change during the making process and so precautions shall be taken to minimize this. The fine-cut tobacco shall be kept as closely as possible to the specified conditions until used for the making of FCSAs. This can be done in one of two ways:

1) only sufficient fine-cut tobacco shall be removed from the conditioning enclosure to make a limited number of fine-cut smoking articles such that the fine-cut tobacco is likely to remain in condition during the making process;

2) conditioned fine-cut tobacco shall be placed in separate sealed containers such that the fine-cut tobacco in one container is likely to remain in condition during the making process.

It is not possible to specify the number of fine-cut smoking articles that can be made whilst keeping the fine-cut tobacco in condition, as this will depend on the experience of the operator. It will also depend on the conditions under which the fine-cut smoking articles are made. A period not exceeding one hour is recommended. The condition of the fine-cut tobacco will in any case be determined prior to smoking as there is a requirement to check the mass specified in 9.4.

In addition, it is necessary to ensure that the FCSAs do not change in mass whilst in the smoking room. A smoking run using a linear smoking machine could take up to 2 h and during this time, the mass of the FCSAs could change if they are kept in unsealed containers. It is necessary to ensure that just sufficient FCSAs are kept in sealed containers until they are required to be loaded into the smoking machine. In the case of the rotary machine, this will normally be twenty plus two spares, and in the case of the linear machine, this will normally be five with the two spares kept separately.

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9 Determination of total particulate matter

9.1 Preparation of the fine-cut smoking articles for smoking

9.1.1 Number of samples

If N FCSAs of a given group are to be smoked, $C \times N$ FCSAs shall be prepared from Q FCSAs for conditioning and butt and insertion depth marking.

The multiplier C is usually at least 1,3 to provide extra FCSAs in case some are damaged and for any optional tests which can be required (see 9.4). If selection is necessary, C shall be larger (perhaps 2,0 depending on the selection process).

The precision normally required generally demands that $100 \leq N \leq 200$. This number may be considerably augmented if the variability of the sample is high. It may be reduced when N represents a sub-period sample. N shall never be less than forty when twenty FCSAs are smoked per trap, or less than twenty when five FCSAs are smoked per trap.

At least forty FCSAs are required to be smoked when twenty FCSAs are smoked per trap, thus providing replicate analyses and data replication.

The N FCSAs to be smoked shall be tested in $n = N/q$ determinations if q FCSAs are smoked into one trap. As far as possible, these n determinations should correspond to different test portions of the test sample. Selection of each test portion will depend upon the form of the test sample.

9.1.2 Selection of test portions from a bulk of Q FCSAs

For each group of fine-cut smoking articles, the test sample is in the form of a single bulk, consisting of Q FCSAs. Select at random $C \times N$ FCSAs so that every FCSA has an equal probability of being selected.

9.1.3 Duplicate test portions

Provided that the sample of laboratory fine-cut smoking articles is sufficiently large ($> 2\ C \times N$), a duplicate set of n test portions should be reserved. In this event, the parallel selection of a test portion and its duplicate is desirable.

9.2 Marking the insertion depth and butt length

9.2.1 Standard insertion depth

The standard insertion depth for FCSAs made as in 8.3 shall be 13 mm ± 0,5 mm.

9.2.2 Standard butt length

The standard butt length for FCSAs made as in 8.3 shall be 27 mm.

9.2.3 Marking the insertion depth and butt length

The end of the tube that is attached to the making device shall be the one that is placed in the holder. Mark it on the lap to facilitate marking of insertion depth and butt length.

The insertion depth and butt length shall be marked on the FCSAs before conditioning. It is recommended that two thin lines be drawn using a fine, soft-tipped marker. Draw the first line at the insertion depth to an accuracy of 0,5 mm from the butt end of the FCSAs, corresponding to the standard depth of insertion, and the second at the standard butt length to an accuracy of 0,5 mm from the butt end for the particular FCSA group.

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Care should be taken to avoid damaging the FCSAs during marking. Any FCSAs accidentally torn or punctured during marking, or any found during marking to be defective, shall be discarded and replaced with spare FCSAs from the test portion.

If FCSAs are to be smoked on a smoking machine on which the butt length in accordance with 9.2.2 can be pre-set, it is not necessary to mark the butt lengths on the FCSAs themselves.

9.3 Conditioning and testing conditions

Condition all the test portions in the conditioning atmosphere specified in ISO 15592-2 [temperature (22 ± 2) °C and relative humidity (75 ± 3) %] for a minimum of 72 h and a maximum of 10 d.

If, for any reason, laboratory fine-cut tobacco samples or laboratory fine-cut smoking articles are to be kept longer than 10 d, store them in the original packaging or in airtight containers just large enough to contain the sample.

The testing atmosphere in the laboratory where the smoking is to be carried out shall be in accordance with ISO 15592-2 [temperature (22 ± 2) °C and relative humidity (60 ± 5) %].

Transfer the test portions to the smoking location in airtight containers just large enough to contain the test portions (see 8.4).

9.4 Preliminary tests before smoking

A check of the average mass of the FCSAs from each test portion shall be made by weighing. Each average mass shall differ from the combined average tube mass and specified fine-cut tobacco mass by no more than 10 mg.

IMPORTANT — It is important that the instructions given in 8.4 are observed, otherwise the average mass is likely to exceed this specification.

Determine the following data, if it is required in the test report:

a) total length of the FCSA wrapper to the nearest 0,5 mm;

b) diameter: FCSAs are soft and often irregular in shape, so tests for diameter which impose pressure on the article or rely on pneumatic methods may give rise to inaccurate data and shall be avoided; the diameter may be determined by measurement of slit wrapper tubes using a scale graduated in millimetres;

c) draw resistance of an FCSA in accordance with ISO 6565, except that the holder specified in 5.2 shall be used; it is essential to check that the holder is free of leakage; use the leakage tester supplied with the smoking machine and test the holder according to the instructions given with the leakage tester;

d) average mass of the conditioned FCSAs selected for smoking (in milligrams per FCSA);

e) water content (as a mass fraction) of the conditioned laboratory fine-cut tobacco sample, determined in accordance with ISO 6488.

9.5 Smoking and collection of particulate matter

9.5.1 Smoking plan

Choose a smoking plan. Examples of plans are given for information in ISO 4387:2019, Annex A.

The plan shall show the number of FCSAs to be smoked into each smoke trap, q, and the number of the FCSAs in the test sample for conditioning ($C \times N$).

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9.5.2 Preparation of smoke traps and FCSA holders

For all operations, the operator shall prevent contamination from the fingers by wearing gloves (5.11) of a suitable material.

Insert glass fibre filter pads that have been conditioned in the test atmosphere (given in ISO 15592-2) for at least 12 h into their holders, and assemble, placing the rough side of the glass fibre filter pad so that it will face the oncoming smoke. After assembly, examine the filter holders to ensure that the glass fibre filter pads have been properly fitted. Fit the sealing devices (end caps). Weigh the assembled smoke traps to the nearest 0,1 mg. Because of the possibility of absorption of water by smoke traps and solvent, determine a value for the sample blank. Prepare sample blanks by treating additional smoke traps (at least 2 per 100 FCSAs) in the same manner as that used for smoke collection.

9.5.3 Setting up the smoking machine

9.5.3.1 General

A smoking machine shall be used in accordance with ISO 3308 except for the holder (5.2).

If necessary, replace any protective filters on the smoking machine. Switch on the smoking machine and allow it to warm up on automatic cycling for at least 20 min.

With the smoking machine warmed up, check that the puff duration, puff frequency and puff volume on each channel are in accordance with the standard conditions.

9.5.3.2 Measurement of puff duration

A timer (5.5) shall be used to measure the period of time, which elapses between the triggering operations, which begin and end a puffing action of the smoking machine.

9.5.3.3 Checking of puff frequency

Measure the period of time, which elapses between the triggering operations, which begin successive puffing actions of the smoking machine, thus determining the puff frequency.

9.5.3.4 Measurement of puff volume

The displacement of the bubble in a soap bubble flow meter (5.4) gives a direct measurement of puff volume and provides a check for leaks in the system. It shall be connected through a standard pressure drop device of 1 kPa ± 5 % to the FCSA holder of the smoking machine channel under test. Before use on a series of measurements, the instrument shall be wetted twice with detergent solution and then allowed to drain. Make measurements until the values obtained are repeatable.

It is recommended to use the detergent solution as specified by the supplier of the soap bubble flow meter in the corresponding manual.

Fit the prepared smoking trap or traps and FCSA holders on to the smoking machine. Check the operation of the latex sleeve on each FCSA holder by applying the vacuum source and ensuring that the sleeve opens completely. Attach a plastic insert of an appropriate size for the FCSAs to be smoked to the tube from the soap bubble flow meter indicator. Prepare the soap bubble flow meter by wetting the inside of the tube with the detergent solution to above the top graduation mark. Connect the indicator to the FCSA holder in channel 1 and determine the puff volume; adjust, if necessary, to (35 ± 0,3) ml.

Repeat the determinations until this necessary precision of measurement is obtained. If the number of replicates exceeds three, continue until the correct precision is obtained but replace the filter disc before smoking, reweigh the smoke trap and recheck the puff volume with the new filter disc in place.

Measure the temperature and relative humidity of the air surrounding the smoking machine and note the atmospheric pressure.

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9.5.4 Procedure for smoking run

Insert the conditioned FCSAs from the test portion into the FCSA holders so that the insertion is at the standard depth (see 9.2.1). Avoid deformation of the wrapper in order to minimize leaks. Any FCSAs found to have obvious defects, or which have been damaged during insertion, shall be discarded and replaced with spare, conditioned FCSAs.

Ensure that the FCSAs are positioned correctly so that the angle formed by the longitudinal axis of the FCSA and the horizontal plane shall be as small as possible; the axis of FCSAs shall coincide with the axis of the channels. Adjust the position of each FCSA so that when the burning coal reaches the butt mark, the puff termination device is activated. If the burning through of 100 % cotton thread (48 ± 4) mg/m is used to terminate smoking at the butt mark, the cotton shall just touch the FCSA at the butt mark, without modifying the FCSA positioning.

Set the puff counters to zero and light each FCSA at the beginning of its first puff. A non-flame method of ignition shall be used. Should it be necessary to re-light an FCSA, a hand-held electrical lighter may be used. When each butt mark has been reached, remove the burning coal from the FCSA and note the final reading of the puff counters. After the smoking process is complete, leave the FCSA butt in place for at least 30 s to enable deposition of any residual smoke in the trap.

Avoid disturbance of the smoking by artificial removal of ash. Allow the ash to fall naturally into the ashtray.

Then, if required, a new FCSA shall be inserted and the smoking process repeated until the predetermined number of FCSAs, in accordance with the smoking plan, has been smoked into the smoke trap. Immediately begin the determination of total particulate matter as described in 9.6.

For puffs taken after the fine-cut smoking article has been extinguished or removed from its holder (clearing puffs), follow the requirements given in ISO 4387.

9.6 Removal of smoke traps and FCSA holders

Remove the smoke traps from the smoking machine [gloves (see 5.11) shall be worn]. Where necessary, remove the FCSA holder from the smoke trap. Cover the front and back apertures of the trap with the sealing devices (see 5.10).

It is recommended that the removal of the holder be conducted with the smoke trap held with its FCSA-facing side downwards to avoid any possible contaminants from the FCSA holder reaching the glass fibre filter pad.

Immediately after smoking, weigh the smoke traps to the nearest 0,1 mg. Check the back of glass fibre filter pad to ensure that there are no brown stains indicating overloading or damage. Discard any glass fibre filter pad showing such stains or damage.

Glass fibre filter pads of 44 mm diameter are capable of retaining up to 150 mg of total particulate matter (TPM) and glass fibre filter pad of 92 mm diameter can retain 600 mg of TPM. If, during smoking, this mass is exceeded, the results are invalid, and a further smoking test shall be carried out in which the number of FCSAs is reduced, and a calculation made to allow for the reduced number smoked.

9.7 Calculation of total particulate matter

The TPM content is given by Formula (1):

$$m_{\mathrm{TPM}} = \frac{(m_1 - m_0)}{q} \tag{1}$$

where

m_{TPM} is the TPM content for each channel, in milligrams per FCSA;

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m_0 is the mass of the smoke trap before smoking, in milligrams;

m_1 is the mass of the smoke trap after smoking, in milligrams;

q is the number of FCSAs smoked into the trap.

9.8 Treatment of total particulate matter for the determination of water and nicotine

9.8.1 Extraction procedure

Remove the sealing devices from the smoke trap [gloves (see 5.11) shall be worn], open it and remove the glass fibre filter pad with forceps. Fold it twice, total particulate matter inwards, being careful to handle only the edge with forceps and gloved fingers. Place the folded glass fibre filter pad in an appropriately shaped dry flask (maximum 150 ml for 44 mm pads, maximum 250 ml for 92 mm pads). Wipe the inner surface of the filter holder front with two separate quarters of an unused conditioned glass fibre filter pad and add these to the flask. Pipette solvent (propan-2-ol containing the internal standards for both nicotine and water determinations) into the flask (20 ml for 44 mm pads, or 50 ml for 92 mm pads). See ISO 10315 and ISO 10362-1.

Stopper the flask immediately and shake gently on an electric shaker for at least 20 min, ensuring that the pad does not disintegrate. The shaking time should be adjusted to ensure full extraction of the nicotine and water in the particulate matter.

Follow the same procedure with each of the blank smoke traps used for the determination of water.

9.8.2 Water determination and calculation of DPM

Carry out the water determination of the solution in each flask in accordance with ISO 10362-1.

The DPM content is given by Formula (2):

$$m_{\mathrm{DPM}} = m_{\mathrm{TPM}} - m_{\mathrm{w}} \tag{2}$$

where

m_{DPM} is the DPM content for each smoke trap, in milligrams per FCSA;

m_{TPM} is the TPM content, in milligrams per FCSA;

m_{w} is the water content in the TPM, in milligrams per FCSA.

9.8.3 Nicotine determination and calculation of NFDPM

Carry out the nicotine determination of the solution in each flask in accordance with ISO 10315.

The NFDPM content is given by Formula (3):

$$m_{\mathrm{NFDPM}} = m_{\mathrm{DPM}} - m_{\mathrm{N}} \tag{3}$$

where

m_{NFDPM} is the NFDPM content for each smoke trap, in milligrams per FCSA;

m_{DPM} is the DPM content, in milligrams per FCSA;

m_{N} is the nicotine content in the TPM, in milligrams per FCSA.

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10 Test report

10.1 General

The test report shall show the method used and the results obtained. It shall also mention any operating conditions not specified in this document, or regarded as optional, as well as any circumstances that may have influenced the results. The test report shall include all details required for complete identification of the sample. If appropriate, the information given in 10.2 to 10.5 shall be recorded.

10.2 Characteristic data about the fine-cut tobacco

Fine-cut tobacco identification, in the case of a commercial fine-cut tobacco, should include the following:

a) name of the manufacturer, country of manufacture;

b) brand name and sub-brand name;

c) batch number (of that product sampled that day);

d) marks on any tax stamp;

e) printed smoke yields (if any).

10.3 Sampling

The following particulars shall be included:

a) type of sampling procedure;

b) date of sampling;

c) place of purchase or sampling;

d) kind of sampling point;

e) sampling point (e.g. address of retail outlet or machine number);

f) number of laboratory fine-cut smoking articles.

10.4 Description of test

The following particulars shall be included:

a) reference to this document, i.e. ISO 15592-3:2022;

b) date of test;

c) type of smoking machine used;

d) type of smoke trap used;

e) total number of FCSAs smoked;

f) number of FCSAs smoked into each smoke trap;

g) butt length;

h) temperature and relative humidity in the smoking room at the time of smoking;

i) atmospheric pressure (in kilopascals) during smoking operation if outside the specified limits.

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10.5 Test results

The expression of the laboratory data depends on the purpose for which the data are required and the level of laboratory precision. Confidence limits shall be calculated and expressed on the basis of the laboratory data before any rounding has taken place.

The following data shall be given for the test sample. If a wrapper or a tube, different from the one defined in Annex A and 7.2, has been used, the data shall also be given for that wrapper or tube.

a) average length of the FCSA wrapper, in millimetres to the nearest 0,1 mm;

b) butt length to which FCSAs were smoked, in millimetres;

c) average diameter of the FCSAs, in millimetres to the nearest 0,2 mm;

d) average draw resistance of the conditioned FCSAs, in Pascal or mm WG (water gauge) to the nearest 10 Pa or 1 mm WG;

e) average mass, in milligrams per FCSA, of the conditioned tube;

f) average mass, in milligrams per FCSA, of the conditioned fine-cut tobacco;

g) average mass, in milligrams per FCSA, of the conditioned FCSAs selected for the smoking run;

h) water content, as a mass fraction in percent, of the conditioned laboratory fine-cut tobacco sample (see ISO 6488);

i) average number of puffs per FCSA for each channel, to the nearest 0,1 puff;

j) TPM content, in milligrams per FCSA, for each channel to the nearest 0,1 mg, and the average per FCSA to the nearest 1 mg;

k) DPM content, in milligrams per FCSA, for each channel to the nearest 0,1 mg, and the average per FCSA to the nearest 1 mg;

l) smoke nicotine content, in milligrams per FCSA, for each channel to the nearest 0,01 mg, and the average per FCSA to the nearest 0,1 mg;

m) NFDPM content, in milligrams per FCSA, for each channel to the nearest 0,1 mg, and the average per FCSA to the nearest 1 mg;

n) information concerning the re-lighting or any other problem concerning the smoking of any FCSA.

11 Precision and collaborative study

11.1 Collaborative wrapper study

An international collaborative study on the precision of the method was conducted in 2020.

In this study 9 laboratories, 2 wrappers (a wrapper according to Annex A and a wrapper type A of ISO 15592-3:2008) and 1 fine-cut tobacco blend were involved.

11.2 Precision

The difference between two single results, found on matched samples by the same operator using the same apparatus within the shortest feasible time interval, will exceed the repeatability limit, *r*, on average not more than once in 20 cases in the normal and correct operation of the method.

Single results on matched samples reported by two laboratories will differ by more than the reproducibility limit, *R*, on average not more than once in 20 cases in the normal and correct operation of the method.

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