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

Part 1: Sampling

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

Partie 1: Échantillonnage



Reference number
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Contents

Page

Foreword.....	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and definitions.....	1
4 Sampling possibilities.....	4
5 Mode for sampling at one time	4
5.1 General.....	4
5.2 Procedure for sampling at the manufacturer's premises or at importer's or distributor's warehouse	4
5.3 Procedure for sampling at the point of sale.....	6
6 Procedure for sampling over a period of time.....	7
6.1 General.....	7
6.2 Procedure for sampling over a period of time at the manufacturer's premises or at importer's and distributor's warehouse.....	7
7 Constitution of the test sample	7
7.1 General requirements.....	7
7.2 Identification of the test samples.....	8
7.3 Initial selection	8
7.4 Division of the increments into portions	8
7.5 Provision of the test portion.....	9
7.6 Labelling	9
8 Statistical evaluation and reporting.....	9
8.1 Statistical evaluation	9
8.2 Outliers.....	9
9 Sampling report	9
Annex A (normative) Sampling for the determination of mean values of total and nicotine-free dry particulate matter	11
Annex B (normative) Sampling for determination of the values of the parameters of fine-cut tobacco	13
Annex C (informative) Background to the choice of sampling procedures.....	14
Bibliography	16

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 3.

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 part of ISO 15592 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 15592-1 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*
- *Part 4: Classification of wrappers*
- *Part 5: Fine-cut tobacco to be used with specified wrappers*
- *Part 6: Effect of incorporation of loose filters*

Annexes A and B form a normative part of this part of ISO 15592. Annex C is for information only.

Introduction

When this part of ISO 15592 was prepared there were no existing national standards, rules, regulations or laws which had to be taken into account. However, experience with manufactured cigarettes suggests that two different procedures are required, as follows:

- sampling at the point of sale;
- sampling at the producer's premises or importer's and distributor's warehouses.

The principle underlying all sampling procedures is to produce a sample representative of the whole. With manufactured cigarettes it is possible to sample from a number of sources and mix the sample before sub-sampling to produce a sample for testing. With fine-cut tobacco, this is not possible since mixing of the tobacco with very long, fine strands is ineffective and results in the degradation of the tobacco. Thus, if the analysis is to be performed on smoking articles made from tobacco, it is necessary to make smoking articles from all samples and then to mix the fine-cut smoking articles before sub-sampling. This may require sampling a large quantity of tobacco, and making a large number of smoking articles.

Sophisticated sampling plans are often too expensive to be used. The two procedures in this part of ISO 15592 are both simple and reliable.

Sampling is carried out either as a single procedure or as part of a series of samplings.

Sampling is carried out "at one point in time", for example tobacco available for distribution from a factory/warehouse or available at a retail outlet on the market on a particular day. When a sample is required which represents fine-cut tobacco available over an appreciable period of time (e.g. fine-cut tobacco representing several months' production) a number of sub-period samples will be taken at different times and the test results combined.

The sampling plan depends upon the purpose of sampling (e.g. determination of physical properties or of smoke constituents). Further background considerations on the choice of sampling procedures are given in an informative annex C. It concludes that determinations of smoke yield should be made on the population manufactured for sale, sampled at manufacturers' factories or importers' warehouses.

Annex A (normative) establishes procedures for sampling fine-cut tobacco which is intended to be made into fine-cut smoking articles for the determination of the mean values of total and nicotine-free dry particulate matter. Detailed sampling plans are given.

Annex B (normative) establishes methods for sampling fine-cut tobacco which are intended for the determination of the mean values of parameters of fine-cut tobacco itself or the determination of mean values of parameters of fine-cut smoking articles made from the fine-cut tobacco.

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

Part 1: Sampling

1 Scope

This part of ISO 15592 specifies two methods of sampling a population of fine-cut tobacco manufactured for sale for the preparation of smoking articles.

It provides information on the statistical treatment of data and provides guidance based on practical experience of the order of ranking when a product is sampled in accordance with the specified procedures, in particular, when smoking articles are made from the sampled fine-cut tobacco and smoked for the determination of nicotine-free dry particulate matter (NFDPM).

NOTE Suitable procedures for the determination of NFDPM will be described in ISO 15592-3.¹⁾

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this part of ISO 15592. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 15592 are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 5725-2, *Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method.*

3 Terms and definitions

For the purposes of this part of ISO 15592, 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

wrapper

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

1) In course of preparation.

3.3

fine-cut smoking article

FCSA

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

3.4

brand

manufacturer's term or name used to denominate a distinct blend of fine-cut tobacco that will be recognized by the consumer and which distinguishes it from other fine-cut tobacco

3.5

sub-brand

manufacturer's term or name used to denominate a distinct blend of fine-cut tobacco, retaining the original brand name, but with an additional description intended to denote a particular characteristic

EXAMPLES Bright, dark.

3.6

sale unit

quantity of fine-cut tobacco ready to be offered for sale to the public

NOTE 1 The commonly sold pouch of 50 g fine-cut tobacco is used as the basis of this part of ISO 15592 but fine-cut tobacco is also sold in other size pouches. The method of sampling different sizes is dealt with in the appropriate sections.

NOTE 2 Fine-cut smoking tobacco is also sold in packaging forms other than pouches but throughout this part of ISO 15592 the unit of sale is referred to as a pouch.

3.7

population

aggregate of sale units of the fine-cut tobacco to be sampled, intended for sale to consumers in a given geographical area over a given time period

NOTE The definition includes different sub-populations, two of which are given in 3.7.1 and 3.7.2.

3.7.1

population available to consumers

aggregate of sale units in retail outlets in a given geographical area, at any time in a given time period

[ISO 8243:1991, definition 3.2.1]

3.7.2

population manufactured for sale

aggregate of sale units at a manufacturer's premises available for commercial distribution in a given geographical area, at any time in a given time period

[ISO 8243:1991, definition 3.2.2]

3.8

strata

various lowest levels of the particular population of the samples

EXAMPLE Samples from different machines, packaging types, etc., which arrive at the sampling point.

3.9

increment

sample of fine-cut tobacco taken at one time, at one sampling point, to be combined to produce the gross sample

3.10

gross sample

aggregate of the increments

[ISO 8243:1991, definition 3.4]

3.11**sub-period sample**

that part of the whole sample taken in a brief period when sampling over a long period of time

[ISO 8243:1991, definition 3.5]

3.12**laboratory sample**

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

[ISO 8243:1991, definition 3.6]

3.13**test sample**

fine-cut tobacco for test taken at random from the laboratory sample, which is representative of each of the increments making up the laboratory sample

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**preliminary test sample**

fine-cut tobacco obtained for preliminary tests

3.16**laboratory smoking articles**

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

3.17**place of purchase**

town, village or district within the area to be sampled, or that part of the area where the fine-cut tobacco is available

NOTE Examples of boundaries are those of cantons, local government districts, electoral areas, postal code areas or any boundaries in accordance with the geographical context, or others.

3.18**sampling point**

specific location (e.g. shop, specialist tobacco shop, vending machine, place in warehouse, place in manufacturer's premises, etc.) from which an increment is to be taken

3.19**factory**

place of manufacture or its associated distribution depots or the warehouse of an importer

[ISO 8243:1991, definition 3.11]

3.20**bundle**

commercial package available within a manufacturer's premises (normally 10 pouches)

EXAMPLE Pouches of 50 g fine-cut tobacco are usually put into bundles of 500 g fine-cut tobacco.

NOTE This may also be referred to as the "retailer unit".

4 Sampling possibilities

This part of ISO 15592 specifies two different procedures for sampling a population of fine-cut tobacco, according to whether sampling is undertaken at the producer's premises or importer's and distributor's warehouses, or at the point of sale, as follows.

- a) Sampling "at one point in time" provides an instantaneous estimate of one or more characteristics of the fine-cut tobacco. Sampling is carried out within as short a period as possible, not exceeding 14 days.
- b) Sampling "over a period of time" provides a continuous estimate of one or more characteristics of the fine-cut tobacco. It can be considered for practical purposes as a series of samples each taken "at one point in time".

The various sampling possibilities are given in Table 1, together with the number of the subclause in which they are described.

Table 1 — Sampling possibilities

Sampling procedure	Sampling mode	
	At one time (instantaneous)	Over a period (continuous)
A At producer's premises or importer's and distributor's warehouse	5.2	6.2
B At point of sale	5.3	

5 Mode for sampling at one time

5.1 General

When a sale unit does not consist of a pouch of 50 g fine-cut tobacco, the number of sale units sampled should be adjusted to produce the required quantity of fine-cut tobacco.

Two alternative sampling procedures are described:

- in 5.2, a procedure for sampling at the premises of the manufacturer or importer's or distributor's warehouse, and
- in 5.3, a procedure for sampling at the point of sale.

5.2 Procedure for sampling at the manufacturer's premises or at importer's or distributor's warehouse

5.2.1 Principles

Sampling is, in general, carried out by an independent organization, which will send to the manufacturer an accredited person referred to below as "the sampler".

Sampling by an outside organization, which shall only be done with the manufacturer's consent, shall be done within given short time periods (days) when the sampler visits the manufacturer's premises.

If the manufacturer so requests, the sampler will take a replicate sample for the manufacturer's use (see 5.2.4).

Samples shall only be taken from the finished product which is packed and ready for commercial distribution. All factories, stock rooms and warehouses containing finished products shall be included in the population to be sampled.

The sampler shall bring written details of the purpose of the test, name of the fine-cut tobacco and number of sale units. Three copies shall be provided, one for the sampler's record, a second to be packed with the samples, and a third for the manufacturer, to act as a receipt for the goods taken.

A test sample consisting of 500 g fine-cut tobacco may be obtained for preliminary test purposes.

If the results of the preliminary test suggest the possibility of unusual variation, samples shall be taken from each part of the finished products as described in 5.2.

5.2.2 Sampling

For each increment required, draw one bundle (usually 500 g fine-cut tobacco) at random from the population to be sampled, i.e. at each sampling point selected in the manufacturer's premises.

If the population has several strata, e.g. pouches from different machine rooms or factories, or different packaging types, then the increments should be drawn from all the strata in proportion to their respective sizes.

If the sampler finds that the stock available is not adequate to take the number of increments required, he shall arrange a further visit to complete the sampling but samples from different populations shall be considered as different laboratory samples.

5.2.3 Constitution of the gross sample

The gross sample is the aggregate of the increments. However, for reasons of convenience and also representativeness, it is preferable to prepare the laboratory sample directly from the increment (3.9). This is particularly important in order to secure matched laboratory samples when several laboratories are to run tests.

5.2.4 Constitution of the laboratory sample

If fine-cut tobacco of the same name and characteristics is required for several tests, sufficient sale units shall be obtained from each sampling point. If several laboratories are to run tests, an equal number of sale units from each sampling point shall be contained in each laboratory sample.

Each laboratory sample shall be marked with at least the following information:

- a) name of the fine-cut tobacco and its characteristics;
- b) date of sampling;
- c) factory/warehouse at which the sale unit was taken;
- d) sampling point within the factory/warehouse;
- e) order number of sale unit of that day;
- f) destination (i.e. the laboratory to which the samples are to be sent);
- g) marks on stamp (if any);
- h) printed smoke yields (if any);
- i) manufacturer's pack codes (if any).

All the samples shall be packed securely with adequate protection against damage (mechanical damage, severe changes in humidity, temperature, etc.) and shall be sent to each laboratory by the most expeditious means.

A list of the samples dispatched shall be sent on the day of dispatch, under separate cover, to each laboratory.

5.3 Procedure for sampling at the point of sale

5.3.1 Selection of the places of purchases

The required number of increments and the number of places of purchase to be used will depend on the purpose of the test and are given in annex A, clause A.2.

5.3.2 Selection of the sampling points

The increments obtained in each place of purchase shall originate from sampling points which are distributed over separate locations throughout the place of purchase.

The choice of sampling points shall, whenever possible, reflect the pattern of retail distribution of fine-cut tobacco in the sampling place to be sampled. This is usually done by defining, for each sampling scheme, several kinds of sampling points (e.g. automatic vending machines, supermarkets, specialist tobacco shops).

Each kind of sampling point is sampled at random throughout the place of purchase and, in total, the sample from each kind of sampling point shall make up a defined proportion of the whole sample. (This is called a quota from each kind of sampling point.)

Sampling shall only be carried out at another kind of sampling point after two unsuccessful attempts have been made at sampling points of the specified kind.

5.3.3 Constitution of the gross sample

The gross sample is the aggregate of the increments. However, for reasons of convenience and representativeness, it is preferable to prepare the laboratory sample directly from the increment (3.9). This is particularly important in order to secure matched laboratory samples when several laboratories are to run tests.

5.3.4 Constitution of the laboratory sample

If fine-cut tobacco of the same name and characteristics is required for several tests, sufficient sale units shall be obtained from each sampling point. If several laboratories are to run tests, an equal number of sale units from each sampling point shall be contained in each laboratory sample.

A test sample consisting of 500 g fine-cut tobacco may be obtained for preliminary test purposes.

If the results of the preliminary test suggest the possibility of unusual variation, separate samples shall be taken from each kind of sampling point as described in 5.3.

Each laboratory sample shall be marked with at least the following information:

- a) name of the fine-cut tobacco and its characteristics;
- b) date of sampling;
- c) place of purchase;
- d) kind of sampling point (if defined);
- e) sampling point (address of retail outlet);
- f) destination (i.e. the laboratory to which the samples are to be sent);
- g) marks on stamp (if any);
- h) printed smoke yields (if any);
- i) manufacturer's pack codes (if any).

The fine-cut tobacco in the gross sample shall be obtained in as short a time as possible. This time should not exceed 14 days.

All the samples shall be packed securely with adequate protection against damage (mechanical damage, severe changes in humidity, temperature, etc.) and shall be sent to each laboratory by the most expeditious means.

A list of the samples dispatched shall be sent on the day of dispatch, under separate cover, to each laboratory.

6 Procedure for sampling over a period of time

6.1 General

For some purposes a sample representing fine-cut tobacco available over a period of time (e.g. 4 to 6 months) is required and can be obtained by dividing the required sample into a number of sub-period samples which are obtained and tested at different times. It is important that each sub-period sample be tested during the next sub-period and not saved in order to test the whole sample at the end of the period. This avoids potential problems connected with ageing of the sample and ensures that variations over time in both the fine-cut tobacco and the laboratory determinations are taken into account in the measure of sample variability.

6.2 Procedure for sampling over a period of time at the manufacturer's premises or at importer's and distributor's warehouse

The time period shall be divided into at least four equal sub-periods, one sub-period sample taken in each sub-period from every manufacturer's premises (or importer's and distributor's warehouse) where the fine-cut tobacco is made (or imported and distributed). Whenever possible, the number of sub-periods multiplied by the number of sampling points should equal the number of increments required in the bulk sample. The total number shall be the same as that required for a sample at one point in time and they shall be equally divided between sub-periods.

At each manufacturer's premises, no more than one increment shall be drawn from a sampling point. Sampling points shall be selected from all the possible sample points in the manufacturer's premises.

Principles, sampling and constitution shall be as described in 5.2.

The sampling procedure is illustrated in Table 2.

7 Constitution of the test sample

7.1 General requirements

In general, the laboratory sample will contain fine-cut tobacco for a number of different kinds of test. Each may require a different size of test sample (e.g. condensate and nicotine can be determined as one test, after the fine-cut tobacco is made into laboratory smoking articles but determination of nicotine in tobacco is a separate test requiring a smaller test sample). The sample for each kind of test shall contain fine-cut tobacco from every increment of the sample, except in the case where the possibility envisaged in 7.2 is used. It shall never be smaller than the sale unit originally sampled.

For nearly all kinds of tests there will be several individual determinations (replicates, smoking channels) carried out at each laboratory. At some stage the test sample or the laboratory smoking articles made from it will be divided into test portions, one for each individual determination.

Each laboratory should arrange its work as described in 7.2 to 7.6.

Table 2 — Sampling at the manufacturer's premises over a period of time

Type of sampling	Manufacturer's premises 1	Manufacturer's premises 2
Strata	Machines A B C D	Machines E F
Sampling points	Warehouse	Warehouse
Increments (2 in this case)	6 bundles 1; 2; 3; 4; 5; 6;	2 bundles 7; 8;
Sub-period sample	8 bundles 1; 2; 3; 4; 5; 6; 7; 8;	
Sampling sub-periods	January February March April May	
Gross sample (theoretical in this case)	5 samples of 8 bundles each: total 40 bundles (5 × 2 increments: total 10 increments)	
Laboratory sub-period sample	Constitution of the laboratory samples: 2 pouches per bundle for laboratory A 2 pouches per bundle for manufacturer's laboratory Remainder for "reserve" Size: 16 pouches per laboratory sample	
NOTE Machines B and D are twice as fast as the other machines and hence the sampling rate is twice as high.		

7.2 Identification of the test samples

The increments intended to form the laboratory sample are first individually identified. They are then inspected and, if careful visual examination shows that samples are not of the same increment of fine-cut tobacco, they are separated so that separate tests can be carried out on each of them.

7.3 Initial selection

If the laboratory sample is constituted of K increments, and k individual determinations are to be carried out (i.e. k test portions are required), then the increments of any version for which $K < k$ are discarded.

7.4 Division of the increments into portions

If the laboratory sample still contains several versions with K_1, K_2 , etc. increments, divide the k test portions (which will be formed later) between the versions in the proportion K_1, K_2 , etc. Within each version, divide the increments into test portions of approximately equal size (e.g. for 5 determinations and 13 increments, 2 groups of 2 increments and 3 groups of 3 increments).

7.5 Provision of the test portion

Take an equal number of pouches of fine-cut tobacco from each increment in a group to provide a test portion on which one determination will be carried out. Alternatively, make fine-cut smoking articles from each increment and provide a test portion consisting of fine-cut smoking articles.

A different number of pouches of fine-cut tobacco may be taken from increments in another group if it contains more or fewer increments.

7.6 Labelling

Ensure that each test portion is labelled to show which increments are represented.

NOTE This information may be needed later for the statistical analysis. If the variability of the sample is required, see clause 8.

8 Statistical evaluation and reporting

8.1 Statistical evaluation

This part of ISO 15592 is concerned only with sampling and the report of the results from the laboratory or the sampling organization to the users.

This part of ISO 15592 does not consider problems of comparisons between laboratories or of predicting results of one laboratory from those at another laboratory. ISO 5725 (all parts) considers comparisons between laboratories.

ISO 5725-1 and ISO 5725-2 define various measures of reproducibility and repeatability, but these are concerned with variations between and within laboratories due to testing errors and techniques. They are not directly relevant to sampling variations.

The combined variations of tobacco products (see annex C) and the analytical procedures are important.

It is strongly recommended when interpreting the results to take into account the confidence interval of the mean value. The method described in ISO 2602 to calculate confidence intervals is not applicable because samples taken according to this part of ISO 15592 are not strictly random.

There are many reasons for sampling commercial fine-cut tobacco (e.g. to check that it complies with the specification marked on the pouch, to publish comparative tables, and to see whether the smoke yields of fine-cut smoking articles made from one population are higher or lower than another). The statistical evaluation of the results will, therefore, depend on the purposes of the sampling, and users must interpret results in the light of those reasons and prepare tables appropriate for their needs.

8.2 Outliers

In any body of experimental data there might be outliers in which something may have gone wrong to give a faulty result. The tests for outliers described in ISO 5725-2 shall be used and the recommended criteria for rejecting observations shall be followed.

9 Sampling report

The sampling report shall include the following particulars:

- a) dates between which sampling was carried out;
- b) areas from which samples were drawn (or the area served by the factories/warehouses sampled);

ISO 15592-1:2001(E)

- c) number of times sampling was carried out and the number of increments sampled;
- d) number of places sampled, principles of factory/warehouse sampling (detailed tables of number of increments from each factory/warehouse are not necessary);
- e) note on anomalies, missing or re-tested values, very variable fine-cut tobacco, etc.;
- f) intentional changes to the product, e.g. change in printed smoke yield (C.2);
- g) any details specified in annexes A and B.

Annex A (normative)

Sampling for the determination of mean values of total and nicotine-free dry particulate matter

A.1 Procedure for sampling at the manufacturer's premises or importer's and distributor's warehouse at one point in time

A.1.1 Sampling

To make up each increment required, draw one or more bundles of fine-cut tobacco at random from each sampling point to form the necessary gross sample.

Take the increments from as many sampling points as possible (at least 10), distributed between the factories where the fine-cut tobacco is made or imported, and distributed as far as possible in proportion to the production at these factories, provided that every manufacturer's premises are sampled.

If the population has several strata (e.g. pouches of different size or from different machine rooms), then the increments should be drawn from all strata in proportion to their respective sizes.

A.1.2 Constitution of the laboratory sample

From each increment, take portions for the test laboratory and manufacturer (if required) in equal proportions, keeping the remainder as a reserve sample. Label each portion. The laboratory sample for each test of a population shall comprise the greater of 40 sales units or 2 000 g fine-cut tobacco (comprising a minimum of 10 sales units), divided equally, or as nearly so as possible, among the increments.

A.2 Procedure for sampling at the point of sale at one point in time

A.2.1 Selection of the places of purchase

NOTE The criteria specified in A.2.1.1 to A.2.1.4 are shown in Table A.1.

A.2.1.1 If the area in which the fine-cut tobacco is sold encompasses more than 20 places of purchase, 2 increments each shall be obtained in 20 randomly selected places of purchase in the area in which the fine-cut tobacco is sold.

A.2.1.2 If the area in which the fine-cut tobacco is sold encompasses 11 to 20 places of purchase, 4 increments each shall be obtained in 10 randomly selected places of purchase in which the fine-cut tobacco is sold.

A.2.1.3 If the area in which the fine-cut tobacco is sold encompasses 6 to 10 places of purchase, 8 increments each shall be obtained in five randomly selected places of purchase in the area in which the fine-cut tobacco is sold.

A.2.1.4 If the area in which the fine-cut tobacco is sold encompasses one, two, three, four or five places of purchase, 40, 20, 14, 10 and 8 increments each shall be obtained in the one, two, three, four or five places of purchase in which the fine-cut tobacco is sold.

Table A.1 — Sampling at the point of sale at one point in time — Selection of the place of purchase

Number of places of potential purchase per area	Number of increments to be obtained	Number of places of purchase
More than 20	2	20 at random
11 to 20	4	10 at random
6 to 10	8	5 at random
5	8	5
4	10	4
3	14	3
2	20	2
1	40	1

A.2.1.5 An alternative sampling procedure to that given in A.2.1.1 to A.2.1.4 may be used. This is independent of the size of the sales area, and not at random, but is satisfactory provided that the sampling is carried out in at least six sampling points. If used, a total of at least 40 increments shall be obtained, which should, as far as possible, be evenly distributed among the sampling points.

A.2.1.6 Within each place of purchase, sampling points shall be selected in accordance with 5.3.2. Increments shall be marked in accordance with 5.3.4.

A.1.2.7 The volume of sampling shall be expressly stated in the report, giving the number of places of purchase.

A.2.2 Constitution of the laboratory sample

From each increment, take portions for the test laboratory and manufacturer (if required) in equal proportions, keeping the remainder as a reserve sample. Label each portion. The laboratory sample for each test of a population shall comprise the greater of 40 sales units or 2 000 g fine-cut tobacco (comprising a minimum of 10 sales units), divided equally, or as nearly as possible, among the increments.

A.3 Sampling over a period of time

A sample representing a period of time shall be obtained from the manufacturer’s premises by dividing the sample specified in A.2 into a number of sub-period samples taken at different times, as specified in clause 6.

A.4 Constitution of the laboratory fine-cut smoking articles

This depends on the analytical smoking procedure to be used. Some procedures involve smoking 20 fine-cut articles per trap, whereas others use only five fine-cut articles per trap. The test sample shall comprise sufficient fine-cut tobacco for an appropriately planned experiment to be made.

Annex B (normative)

Sampling for determination of the values of the parameters of fine-cut tobacco

B.1 General

The properties of fine-cut tobacco can be measured on any sample of a product. However, except for experiments specifically designed as journey or storage tests (e.g. to examine the protective properties of pouches or packaging), the data will only be meaningful when the properties are evaluated immediately after manufacture. For this reason this annex limits certain of the options generally available in this part of ISO 15592.

B.2 Procedure for sampling at the manufacturer's premises or at the importer's or distributor's warehouse at one point in time

To make up each increment required, draw one or more bundles of fine-cut tobacco at random from each sampling point to form the necessary gross sample. Take the increments from as many sampling points as possible (at least 10), distributed between the factories where the fine-cut tobacco is made or imported, as far as possible in proportion to the production at these factories, provided that every manufacturer's premises are sampled.

If the population has several strata (e.g. pouches of different sizes or from different machine rooms), then the increments should be drawn from all strata in proportion to their respective sizes.

B.3 Sampling over a period of time

A sample representing a period of time can be obtained from the manufacturer's premises by dividing the sample specified in B.2 into a number of sub-period samples taken at different times, as specified in clause 6.

B.4 Constitution of the laboratory and test samples

The size of the laboratory and test samples should depend on the following:

- a) the parameter to be evaluated;
- b) the number of independent tests required;
- c) the number of replicate results required for each parameter;
- d) the number of pouches of fine-cut tobacco required to produce each result in b).

NOTE Some tests are destructive, while others are not.

Annex C (informative)

Background to the choice of sampling procedures

C.1 General

It is particularly difficult to recommend a general method of sampling fine-cut tobacco. The objective of sampling is, clearly, to provide a representative sample, but the problem arises because the specific purpose for which tests are required affects the recommendations.

C.2 Variability

Variability arises from the methods used to test fine-cut tobacco (e.g. see ISO 15592-3²) but there are also appreciable contributions to the variability of the product as fine-cut tobacco manufacture continues over a period of time. These are reflected in sources of variability described below.

- a) **Short-term variability:** The moisture content of the tobacco varies around its target value. Porosity and other properties of the wrapper used to make fine-cut smoking articles display similar variability. Thus, the design characteristics of the fine-cut smoking article being made at any one time vary in a random fashion around their target values, and these variations give rise to corresponding variations in smoke yields.
- b) **Medium-term variability:** Superimposed on the sources of short-term variability are the sources of medium-term variability, such as batch-to-batch changes in materials, grade substitutions in the blend, wear of machinery, etc.
- c) **Long-term variability:** In the long term there are changes in the blend due to different crop years. Machinery replacement programmes and the upgrading of manufacturing processes can influence the product. Suppliers of non-tobacco materials may also change. All these sources of long-term variability are added to both the short-term and medium-term contributions.

These terms are described for practical convenience, but it should be remembered that these sources of variability operate as a continuum over time. Experience over numerous years has shown that when attempting to estimate a “true” overall mean (i.e. over all production runs) the contribution to the variability of medium-term effects is larger than that of short-term effects, with the influence of long-term effects being larger than either of these.

For samples taken according to A.1, the implications are that 95 % confidence limits (for the mean of the smoke yields of fine-cut smoking articles made from the fine-cut tobacco) calculated from the sample data reflect only short-term variability. Increasing the size of the sample taken at any one point in time can only reduce the effect of the short-term sources of variability on the precision of the mean of the sample. Thus, the mean of a sample taken at a single time point is of limited value in predicting the mean likely to be obtained from any later sample, no matter how big these samples might be.

For samples taken according to A.3, the implications are that the 95 % confidence limits (for the mean of the smoke yields of fine-cut smoking articles made from the fine-cut tobacco) calculated from the sample data reflect short-term and medium-term variability. In this case increasing the number of sub-period samples reduces the confidence limits. However, unless the sampling period is greatly extended, the calculated confidence limits will still not reflect the long-term variability. Experience of sampling at point of sale (A.2) has shown that the data are often of little value. The rotation of stocks in retail outlets is often very poor so that old pouches appear for sale on shelves and the conditions for storage are frequently far from ideal.

2) In course of preparation.

Determinations of the smoke yields of fine-cut smoking articles made from the fine-cut tobacco on a point of sale sample reflect the smoke yields from the product available to a buyer of that particular increment. However, a gross sample made up from point-of-sale increments may have a wider inherent variability arising from unspecified periods of manufacture, and may possibly include fine-cut tobacco manufactured before and after intentional design changes.

C.3 Recommendations

These factors lead to the strong recommendation that determinations be made on the population manufactured for sale, sampled at manufacturers' factories or importers' warehouses.

Bibliography

- [1] ISO 2602, *Statistical interpretation of test results — Estimation of the mean — Confidence interval.*
- [2] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions.*
- [3] ISO 8243:1991, *Cigarettes — Sampling.*
- [4] ISO 15592-3, *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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