INTERNATIONAL STANDARD

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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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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

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.

ISO 15592-3 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

ISO 15592 consists of the following parts, under the general title *Fine-cut tobacco and smoking articles made from it — Methods of sampling, conditioning and analysis*:

— *Part 1: Sampling*

— *Part 2: Atmosphere for conditioning and testing*

— *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*

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 CORESTA work (see Annex A) and the scientific literature have shown that the quantity of tobacco, the type of wrapper chosen and the size of the articles made vary widely between consumers and between countries.

When the article is smoked, the yield of tar and nicotine is determined by the construction of the article. This part of ISO 15592 has been developed to specify how articles are made in the laboratory, how they are smoked and how information relevant to a fine-cut tobacco sample can be given to the consumer.

Four smoking articles are made using two masses of tobacco and two standard wrappers (see Annex B) with different properties. When these articles are smoked using a routine analytical smoking machine, the resultant data give a matrix of four points offering an indication of how the choice of wrapper and tobacco mass alter the tar and nicotine yields from the tobacco sample. A comparison of yields by choice of the same parameters of the matrix can provide a means of ranking brands.

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 of any one of the matrix points with the single result from factory-manufactured cigarettes according to ISO 4387 is of limited value.

This part of ISO 15592 also gives further necessary procedures as follows.

If there is a need to classify a wrapper of unknown properties, this classification is made according to Annex C.

Fine-cut tobacco is sometimes sold with a statement that it may be used with a specified wrapper with or without a filter. The appropriate measurements are made according to Annex D.

A loose filter is incorporated in some fine-cut smoking articles at the time of making. The effect of the filter is determined according to Annex E.

ISO/TC 126/WG 7, which included experts both from the tobacco industry and from regulatory laboratories, was responsible for the planning, conduct and analysis of the data from a collaborative study by 20 laboratories to determine the repeatability and reproducibility of the method specified in this part of ISO 15592.

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

1 Scope

This part of ISO 15592 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, generated and collected using a routine analytical smoking machine.

It specifies the method for the classification of unknown wrappers by comparison of fine-cut smoking articles made using them with those made using a standard wrapper.

It specifies the method of making of fine-cut tobacco smoking articles using specified wrappers both with and without attached filters, and the method of determination of the yield of a fine-cut smoking article made using a loose filter and tobacco inserted into a wrapper.

2 Normative references

The following referenced documents are indispensable for the application 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 3308:2000, *Routine analytical cigarette-smoking machine — Definitions and standard conditions*

ISO 4387:2000, *Cigarettes — Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine*

ISO 6488:—[1)], *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 smoke condensates — Gas-chromatographic method*

ISO 10362-1, *Cigarettes — Determination of water in smoke condensates — 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*

1) To be published. (Revision of ISO 6488-1:1997)

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 — Requirements and application*

3 Terms, definitions and abbreviated terms

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

3.1
fine-cut tobacco
FCT
tobacco produced to be used by consumers for making their own smoking articles

3.2
fine-cut smoking article
FCSA
article, suitable for smoking, produced by combining fine-cut tobacco with a wrapper

3.3
wrapper
material specially prepared and supplied in a form suitable for enclosing fine-cut tobacco so as to produce a fine-cut smoking article

3.4
total particulate matter
TPM
that portion of the mainstream smoke which is retained in the smoke trap

3.5
dry particulate matter
DPM
total particulate matter after deduction of its water content

3.6
nicotine-free dry particulate matter
NFDPM
dry particulate matter after deduction of its nicotine content

3.7
clearing puff
any puff taken after the fine-cut smoking article has been extinguished or removed from its holder

3.8
smoking process
use of a smoking machine to smoke fine-cut smoking articles from lighting to final puff

3.9
smoking run
specific smoking process to produce such smoke from a sample of fine-cut smoking articles as is necessary for the determination of the smoke components

3.10
laboratory sample
sample intended for laboratory inspection or testing and which is representative of the gross sample or the sub-period sample

3.11
conditioned laboratory fine-cut tobacco sample
sub-sample of the fine-cut tobacco selected from the laboratory sample and conditioned prior to making laboratory fine-cut smoking articles

3.12
laboratory smoking articles
fine-cut smoking articles made from the laboratory sample or test sample of fine-cut tobacco

3.13
test sample
fine-cut smoking articles for test taken at random from the laboratory fine-cut smoking articles and which are representative of the laboratory fine-cut smoking articles

3.14
test portion
group of fine-cut smoking articles made from the test sample(s), or a sample of fine-cut tobacco, prepared for a single determination and which is a random sample from the test sample or conditioned sample

3.15
conditioned sample
conditioned fine-cut smoking articles for preliminary tests and for smoking in particulate matter determinations

3.16
butt length
length of unburned fine-cut smoking article remaining at the moment when the smoking is stopped

3.17
insertion depth
length from the butt end to which a fine-cut smoking article is inserted into the holder

3.18
filling value
filling capacity
filling power
measure of the volume occupied by a given mass of fine-cut tobacco when a given pressure is applied

3.19
firmness
property of a tobacco rod measured through its deformation when subjected to a given load

3.20
monitor test piece
cigarette taken from a batch produced under specially strict and controlled manufacturing conditions

NOTE The cigarettes of such a batch show the greatest possible homogeneity with regard to their physical, chemical and smoke yield characteristics.

4 Principle

The fine-cut tobacco is sampled and conditioned prior to article manufacture. Using a laboratory making device, four types of fine-cut smoking articles (FCSAs) are made, using two designs each with two different wrapper types. The FCSAs are conditioned, then smoked by a routine analytical smoking machine, with simultaneous collection of the total particulate matter in a glass fibre filter trap. If considered necessary, the consistency of the laboratory smoking process and subsequent analytical procedures are 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.

NOTE In countries that are not in a position to use gas-chromatographic methods, reference should be made to ISO 3400 for the determination of total nicotine alkaloids, and the determination of water in smoke condensate should be performed by the method described in ISO 10362-2. In such cases, values obtained for nicotine and water in smoke condensate 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, complying with the general requirements of ISO 3308, except that the holder described in 4.8 of ISO 3308:2000 shall be replaced by a holder as described in 5.2.

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 and 5,2 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 for laboratory testing shall be constructed so that it first compresses the predetermined quantity of tobacco in a controlled manner and then fills a preformed wrapper tube with the compressed tobacco portion. The device shall be suitable for the dimensions of the length and diameter of the wrapper tube specified in 7.2.2 or 7.2.3.

NOTE Making devices, capable of producing FCSAs with an inner diameter of 5,2 mm and 7,2 mm, and with a length of 70 mm, are commercially available. They are also available for use with the preformed filtered or non-filtered tubes currently offered for sale.

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 filter disc holders may be affected by static electricity, necessitating the use of an antistatic device.

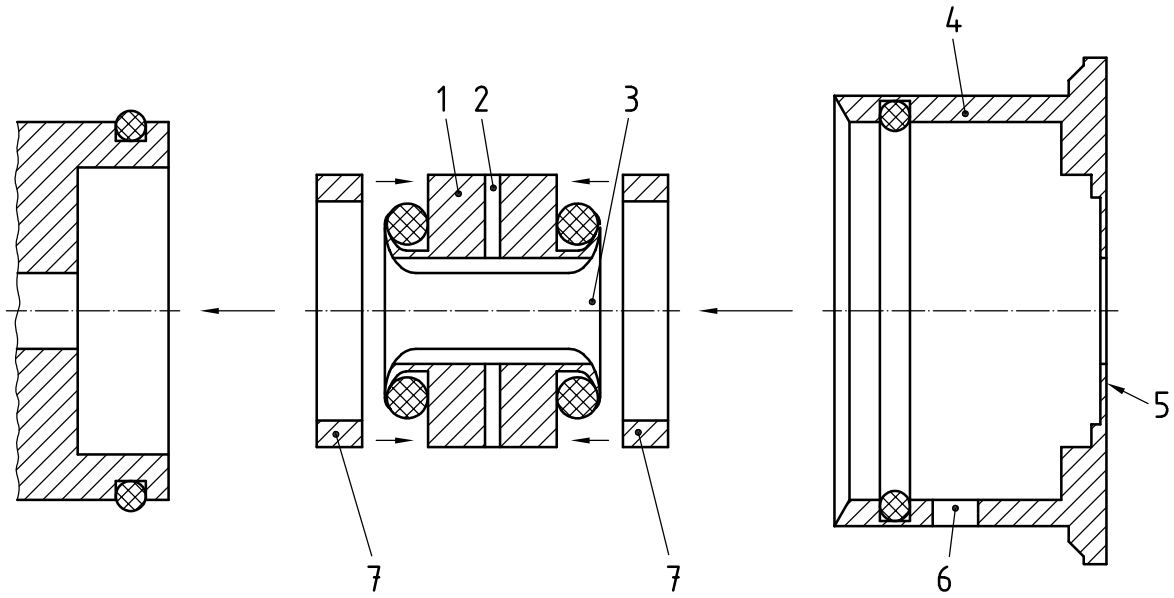
5.7 Conditioning enclosure, carefully maintained under the conditions specified in ISO 15592-2.

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 wrappers which have been slit and measured to the nearest 0,5 mm.

Dimensions in millimetres



a) Receiver assembly (adapter for rotary machine or trap front for linear machine)

b) Bobbin and sleeve assembly

c) Cap (with integrated positioning ring)

Key

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

	FCSA diameter	
	5,2	7,2
Bobbin		
Internal diameter	7,0	9,0
Length	12,2	12,2
Latex sleeve before insertion in holder		
Internal diameter	4,5	6,0
Length	19,0	19,0
Wall thickness	0,35	0,35

Figure 1 — FCSA holder

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

5.11 Gloves, cotton or non-talc surgical gloves.

6 Sampling

A laboratory sample (see 3.10) of fine-cut tobacco shall be provided by a sampling scheme such as one of those given in ISO 15592-1. This sample will normally contain tobacco taken randomly from the different parts of the population under test.

7 Wrappers used for making FCSAs

7.1 General

At the time of preparation of this part of ISO 15592, a great proportion of available wrappers fall into two main classes. These may be usefully designated as A and B. This part of ISO 15592 specifies a standard wrapper of each class for use in order to determine the NFDPM of a sample of fine-cut tobacco (see B.2).

7.2 Manufacture of standard tubes from wrappers

7.2.1 The laboratory device for making laboratory smoking articles (see 5.3) relies on the wrapper being supplied in the form of a tube. For this reason the wrappers specified in this clause may need to be made into tubes prior to use, but are available commercially. Experience has shown that it is necessary to use cylindrical formers of diameters 5,0 mm and 7,0 mm as appropriate.

7.2.2 Standard wrappers from each of the two classes shall be premade into tubes 70 mm long with an internal diameter of 5,2 mm. The tube shall be made from a wrapper not exceeding 20,0 mm in width.

7.2.3 Further standard wrappers from each of the two classes shall be premade into tubes 70 mm long with an internal diameter of 7,2 mm. The tube shall be made from a wrapper not exceeding 26,0 mm in width.

8 Making of the fine-cut laboratory smoking articles

8.1 General

Four types of FCSAs (see 3.12) shall be made from each laboratory sample (see 3.10) of fine-cut tobacco.

If the tobacco blend to be tested contains tobacco of high filling value, the masses of the tobacco shall be adjusted appropriately.

NOTE At the time of preparation of this part of ISO 15592 there is no internationally agreed definition of filling value or firmness of smoking articles (see 3.18 and 3.19). However, different types of apparatus for measuring each parameter are commercially available and generally used in the tobacco industry.

It is possible to allow for differences in filling value to control the firmness of the smoking articles made from the tobacco, for example if the volume occupied by a given mass of 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 tobacco is adjusted, this shall be mentioned in the test report.

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

8.2 Specification of the FCSAs to be made

The four types of laboratory FCSAs shall be made as follows.

a) Article 1:

400 mg of conditioned fine-cut tobacco shall be enclosed in a wrapper conforming to standard wrapper A (see B.2) and made into a tube specified in 7.2.2.