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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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*.

This second edition cancels and replaces the first edition (ISO 15592-3:2003), which has been technically revised.

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 and even by the same consumer throughout the day. A wide variation of wrappers is available throughout the world and in some countries pre-made filter tubes are sold in combination with the tobacco.

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. Fine-cut smoking articles made by consumers may therefore differ from the fine-cut smoking articles made for the purpose of testing described in this part of ISO 15592.

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, *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*

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 use*

3 Terms and definitions

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

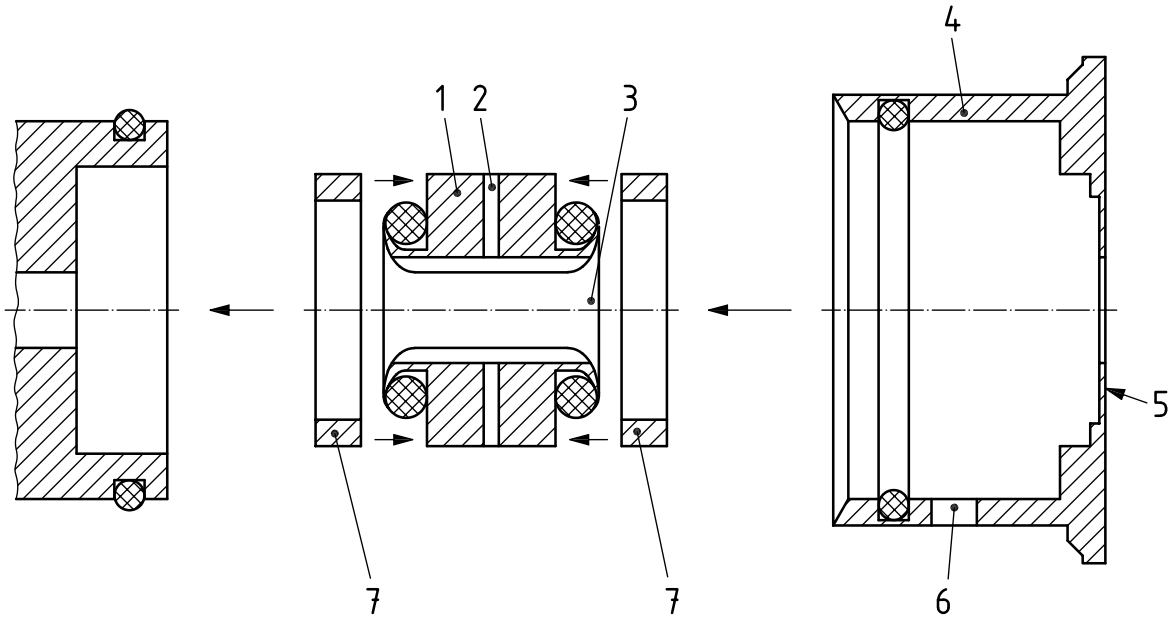
5.9 Apparatus for the determination of diameter, suitable for measuring to the nearest 0,2 mm.

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

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.

Dimensions in millimetres



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

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 fell 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 Clause B.2).

7.2 Manufacture of standard tubes from wrappers

7.2.1 The laboratory device for making laboratory smoking articles (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 prefabricated 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 prefabricated 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.

7.3 Determination of standard tube mass

Weigh 10 tubes made in accordance with 7.2.2 and 7.2.3 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 article.

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 was 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 Clause B.2) and made into a tube specified in 7.2.2.

b) Article 2:

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

c) Article 3:

750 mg of conditioned fine-cut tobacco shall be enclosed in a wrapper conforming to standard wrapper A (see Clause B.2) and made into a tube specified in 7.2.3.

d) Article 4:

750 mg of conditioned fine-cut tobacco shall be enclosed in a wrapper conforming to standard wrapper B (see Clause B.2) and made into a tube specified in 7.2.3.

8.3 Preparation and selection of the FCSAs for test

8.3.1 Use the making device (5.3) to prepare sufficient smoking articles required by the smoking plan and to allow for rejection due to damage or unacceptable quality. The end of the FCSA attached to the making device shall be identified for subsequent insertion in the article holder (see 9.3.3).

8.3.2 As the physical properties of FCSAs are very variable, 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 tobacco plus wrapper mass by more than 20 mg shall be rejected. The making may be performed by taking an individual weighed tobacco portion, making the article and then checking the weight, or a number of tobacco portions may be weighed in the shortest possible time, articles made and then selected by weight.

8.3.3 The mean mass of the 22 FCSAs after reconditioning shall be within $\pm$ 10 mg of the target mass of the tobacco plus wrapper mass (see 9.5).

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

8.4 Special precautions

The 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 tobacco moisture content may change during the making process and so precautions shall be taken to minimize this. Tobacco shall be kept as close 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 tobacco shall be removed from the conditioning chamber to make a limited number of articles such that the tobacco is likely to remain in condition during the making process;

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

NOTE It is not possible to specify the number of articles that can be made whilst keeping the tobacco in condition, as this will depend on the experience of the operator. It will also depend on the conditions under which the articles are made. As a guide, a period not exceeding one hour is recommended. The condition of the tobacco will in any case be determined prior to smoking as there is a requirement to check the weight specified in 9.5.

In addition, it is necessary to ensure that the FCSAs do not change in weight whilst in the smoking room. A smoke run using a linear smoking machine could take up to 2 h and during this time the weight of the FCSAs could change if they are kept in unsealed containers. It is necessary to ensure that just sufficient articles 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.

9 Determination of total particulate matter

9.1 Symbols

The following symbols are used in 9.2 and 9.6:

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 $\geqslant 1$, 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 smoking articles, see 3.12).

9.2 Preparation of the fine-cut smoking articles for smoking

9.2.1 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.

NOTE The multiplier C is usually at least 1,3 to provide extra FCSAs in case some are damaged and for any optional tests which may be required (see 9.5). If selection is necessary, C will have to be larger (perhaps 2,0 depending on the selection process).

The precision normally required generally demands that $100 \leqslant N \leqslant 200$. 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.

9.2.2 Selection of test portions from a bulk of Q FCSAs

For each group of 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.

9.2.3 Duplicate test portions

Provided that the sample of laboratory 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.

9.3 Marking the insertion depth and butt length

9.3.1 Standard insertion depth

The standard insertion depth for FCSAs made as in 8.3.1 shall be 13 mm ± 0,5 mm.

9.3.2 Standard butt length

The standard butt length for FCSAs made as in 8.3.1 shall be 27 mm.

9.3.3 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.

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 9.3.2 can be pre-set, it is not necessary to mark the butt lengths on the FCSAs themselves.

9.4 Conditioning and testing conditions

Condition all the article 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 samples or laboratory 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 article test portions to the smoking location in airtight containers just large enough to contain the portions (see 8.4).

9.5 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 mass of the wrapper and specified tobacco mass by no more than 10 mg.

IMPORTANT — It is important that the instructions given in 8.4 be 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) nominal 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 tobacco in the conditioned FCSA, determined in accordance with ISO 6488.

9.6 Smoking and collection of particulate matter

9.6.1 Smoking plan

Choose a smoking plan; examples of plans are given for information in Annex A of ISO 4387:2000.

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

9.6.2 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 filter discs 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 filter disc so that it will face the oncoming smoke. After assembly, examine the filter holders to ensure that the discs 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.

9.6.3 Setting up the smoking machine

9.6.3.1 General

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

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

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

9.6.3.2 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.

9.6.3.3 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.

9.6.3.4 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 also provides a check for leaks in the system. It shall be connected through a standard pressure drop device of 1 kPa $\pm$ 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.

The bubble flow meter shall contain a mass fraction of 15 % aqueous solution of a surface active agent.

NOTE Teepol L® [1)] has been found to be satisfactory.

1) Teepol L is the trade name of a product supplied by Shell. This information is given for the convenience of users of this part of ISO 15592 and does not constitute an endorsement by ISO of this product. Equivalent products may be used if they can be shown to lead to the same results.

Fit the prepared smoking trap or traps and FCSA holders on to the 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 for all remaining channels in turn.

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.

9.6.4 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 9.3.1). 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) tex 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.

Zero the puff counters 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 9.7.

9.7 Removal of smoke traps and FCSA holders

Remove the smoke traps from the smoking machine [gloves (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 (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 filter disc.

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

Glass fibre filter discs of 44 mm diameter are capable of retaining up to 150 mg of total particulate matter (TPM) and discs of 92 mm diameter are capable of retaining 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.

9.8 Calculation of total particulate matter

The TPM content, m_{TPM}, for each channel, expressed in milligrams per FCSA, is given by Equation (1):

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

where

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.

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

9.9.1 Extraction procedure

Remove the sealing devices from the smoke trap [gloves (5.11) shall be worn], open it and remove the filter disc with forceps. Fold it twice, total particulate matter inwards, being careful to handle only the edge with forceps and gloved fingers. Place the folded disc in an appropriately shaped dry flask (maximum 150 ml for 44 mm discs, maximum 250 ml for 92 mm discs). Wipe the inner surface of the filter holder front with two separate quarters of an unused conditioned filter disc 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 discs, or 50 ml for 92 mm discs). 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 disc 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.

9.9.2 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, m_{DPM}, for each trap, expressed in milligrams per FCSA, is given by Equation (2):

$$m_{DPM} = m_{TPM} - m_W \quad (2)$$

where

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

m_W is the water content in the TPM, in milligrams per FCSA.

9.9.3 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, m_{NFDPM}, for each trap, expressed in milligrams per FCSA, is given by Equation (3):

$$m_{\mathrm{NFDPM}} = m_{\mathrm{DPM}} - m_{\mathrm{N}} \quad (3)$$

where

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

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

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 part of ISO 15592, 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 manufacturer, country of manufacture;

b) brand name and sub-brand name;

c) pouch 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 FCSAs in the laboratory sample.

10.4 Description of test

The following particulars shall be included:

a) reference to this part of ISO 15592, i.e. ISO 15592-3:2008;

b) date of test;

c) type of smoking machine used;

d) type of smoke trap used;

e) total number of FCSAs smoked in the entire determination on that FCSA group;

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.

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 each of the varieties of four articles made from the fine-cut tobacco. If a specified wrapper has been tested, the data shall also be given for that wrapper.

a) average length of the FCSA wrappers, 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 0,1 mm;

d) average draw resistance of the conditioned FCSAs, in pascals to the nearest 10 Pa;

e) average mass, in milligrams per FCSA, of the conditioned fine-cut smoking articles selected for the smoking operation;

f) average mass, in milligrams per FCSA, of the wrapper;

g) water content, as a mass fraction in percent, of the tobacco in the conditioned FCSAs (see ISO 6488);

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

i) 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;

j) 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;

k) 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;

l) 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;

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

10.6 Expression of test results

The data obtained for NFDPM and nicotine from the four articles specified in 8.2 shall be reported in the form of a matrix that demonstrates the effects of fine-cut tobacco mass and wrapper specification. See Table 1 for an example.

Table 1 — Example of the data matrix

		Standard wrapper			
		A		B	
Tobacco mass	**Diameter**	**NFDPM** mg/FCSA	**Nicotine** mg/FCSA	**NFDPM** mg/FCSA	**Nicotine** mg/FCSA
400 mg	5,2 mm				
750 mg	7,2 mm				

11 Precision

An international collaborative study on the precision of the method was conducted in 2001. It involved 20 laboratories, 2 wrappers and 3 blends of tobacco. A monitor test piece was also analysed.

The blends were

a) a finely cut fire-cured tobacco blend used for rolling and mainly sold in the Netherlands;

b) a slightly more coarsely cut blend of roughly equal proportions of fire- and flue-cured tobaccos, mainly sold in Europe;

c) a blend of flue-cured tobacco with a small proportion of air-cured tobacco somewhat more coarsely cut than either blend 1 or 2, mainly sold in Scandinavian countries.

The monitor test piece and its use were as described in ISO 16055. It is a filter-tipped cigarette taken from a batch produced under specially strict and controlled manufacturing conditions. It is used to monitor the stability of the analytical processes when using a smoking machine.

The difference between two single results, found on matched blend 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 blend 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.

Data analysis gave the estimates as summarized in Tables 2 and 3 for NFDPM and nicotine respectively. For the purpose of calculating r and R, one test result was defined as the mean yield obtained from smoking 20 fine-cut smoking articles or 20 monitor test pieces in a single run.

Table 2 — Estimates given by data analysis for NFDPM

Blend	Tobacco mass mg	Value	NFDPM mg/FCSA made with wrapper A	NFDPM mg/FCSA made with wrapper B
Blend 1	400	Mean	9,4	12,1
		r	1,41	1,61
		R	2,69	3,74
	750	Mean	15,7	19,8
		r	1,64	1,72
		R	3,13	4,06
Blend 2	400	Mean	8,2	10,7
		r	1,02	1,31
		R	2,70	3,55
	750	Mean	14,6	18,4
		r	1,50	1,57
		R	3,07	5,46
Blend 3	400	Mean	7,7	10,1
		r	0,97	1,27
		R	2,32	3,06
	750	Mean	13,9	17,4
		r	1,53	1,76
		R	3,32	4,52
Monitor		Mean	15,0	
		r	0,94	
		R	1,98	

Table 3 — Estimates given by data analysis for nicotine

Blend	Tobacco mass mg	Value	Nicotine mg/FCSA made with wrapper A	Nicotine mg/FCSA made with wrapper B
Blend 1	400	Mean	1,00	1,25
		r	0,136	0,173
		R	0,349	0,453
	750	Mean	1,74	2,09
		r	0,220	0,239
		R	0,402	0,547
Blend 2	400	Mean	0,65	0,82
		r	0,083	0,108
		R	0,236	0,334
	750	Mean	1,14	1,39
		r	0,136	0,161
		R	0,286	0,432
Blend 3	400	Mean	0,51	0,65
		r	0,073	0,080
		R	0,192	0,228
	750	Mean	0,93	1,14
		r	0,103	0,110
		R	0,224	0,300
Monitor		Mean	1,21	
		r	0,087	
		R	0,216	

Annex A
(informative)

Summary of report of the CORESTA task force on roll-your-own (fine-cut) tobacco

This report provides details of the technical studies carried out by the task force established by CORESTA, to meet the following defined objectives (see reference [1]):

— to document how roll-your own (RYO) smokers in different countries make their cigarettes;

— to establish the effects of materials and cigarette design on particulate matter and nicotine yields of RYO cigarettes;

— to make recommendations to the Smoke Study Group on the relevance to the consumer of proposed standard procedures;

— to consider the establishment of a recommended method to give meaningful comparisons among fine-cut tobaccos or fine-cut smoking articles (FCSAs).

The above objectives are quoted from the original wording. The term “fine-cut smoking article” was adopted very early in the work of the task force as the term “cigarette” is used for a manufactured product.

The work carried out meets the first three of these objectives, and provides the basis for a standard method in line with the fourth. The following are the key outputs and conclusions of the work.

Consumer practices for making their smoking articles differ widely both between and within markets. There are wide variations in mass, diameter and shape, as well as a range of wrappers. The choices made by the consumer are crucial in determining smoke yields.

Current and proposed national procedures for measuring NFDPM and nicotine yields from fine-cut smoking articles (FCSAs) are inadequate, and do not provide consumers with sufficient information about the effect of the choices available when making their fine-cut tobacco products.

Consumers should be advised of the effect of their choices on smoke yields and a 2 by 2 matrix (reflecting the effects of tobacco quantity and wrapper used) provides the simplest approach to do so. A wrapper classification system is defined and recommended.

Making devices to provide satisfactory articles from fine-cut tobaccos and wrapper materials have been developed for both of the diameters required, to accommodate the two different tobacco masses, and have been evaluated successfully. Articles produced using these devices, following a defined protocol, seem to be sufficiently consistent for a standard method. However, the ends of the articles are neither as firm nor as cylindrical as those of manufactured cigarettes. This is considered to be a major cause of the high variability found in the results from smoking tests.

Smoking of laboratory-made articles results in considerable variation in smoke yields, even when the articles have been made under controlled conditions, and attention is required to reduce this in the smoking procedure adopted. Consumer-made articles are still more variable, and require additional care in smoking.

Practical difficulties were identified by the majority of the participating laboratories, and higher levels of variability were experienced when smoking FCSAs using the Labyrinth Seal holder specified in ISO 3308. An alternative holder has been developed that reduces this variability, especially when used with an increased insertion depth to allow for the irregular and under-filled end of the article.

Even with the improvements established by the work set out in this report, variability is inevitably greater for data from laboratory-produced FCSAs than that from manufactured cigarettes.

This report does not attempt to define what level of variability is necessary to provide a satisfactory standard for FCSAs that could be used for regulatory purposes. CORESTA considers that this requires the input of regulatory laboratories, and is willing to make its documented experimental information available to ISO to allow the method to be completed under its auspices.

CORESTA and its member companies will offer full cooperation in finalizing a standard, based on the technically sound principles arising from the practical studies detailed in this report. All of the data reported here are available from CORESTA for further analysis.

Annex B
(normative)

Wrapper specification

B.1 General

The determination of NFDPM of a sample of fine-cut tobacco by the method specified in this part of ISO 15592 requires the manufacture of FCSAs using two wrappers, designated A and B. This annex gives the technical specification of these wrappers.

B.2 Specification of standard wrappers

The specifications of the standard wrappers A and B are given in Table B.1.

Table B.1 — Wrapper specification

Parameter	Unit	Wrapper A			Wrapper B			Test method
Fibre furnish		100 % wood pulp			20 % wood pulp 80 % hemp/flax			ISO 9184 [2]
Colour		White			White			
Marking		Press			Press			
		Mean	**Mean tolerance**	**Individual tolerance**	**Mean**	**Mean tolerance**	**Individual tolerance**	
Substance	g/m^2	25	± 1,00	± 1,50	17,50	± 0,75	± 1,00	ISO 536 [3]
Thickness (10-fold)	µm	410	± 25,00	± 50,00	260	± 15,00	± 30,00	ISO 534 [4]
Filler $CaCO_3$	%	25	± 1,50	± 2,50	10,50	± 1,50	± 2,50	ISO 2144 [5]
Whiteness	%	90	± 3,00	± 4,00	83	± 3,00	± 4,00	ISO 2470 [6]
Opacity	%	73,5	± 3,00	± 4,00	55	± 3,00	± 4,00	ISO 2471 [7]
Air permeability (at a pressure of 1 kP) [a]	(cm^3/min/ cm^2)	50	± 4,00	CV max.[b] 10 %	15	± 5,00	CV max. 15 %	ISO 2965 [10]
Flow/pressure relationship		Linear			Linear			ISO 2965 [10]
Additives: Tripotassium citrate	%	0,60	± 0,10	± 0,15	None			[8]
Sodium acetate	%	0,60	± 0,10	± 0,15	None			[9]

NOTE Mean tolerances are those appropriate during the manufacture of wrapper paper. Individual tolerances are those appropriate for laboratory verification of the properties of individual wrapper products (NB: The individual thickness tolerance is for a 10-fold thickness).

[a] Both papers are natural porous papers.

[b] CV is the coefficient of variation.

Annex C
(normative)

Classification of wrappers

C.1 Introduction

This part of ISO 15592 gives a method for the determination of nicotine-free, dry particulate matter and nicotine of a given sample of fine-cut tobacco. It uses two standard wrappers of specified composition, designated as A and B (see Annex B).

Although the specifications of the papers used for these wrappers are not directly taken from those used commercially, they are representative of the two types of paper used to produce the majority of the commercially available wrappers at the date of this part of ISO 15592. Furthermore, the B paper is representative of those with the lowest porosity and burn rate that are not prone to self-extinguish when made into FCSAs and smoked on a smoking machine.

When the consumer uses fine-cut tobacco, it is used in combination with a range of wrappers to produce fine-cut smoking articles. Wrappers are normally available as leaflets in flat or interleaved booklets, in the form of a roll, or as premade tubes, with and without filters.

This annex describes the procedures to be followed to test wrappers and provides a means by which manufacturers of wrappers may classify them.

It is difficult to classify some wrappers. Although those sold as rolls do not have a predetermined length, they should be classified as being used at a length of 70 mm. Wrappers that consistently cause FCSAs to self-extinguish when smoked on a smoking machine should be classified as Type B.

C.2 Principle

The fine-cut tobacco (FCT) is sampled and conditioned prior to making FCSAs. Samples are taken of the test wrappers or tubes. Using a laboratory-making device, two types of FCSAs are made (one using standard wrapper A and the other using the test sample). The TPM and NFDPM are determined in accordance with Clause 9. The test sample is classified.

C.3 Test procedure

C.3.1 General

All the FCSAs shall be made from samples of tobacco carefully selected from the same batch of FCT.

Condition the tobacco in accordance with ISO 15592-2 [temperature (22 ± 2) °C and relative humidity (75 ± 3) % for a minimum of 72 h and a maximum of 10 d].

C.3.2 Sampling of wrappers for test

Sampling of wrappers, or wrapper material, intended for test shall be representative and care should be taken to ensure that an appropriate sampling scheme is adopted. The sampling scheme to be used will vary depending on the nature of the wrapper and whether it is the source material or the finished wrapper.

For example, bobbins of wrapper in the form of paper are normally split through the diameter of the bobbin and random samples of the resultant strips are used for analysis. To make the sample even more representative, more than one bobbin is sampled from across the paper machine. Sampling of wrappers from the market is much more difficult and care shall be taken to obtain samples from a wide variety of outlets in order to achieve representativeness.

C.3.3 Making of FCSAs for test

C.3.3.1 The wrapper to be tested may be of a length equal to standard wrapper A, or it may be longer or shorter. It may also be of an equal width or it may be wider or narrower or may be a tube.

For all comparisons detailed below, make at least 130 FCSAs using standard wrapper A [see 8.2 c)] and 130 FCSAs using the wrapper under test. This is to provide 100 articles for smoking, sufficient articles for other laboratory tests that may be required, and for replacements in case of damage.

C.3.3.2 If the wrapper is in the form of a leaflet, it shall be preformed into a tube with an inside diameter of 7,2 mm. The length of the tube shall be the length of the wrapper in the leaflet as supplied. The mass of tobacco M_1 to be used shall be determined using Equation (C.1):

$$M_1 = \left(\frac{L_1}{70} \right) \times 750 \text{ mg} \qquad \text{(C.1)}$$

where L_1 is the length, in millimetres, of the leaflet to be tested.

C.3.3.3 If the wrapper is in the form of a tube with or without a filter, and the dimensions of the tobacco section of the tube are other than 7,2 mm diameter and 70 mm long, the mass of tobacco M_2 to be used shall be determined using Equation (C.2):

$$M_2 = \left[\frac{\left(L_2 \times {D_2}^2 \right)}{\left(70 \times 7{,}2^2 \right)} \right] \times 750 \text{ mg} \qquad \text{(C.2)}$$

where

L_2 is the length, in millimetres, of the tobacco section of the tube to be tested;

D_2 is the inner diameter, in millimetres, of the tube to be tested.

NOTE The diameter may be determined by measuring slit wrapper tubes using a scale graduated in millimetres.

C.3.3.4 If the wrapper is supplied in the form of a roll, it shall be preformed into a tube 70 mm long with an inside diameter of 7,2 mm (see 7.2.3). FCSAs shall be made using 750 mg of conditioned tobacco in each tube.

C.4 Determination of FCSA smoke yields

For articles made with the standard wrapper A and those made in accordance with C.3.3.2, C.3.3.3 without a filter, and C.3.3.4, in accordance with 9.2, smoke N samples of each, where $100 \leqslant \ \leqslant 200$ in order to determine TPM, water and nicotine contents and to calculate NFDPM. For articles made in accordance with C.3.3.3 with a filter, smoke N of the test FCSAs in accordance with this part of ISO 15592, except that the holder shall be that specified in ISO 3308 and the butt length shall be as specified in ISO 4387. The holder specified in ISO 3308 determines the article insertion depth so that marking is not necessary.

C.5 Test results

In addition to the data specified in 10.5, the following data shall be reported for each type of wrapper:

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

b) if the wrapper is in the form of a tube, the average diameter of the tube, in millimetres to the nearest 0,1 mm.

C.6 Expression of results

Let the mean yield of NFDPM and/or nicotine of the groups of standard articles (wrapper A) be Y mg per FCSA and that of the test articles be Z mg per FCSA.

If $Z \leqslant 1{,}15\ Y$, then the test wrapper shall be classified as A.

If $Z > 1{,}15\ Y$, then the test wrapper shall be classified as B.

If $Z < 0{,}85\ Y$, the percentage reduction in yield, P, shall be expressed according to Equation (C.3):

$$P = [(Y - Z)/Y] \times 100 \qquad \text{(C.3)}$$

In view of the need for the reduction to be greater than 15 %, a value of P less than 20 % is not meaningful. For this reason, the results shall be expressed to the nearest 10 % when P is 20 % or more.

When P is 20 % or more, the results may be accompanied by the statement:

"This product gives P % lower yields of NFDPM and/or nicotine than standard wrapper A."

Annex D
(normative)

Fine-cut tobacco stated to be used with specified wrappers

D.1 Introduction

Fine-cut tobacco is sometimes sold with a statement that it may be used with a specified wrapper that may be in the form of a leaflet or tube with or without an attached filter. When present, a filter allows the use of the holder described in ISO 3308 when determining smoke yields.

D.2 Principle

The fine-cut tobacco (FCT) is sampled and conditioned prior to article manufacture. Using a laboratory-making device, FCSAs are made of two masses of tobacco with the specified wrappers. The TPM, water and nicotine contents are determined and the NFDPM is calculated.

D.3 Manufacture of FCSAs for test

D.3.1 General

All the FCSAs shall be made from samples of tobacco carefully selected from the same batch of FCT.

Condition the tobacco according to ISO 15592-2 [temperature (22 ± 2) °C and relative humidity (75 ± 3) % for a minimum of 72 h and a maximum of 10 d].

Sample the wrappers in accordance with C.3.2.

D.3.2 Specification of the FCSAs to be made

At least 130 FCSAs of each type shall be made from the FCT and the specified wrappers using an appropriate making device (5.3).

If the fine-cut tobacco blend to be tested contains tobacco of high filling value, the masses of tobacco used shall be adjusted appropriately (see 8.1).

D.3.3 Specified tubular wrappers

D.3.3.1 Density of tobacco

Studies have shown that fine-cut smoking articles made using tubular wrappers with an attached filter had a tobacco bulk density ranging from 220 mg/cm^3 to 300 mg/cm^3. Tests are therefore made on articles made at each of these densities when the wrapper is tubular.

a) Type 1

The mass of tobacco M_1 to be used shall be determined using Equation (D.1):

$$M_1 = \frac{220 \times L_1 \times \pi \times {D_1}^2}{4\,000} \text{ mg} \quad \text{(D.1)}$$

where

L_1 is the length, in millimetres, of the tobacco section;

D_1 is the inner diameter, in millimetres, of the tube to be tested.

b) Type 2

In this FCSA, the mass of tobacco M_2 to be used is given by Equation (D.2):

$$M_2 = \frac{300 \times L_2 \times \pi \times {D_2}^2}{4\,000} \text{ mg} \quad \text{(D.2)}$$

where

L_2 is the length, in millimetres, of the tobacco section;

D_2 is its diameter, in millimetres.

D.3.4 Specified leaflets

If the specified wrapper is in the form of a leaflet, it is appropriate to test it in accordance with this part of ISO 15592 using the two masses specified.

a) Type 1

A leaflet shall be preformed into a tube with an inside diameter of 5,2 mm. The length of the tube shall be the length of the wrapper. The mass M_3 of tobacco to be used shall be determined using Equation (D.3):

$$M_3 = \left(\frac{L_3}{70}\right) \times 400 \text{ mg} \quad \text{(D.3)}$$

where L_3 is the length, in millimetres, of the leaflet to be tested.

b) Type 2

A leaflet shall be preformed into a tube with an inside diameter of 7,2 mm. The length of the tube shall be the length of the wrapper. The mass M_4 of tobacco to be used shall be determined using Equation (D.4):

$$M_4 = \left(\frac{L_4}{70}\right) \times 750 \text{ mg} \quad \text{(D.4)}$$

where L_4 is the length, in millimetres, of the leaflet to be tested.

D.4 Determination of FCSA smoke yields

The FCSAs shall be smoked in accordance with Clause 9, so that it is necessary to choose a smoking plan in order to determine the number of each of the two types of article to be made (see 9.2 and 9.6.1).

Smoke the two types of article in accordance with Clause 9 except that, in the case of filter-tubed wrappers, the holder to be used on the smoking machines is that specified in ISO 3308. The holder described in ISO 3308 determines the article insertion depth so that marking is not necessary. Use the standard butt length of the tipped FCSAs according to ISO 4387.

D.5 Test results

In addition to the data specified in 10.5, the following data shall be obtained for each type of article and reported:

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

b) average length of tobacco portion, in millimetres to the nearest 0,1 mm;

c) average length of the filter, in millimetres to the nearest 0,1 mm (if applicable);

d) average length of the overwrap, in millimetres to the nearest 0,1 mm (if applicable).

D.6 Expression of results

The results shall be expressed in one of the following forms additional to the requirements in 10.6.

a) Specified tubular wrappers

"When this tobacco is used in conjunction with the specified wrappers and smoked according to the standard procedure given in ISO 15992-3, the yields of NFDPM and/or nicotine are … mg and/or … mg [data from D.3.3.1 a)] and … mg and/or … mg [data from D.3.3.1 b)] for articles made from M_1 mg and M_2 mg of tobacco respectively."

If, based on the rounded values, the yields of NFDPM for specified filter tubes do not differ by more than 1 mg, then the expression shall give the mean value of the data from D.3.3.1 a) and D.3.3.1 b).

b) Specified leaflets

"When this tobacco is used in conjunction with the specified wrappers and smoked according to the standard procedure in ISO 15592-3, the yields of NFDPM/nicotine are … mg and/or … mg [data from D.3.4.a)] and … mg and/or … mg [data from D.3.4.b)] for articles made from M_3 mg and M_4 mg of tobacco respectively."

Annex E
(normative)

Effect of incorporation of loose filters

E.1 Introduction

The effect of filters on the yield of NFDPM and nicotine is dependent not only on the filter specification but also on other factors, such as tobacco type and smoke velocity. The influence of these factors, however, is small compared with the overall variability of the experimental method of measurement.

To a first approximation, a filter will cause a similar percentage change in smoke constituent yield regardless of tobacco type and wrapper specification. It is therefore only necessary to make a comparison between articles made with and without the filter.

E.2 Principle

Samples are taken of fine-cut tobacco (FCT) and filters. The FCT is conditioned prior to article manufacture. Using a laboratory-making device, two types of article are made using standard wrapper B (see Clause B.2). A reduced amount of tobacco is used in one type in order to accommodate a filter. The NFDPM and nicotine yields of the two types are compared by smoking in accordance with Clause 9.

E.3 Apparatus

Normal laboratory apparatus and, in particular, the following.

E.3.1 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 a wrapper tube of 70 mm length and of an appropriate diameter to incorporate the filter under test.

NOTE Devices capable of making 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.

E.4 Manufacture of FCSAs for test

E.4.1 General

All the FCSAs shall be made from samples of tobacco carefully selected from the same batch of FCT.

Condition the tobacco in accordance with ISO 15592-2 [temperature (22 ± 2) °C and relative humidity (75 ± 3) % for a minimum of 72 h and a maximum of 10 d].

E.4.2 Specification of the FCSAs to be made

At least 130 of each of two types of FCSAs shall be made from the FCT and standard wrapper B using an appropriate making device (E.3.1) for the diameter of the filter to be tested.

This part of ISO 15592 assumes that the filter is of diameter 7,2 mm. If the filter is of another diameter, the appropriate making device shall be used.

a) Type 1

The FCSA shall be 70 mm long, of 7,2 mm diameter and contain 750 mg of conditioned tobacco.

For other filter diameters, the mass of tobacco (M_1) is given by Equation (E.1):

$$M_1 = \left(\frac{D^2}{7{,}2^2} \right) \times 750 \text{ mg} \qquad \text{(E.1)}$$

where D is the diameter of the filter, in millimetres.

b) Type 2

The FCSA shall be 70 mm long, of 7,2 mm diameter and incorporate the filter under test. The mass of conditioned tobacco (M_2) shall be given by Equation (E.2):

$$M_2 = \left[\frac{(70 - F)}{70} \right] \times 750 \text{ mg} \qquad \text{(E.2)}$$

where F is the length of the filter, in millimetres.

For other filter diameters, the mass of tobacco (M_3) is given by Equation (E.3):

$$M_3 = \left[\frac{(70 - F)}{70} \right] \times \left(\frac{D^2}{7{,}2^2} \right) \times 750 \text{ mg} \qquad \text{(E.3)}$$

where D is defined in a) above.

Incorporation of the filter is difficult and great care is needed, when making this type of article, to avoid leaks appearing in subsequent operations. The filter is inserted in the making device before the tobacco.

E.5 Determination of FCSA smoke yields

The FCSAs shall be smoked in accordance with Clause 9, so that it is necessary to choose a smoking plan in order to determine the number of each of the two types of articles to be made (see 9.2 and 9.6.1).

Smoke the two types of articles in accordance with Clause 9.

E.6 Test results

In addition to the data specified in 10.5, the following data shall be obtained for each type of article:

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

b) average diameter of the filter, in millimetres to the nearest 0,1 mm;

c) average length of the tobacco portion, in millimetres to the nearest 0,1 mm.

E.7 Expression of results

Compare the yields, y_1 and y_2, of the two types of article as a percentage change Q:

$$Q = \left[\frac{(y_1 - y_2)}{y_1} \right] \times 100 \qquad \text{(E.4)}$$

where y_1 and y_2 are the smoking data (which may be either NFDPM or nicotine) obtained from FCSAs type 1 [E.4.2 a)] and type 2 [E.4.2 b)] respectively.

The change may not be significant (bearing in mind the repeatability and reproducibility of the FCSA smoking tests) unless the value is $>$ 20 %. See Clause 11. For this reason, the results should only be expressed to the nearest 10 % when Q is 20 % or more.

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