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INTERNATIONAL STANDARD

ISO 8243

Fifth edition
2013-07-01

Cigarettes — Sampling

Cigarettes — Échantillonnage

Reference number
ISO 8243:2013(E)

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Foreword

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

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2. www.iso.org/directives

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The committee responsible for this document is ISO/TC 126, *Tobacco and tobacco products*.

This fifth edition cancels and replaces the fourth edition (ISO 8243:2006), which has been technically revised.

Introduction

It is difficult to recommend a detailed method of sampling cigarettes, suitable for every purpose. 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 recommendation.

Existing national standards, rules, regulations and laws were taken into account when preparing this International Standard and two different procedures, both of which are simple and reliable, are described:

— sampling at the point of sale;

— sampling at producer's premises or importers' and distributors' warehouses.

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

Since this International Standard was originally written in 1981, its role in providing a basis of sampling cigarettes for the verification of on-pack declarations of smoke constituent yields has become increasingly important. For this reason a guide to the statistical evaluation and reporting of results is included to clarify the statistical basis of the confidence intervals for nicotine-free dry particulate matter (NFDPM), nicotine and carbon monoxide (CO) that are listed in Table 3.

Sampling according to Clauses 4 and 5 of this International Standard provides a representative cigarette sample that might be used for other testing purposes.

The sources of variability arising in cigarette manufacture and in the determination of smoke constituent components are described in Annex B and in ISO/TR 22305. It is recommended that determinations of smoke constituent yields should be made on the population manufactured for sale, sampled at manufacturers' factories or importers' warehouses and that because of variations in cigarette manufacture the "sampling over a period of time" mode should be used wherever possible.

INTERNATIONAL STANDARD ISO 8243:2013(E)

Cigarettes — Sampling

1 Scope

This International Standard specifies two methods of providing representative samples of a population of cigarettes manufactured for sale. Different procedures are specified (see Table 1) according to whether sampling is undertaken at the point of sale or at a factory.

a) Sampling "at one point in time" provides for appraisal of the chosen properties of the cigarettes on that occasion. Sampling is carried out within as short a period as possible.

b) Sampling "over a period of time" provides for on-going appraisals. It can be considered for practical purposes as a series of samples each taken "at one point in time".

Table 1 — Sampling possibilities

Sampling procedures		Sampling mode	
		At one time (instantaneous)	**Over a period** (continuous)
A	At point of sale	Subclause 4.1	[a]
B	At a factory	Subclause 4.2	Clause 5

[a] Procedure A over a period of time is possible but is not specified in this International Standard.

This International Standard provides information on the statistical basis for the treatment of data and gives estimates, based on practical experience, of the typical confidence intervals for nicotine-free dry particulate matter (NFDPM), nicotine and CO yields which may be found when a product is sampled in accordance with this International Standard and smoked in accordance with the procedures specified in ISO 3308, ISO 3402, ISO 4387, ISO 8454, ISO 10315, ISO 10362-1 and ISO 10362-2.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

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*

ISO 8454, *Cigarettes — Determination of carbon monoxide in the vapour phase of cigarette smoke — NDIR method*

ISO 10315, *Cigarettes — Determination of nicotine in smoke condensates — Gas-chromatographic method*

3 Terms and definitions

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

3.1
factory
place of manufacture or its associated distribution depots or the warehouse of an importer

3.2
sale unit
quantity of cigarettes ready to be offered for sale to the public

Note 1 to entry: The commonly sold packet of 20 cigarettes is used as the basis of this International Standard, but cigarettes are also sold loose and in other size packets.

3.3
carton
commercial package available within a factory

EXAMPLE Packets of 20 cigarettes are usually put into cartons of 200 cigarettes.

3.4
place of purchase
town, village or district within the area to be sampled, or that part of the area where the cigarettes are available

Note 1 to entry: 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.

3.5
sampling point
specific location representing different kinds of sampling points (e.g. shop, specialist tobacco shop, auto-vending machine, supermarket, place in warehouse, place in factory) from which an increment is to be taken

3.6
population
aggregate of sale units to be sampled

Note 1 to entry: The definition includes different sub-populations, three of which are given in 3.6.1, 3.6.2 and 3.6.4.

3.6.1
population available to consumers
aggregate of sale units in retail outlets in a given area, at any time in a given time period

3.6.2
population manufactured for sale
aggregate of sale units at a factory

3.6.3
stratification
division of a population into mutually exclusive and exhaustive sub-populations (called strata), which are thought to be more homogeneous, with respect to the characteristics investigated, than the total population

3.6.4
stratified sampling
in a population that can be divided into different mutually exclusive and exhaustive sub-populations (called strata), sampling carried out in such a way that specified proportions of the sample are drawn from the different strata and each stratum is sampled with at least one sampling unit

3.7
increment
sample of cigarettes taken at one time, at one sampling point

3.8
sub-increment
individual groups of cigarettes making up an increment

3.9
sub-period sample
increment taken when sampling over a period of time

3.10
laboratory sample
sample intended for laboratory inspection or testing

3.11
test sample
cigarettes for test taken at random from the laboratory sample and which are representative of each of the increments making up the laboratory sample

3.12
test portion
group of cigarettes randomly selected from the test sample for a determination

3.13
lot
definite quantity of some product, material or service, collected together and submitted for examination

Note 1 to entry: An inspection lot may consist of several batches or parts of batches.

4 Sampling mode: At one time

4.1 Procedure for sampling at the point of sale

4.1.1 Selection of the number and choice of sampling points

The number of sale units to be taken and the number of places of purchase to be randomly sampled is determined by the size of the area in which the cigarettes are sold. Select the appropriate numbers according to Table 2.

Table 2 — Sampling requirements

Total number of sampling points	Number of sampling points to be randomly sampled	Number of sale units to be taken at each sampling point for each laboratory sample
> 20	20	2
> 10 ≤ 20	10	4
≥ 5 ≤ 10	5	8
4	4	10
3	3	14
2	2	20
1	1	40

If the sampling requirements in Table 2 cannot be fulfilled, an alternative may be used with a justification in the sampling report. This may be independent of the size of the sales area, and not at random, but is satisfactory provided that a representative sample is taken. When used, a total of at least 40 sale units, when possible, shall be obtained.

NOTE Table 2 is applicable to a sample of 800 cigarettes. The choice of 800 cigarettes per sample has been the result of balancing various factors which influence the homogeneity of samples and should be large enough to obtain reliable results. Even though there was no single statistical rationale at the time the standard was developed, such sample size has proven to be sufficiently large to adequately represent a production batch and to carry out various analytical tests from the same sample. If there is more than one testing laboratory then the number of sale units is increased appropriately. It is necessary to make sure that each laboratory sample is representative of the population, e.g. if more than one carton is sampled they should be subdivided between the laboratory samples.

When a sale unit does not consist of a packet of 20 cigarettes, adjust the number of sale units sampled to produce the required number of cigarettes. In case of more laboratories, special attention shall be given to the fact that each laboratory has a matched sample.

The sampling points from which the sale unit shall be obtained are to be distributed throughout the place of purchase.

The choice of sampling points shall, whenever possible, reflect the pattern of retail distribution of cigarettes in that sampling place to be sampled. This is usually done by defining several kinds of sampling points for each sampling scheme.

Each kind of sampling point is randomly sampled throughout the place of purchase and, in total, the increment shall make up a defined proportion of the whole sample.

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

4.1.2 Constitution of the laboratory sample

4.1.2.1 From each sale unit take cigarettes in equal proportions for the laboratory samples (see Table 2).

4.1.2.2 If cigarettes of the same name and characteristics are required for several individual determinations, 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.

4.1.2.3 Each laboratory sample shall be so labelled as to link it with all the manufacturing or packaging information that is relevant to the use of the data produced from tests upon it, such as:

a) name of the cigarettes and any other 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 destined);

g) marks on tax stamp, banderole (if any);

h) printed smoke yields (if any).

4.1.2.4 The sample(s) shall be obtained in as short a time as possible, not exceeding 14 d.

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

4.1.2.6 A list of samples in each despatch on that day shall be sent to each laboratory, under separate cover in a separate mailing.

4.1.3 Constitution of the test sample

4.1.3.1 There will be several determinations (e.g. in ISO 4387 replicate smoking runs and smoking channels) carried out at each laboratory and the test sample will be divided into test portions, one for each determination.

4.1.3.2 The sub-increments in the laboratory sample are first individually identified. They are then inspected and, if several versions are found (e.g. cigarettes or packets with visible differences), they are separated so that separate tests can be carried out on each of them.

4.1.3.3 From the laboratory sample cigarettes are taken at random from each sub-increment making up the laboratory sample in such a way as to ensure that the cigarettes are representative of each of the sub-increments.

4.1.3.4 An equal number of cigarettes from each sub-increment is taken to provide a test portion on which one determination will be carried out.

4.1.3.5 Each test portion is labelled to show which sub-increments are represented.

NOTE This information may be needed later for the statistical analysis. If the variability of the sample is required, see Clause 6.

4.1.3.6 Each laboratory shall arrange its work as described in 4.1.3.1 to 4.1.3.5.

4.2 Procedure for sampling at the factory

4.2.1 Principles

4.2.1.1 Sampling is carried out by a suitably trained person. If the manufacturer so requests, the sampler will take a replicate sample for the manufacturer's use (see 4.1.2.1).

4.2.1.2 Samples shall only be taken from the finished product within a given short-time period (days) and should be ready for commercial distribution. All factories, stock rooms and warehouses containing finished products shall be included in the population to be sampled.

4.2.1.3 An external sampler shall bring written details of the purpose of sampling. 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. Factory internal sampling shall be well documented and contain the name of the cigarette, the name of the sampler, sampling locations and date of sampling.

4.2.2 Sampling

4.2.2.1 To make up each increment required, draw one or more cartons or packets of cigarettes at random from each sampling point to form the necessary laboratory samples.

4.2.2.2 Take the increments from as many sampling points as possible distributed between the factories where the cigarettes are made or imported and distributed as far as possible in proportion to the production at these factories, provided that every factory is sampled so as to ensure a statistically satisfactory representation of the population. If the population has several strata (e.g. packets from different machine rooms or factories), then the increments should be drawn from all the strata, in proportion to their respective sizes.

4.2.2.3 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. Samples taken within 5 d shall be considered as one laboratory sample.

4.2.2.4 The procedure for sampling is illustrated in Figure A.1.

4.2.3 Constitution of the laboratory sample

The laboratory samples are prepared as in 4.1.2.

4.2.4 Constitution of the test sample

The test samples are prepared as in 4.1.3.

5 Sampling mode: Over a period of time

5.1 General

The procedures described in Clause 4 are concerned with sampling "at one point in time" [see a) in Clause 1].

For some purposes a sample representing cigarettes available over a period of time (e.g. six months or a year) is required and may be obtained by dividing the sample required into a number of sub-period samples that are obtained and tested at different times. It is important that each sub-period sample be tested at the time of collection 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 cigarettes and the laboratory determinations are taken into account in the measure of sample variability. There may be occasions when it is necessary to sample at a point of sale over a period of time. Since the reasons are difficult to define, this procedure is not specified in this International Standard.

5.2 Procedure for sampling over a period of time at the factory

The time period shall be divided into at least five equal sub-periods. In each sub-period, each sub-period sample shall be taken from every sub-period from every factory where the cigarettes are 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 laboratory 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 factory, 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 factory.

Principles, sampling and constitution shall be as described in 4.2.

The procedure of sampling is illustrated in Figure A.1.

6 Statistical evaluation and reporting

6.1 Statistical evaluation

There are, potentially, many applications that require sampling of cigarettes manufactured for sale, ranging from the determination of smoke constituent yields to measuring physical properties on the unsmoked product. There are also different purposes for testing such samples. For example, for quality monitoring or to provide statistics for checking that cigarettes comply with on-packet declarations, currently of NFDPM, nicotine and CO.

Despite this diversity of application and purpose, the principles of statistical evaluation are similar for most situations. Laboratory measurements made on the product samples are used to estimate a statistic (e.g. a mean value or difference), which then requires qualification in terms of its statistical variation, usually in the form of a confidence interval.

Although this International Standard is primarily concerned with product sampling, it is also necessary, when formulating an appropriate confidence interval, to incorporate components of statistical variation associated with the product measurement. This is reflected in the confidence intervals given in 6.3 for the purpose of checking that cigarettes comply with on-packet declarations of NFDPM, nicotine and CO.

6.2 Outliers

In any body of experimental data there might be outliers, observations 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 its recommended criteria for rejecting observations followed.

6.3 Confidence interval

There are two major sources of statistical variation: the laboratory measurement (analytical) and the product itself. Both the product and the measurements made on it fluctuate over time (both in the short and longer term, see Annex B), while analytical measurements also vary between laboratories, even for matched samples of cigarettes. Rounding of reported values provides an additional source of variability that needs to be taken into consideration.

The method described in ISO 2602 to calculate confidence intervals shall not be used because samples taken in accordance with this International Standard are not strictly random.

NOTE The objective of the confidence interval is to ensure that on average only one in every 20 determinations are likely to be outside of this interval purely by chance.

6.4 Applications to the verification of cigarettes yields

In the context of packet labelling, smoke constituent yields (currently NFDPM, nicotine and CO) are determined from laboratory tests carried out by the manufacturer on cigarettes sampled from production. Checks by a designated laboratory would occur later, after the manufacturer has determined and printed its values on the cigarette packet. It follows, therefore, that the statistic, Z, for which a confidence interval is to be obtained can be defined as:

$$Z = M_m - M_{dl}$$

where

M_m is the manufacturer mean;

M_{dl} is the designated laboratory mean obtained at a later date.

The statistical task is then to combine the relevant components of variance in such a way that a confidence interval describes the extent of the variability in the difference, Z, between the mean values obtained by two separate laboratories on different samples of cigarettes. This has been studied in some depth and ISO/TR 22305[10] sets out the basis for the confidence intervals, Z, given in Table 3 for NFDPM, nicotine and CO. The conclusions within the report are based on practical experience of verifying these measurements in a number of different marketplaces underpinned by a theoretical consideration of the sources of statistical variation. The intervals, Z, given in Table 3 are expressed as percentages of the on-packet declared values.

When samples are obtained at one point in time (i.e. as under 4.1 and 4.2), wider confidence intervals are required since certain longer term components of variance are not 'averaged out' to the same extent.

Table 3 — Confidence interval

Smoke constituent and ISO measurement method	Sampling	
	Over a period of time (Clause 5)	At one point in time (4.1 and 4.2)
NFDPM (ISO 4387 and ISO 10362-1)	± 15 %	± 20 %
Nicotine (ISO 10315)	± 15 %	± 20 %
Carbon monoxide (ISO 8454)	± 20 %	± 25 %
NOTE These confidence intervals will not be smaller than ± 1 mg for NFDPM, ± 1,5 mg for CO and ± 0,1 mg for nicotine.		

NOTE Although practical experience of confidence intervals at the time of the current revision encompasses only the smoke analytes NFDPM, nicotine and CO, confidence intervals for additional smoke constituents may be added to Table 3 derived in accordance with the principles set out in this International Standard as experience develops.

7 Sampling report

The sampling report shall include the following particulars:

a) dates between which sampling was carried out;

b) area from which samples were drawn (or the area served by the factories/warehouses sampled);

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) intentional changes to the product, e.g. change in printed smoke yield values;

f) reference to this International Standard, i.e. ISO 8243.

Annex A
(informative)

Flow chart illustration of the sampling in Clauses 4 and 5

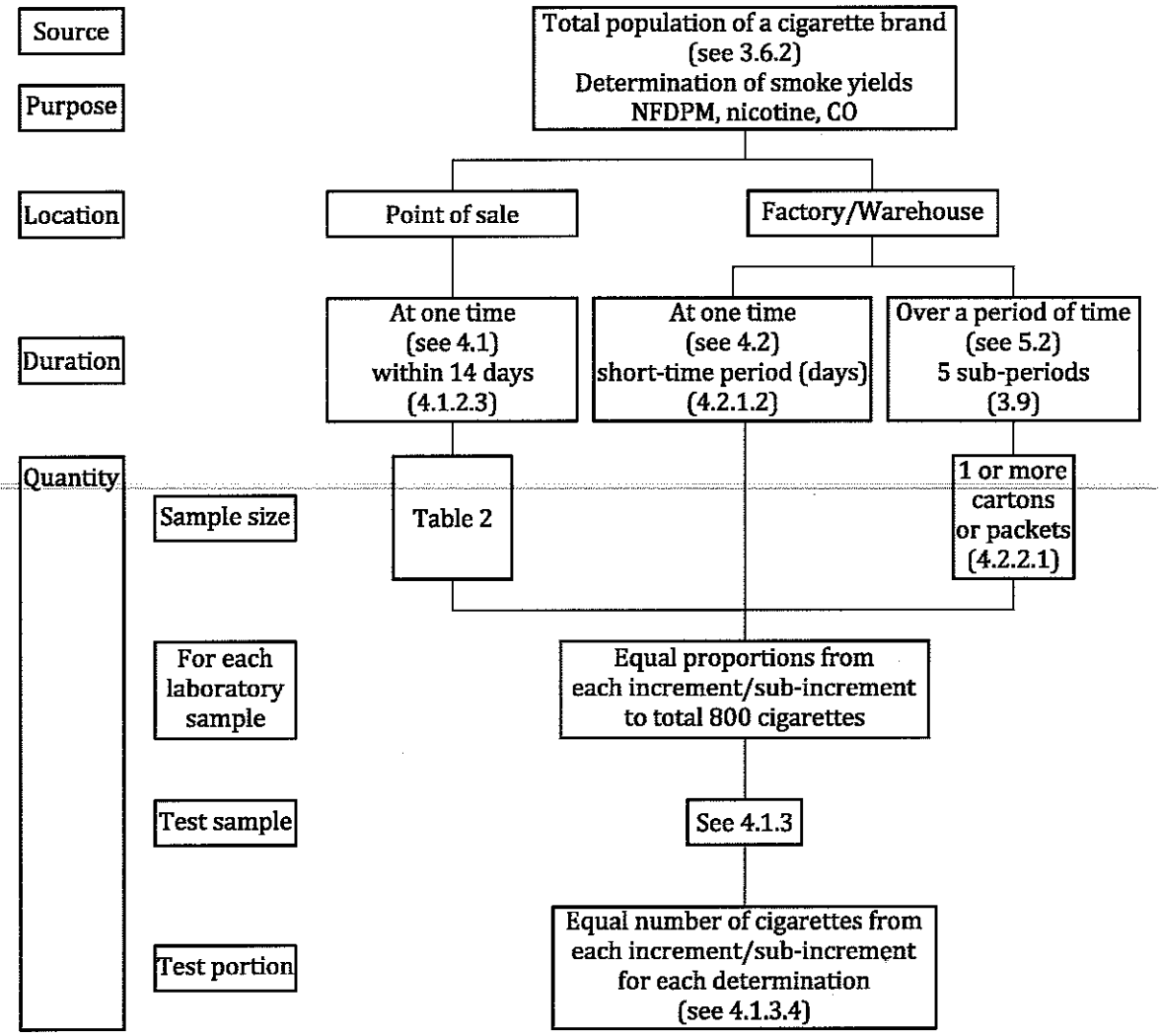

Figure A.1 — Flowchart for cigarette sampling

Annex B
(informative)

Sources of variability concerning the choice of sampling procedures

B.1 General

Variability arises from the methods used to test cigarettes (for example, see ISO/TR 22305), but there are also appreciable contributions to the variability of the product as cigarette manufacture continues over a period of time. These are reflected in sources of variability described in B.2 to B.4.

B.2 Short-term variability

It is impossible to precisely control the mass of every cigarette. The moisture content of the tobacco varies around its target value. Paper porosity contains similar variability. Tipping materials are also variable. Thus, the design characteristics of the cigarettes being manufactured 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.3 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 (paper, tipping, filter papers, filter tows), grade substitutions in the blend and wear of machinery.

B.4 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 (papers, tipping, etc.) may change. All these sources of long-term variability are added to both the short- and medium-term contributions.

B.5 Conclusion

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.

The implications of variability on confidence limits are discussed in Clause 6.

Bibliography

[1] ISO 2602, *Statistical interpretation of test results — Estimation of the mean — Confidence interval*

[2] ISO 3308, *Routine analytical cigarette-smoking machine — Definitions and standard conditions*

[3] ISO 3402, *Tobacco and tobacco products — Atmosphere for conditioning and testing*

[4] ISO 3534-1, *Statistics — Vocabulary and symbols — Part 1: General statistical terms and terms used in probability*

[5] ISO 3534-2:2006, *Statistics — Vocabulary and symbols — Part 2: Applied statistics*

[6] ISO 3534-3, *Statistics — Vocabulary and symbols — Part 3: Design of experiments*

[7] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[8] ISO 10362-1, *Cigarettes — Determination of water in smoke condensates — Part 1: Gas-chromatographic method*

[9] ISO 10362-2, *Cigarettes — Determination of water in smoke condensates — Part 2: Karl Fischer method*

[10] ISO/TR 22305, *Cigarettes — Measurement of nicotine-free dry particulate matter, nicotine, water and carbon monoxide in cigarette smoke — Analysis of data from collaborative studies reporting relationships between repeatability, reproducibility and tolerances*

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INTERNATIONAL STANDARD

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Routine analytical cigarette-smoking machine — Definitions and standard conditions

Machine à fumer analytique de routine pour cigarettes — Définitions et conditions normalisées

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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 3308 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

This fifth edition cancels and replaces the fourth edition (ISO 3308:2000), which has been technically revised. Subclause 5.8, the last formula in Annex C and the figures have been editorially revised. It also incorporates the amendment ISO 3308:2000/Amd.1 2009.

Introduction

Experience and knowledge gained from the use of analytical cigarette-smoking machines has highlighted a need to specify certain requirements, which are addressed in this International Standard.

No machine smoking regime can represent all human smoking behaviour:

— it is recommended that cigarettes also be tested under conditions of a different intensity of machine smoking than those specified in this International Standard;

— machine smoking testing is useful to characterize cigarette emissions for design and regulatory purposes, but communication of machine measurements to smokers can result in misunderstandings about differences in exposure and risk across brands;

— smoke emission data from machine measurements may be used as inputs for product hazard assessment, but they are not intended to be nor are they valid as measures of human exposure or risks. Communicating differences between products in machine measurements as differences in exposure or risk is a misuse of testing using ISO standards.

INTERNATIONAL STANDARD ISO 3308:2012(E)

Routine analytical cigarette-smoking machine — Definitions and standard conditions

1 Scope

This International Standard:

— defines smoking parameters and specifies the standard conditions to be provided for the routine analytical machine smoking of cigarettes;

— specifies the requirements for a routine analytical smoking machine complying with the standard conditions.

Annex A specifies the ambient air velocities surrounding cigarettes in a routine analytical smoking machine, the mechanical design of the enclosures immediately surrounding them, and the methods of air velocity measurement including the location where air velocity is measured.

Annex B describes, as an example, the special characteristics of a typical smoking machine incorporating a piston type of puffing mechanism.

Annex C includes a diagram of a puff profile and illustrates definitions and standard conditions.

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 3402, *Tobacco and tobacco products — Atmosphere for conditioning and testing*

3 Terms and definitions

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

3.1
test atmosphere
atmosphere to which a sample or test piece is exposed throughout the test

NOTE 1 It is characterized by specified values for one or more of the following parameters: temperature, relative humidity and pressure, which are kept within the specified tolerances.

NOTE 2 The test may be carried out either in the laboratory or in a special chamber termed the "test chamber", or in the conditioning chamber, the choice depending on the nature of the test piece and on the test itself. For example, close control of the test atmosphere may not be necessary if the change in properties of the test piece is insignificant over the test period.

NOTE 3 Adapted from ISO 558:1980, definition 2.3.

3.2
butt length
length of unburnt cigarette remaining at the moment when the smoking is stopped

3.3
restricted smoking
condition that exists when the butt end of a cigarette is closed to the atmosphere between successive puffs

3.4
free smoking
condition that exists when the butt end of a cigarette is completely exposed to the atmosphere between successive puffs

3.5
pressure drop
static pressure difference between the two ends of

— the test piece completely encapsulated in a measuring device such that no air can pass through the outer membrane (or wrapping); or

— a pneumatic circuit when it is traversed by an air flow under steady conditions in which the measured volumetric flow, under standard conditions, at the output end is 17,5 ml/s

3.6
draw resistance
negative pressure which has to be applied to the butt end, under test conditions (see ISO 3402) in order to sustain a volumetric flow of 17,5 ml/s, exiting at the butt end, when the cigarette is encapsulated in a measurement device to a depth of 9 mm

NOTE 1 Any ventilation zones and the tobacco rod are exposed to the atmosphere.

NOTE 2 Measurement values are expressed in pascals (Pa). They used to be expressed in millimetres water gauge (mm WG). The values given previously in mm WG are converted into pascals using the following conversion factor: 1 mm WG = 9,8067 Pa.

NOTE 3 The concept of draw resistance may also be subjectively judged when a cigarette is smoked by a consumer/taste panel. Under such circumstances, draw resistance is not measured objectively because the conditions of the formal definition are not met.

3.7
puff duration
interval of time during which the port is connected to the suction mechanism

3.8
puff volume
volume leaving the butt end of a cigarette and passing through the smoke trap

3.9
puff number
number of puffs necessary to smoke a cigarette to a specified butt length

3.10
puff frequency
number of puffs in a given time

3.11
puff termination
termination of the connection of the port to the suction mechanism

3.12
puff profile
flow rate measured directly behind the butt end of a cigarette and depicted graphically as a function of time

3.13
dead volume
volume which exists between the butt end of a cigarette and the suction mechanism

3.14
cigarette holder
device for holding the mouth end of a cigarette during smoking

3.15
smoke trap
device for collecting such part of the smoke from a sample of cigarettes as is necessary for the determination of specified smoke components

3.16
port
aperture of the suction mechanism through which a puff is drawn and to which is attached a smoke trap

3.17
channel
element of a smoking machine consisting of one or more cigarette holders, one trap and a means of drawing a puff through the trap

3.18
compensation
ability to maintain constant puff volumes and puff profiles when the pressure drop at the port changes

3.19
cigarette position
position of a cigarette on the smoking machine

NOTE In particular, it is determined by the angle made by the longitudinal axis of the cigarette and the horizontal plane when a cigarette is inserted into a cigarette holder in an analytical smoking machine.

3.20
mainstream smoke
all smoke which leaves the butt end of a cigarette during the smoking process

3.21
sidestream smoke
all smoke which leaves a cigarette during the smoking process other than from the butt end

3.22
ashtray
device positioned under the cigarettes in their holders to collect ash falling from the cigarettes during smoking

3.23
clearing puff
any puff taken after the cigarette has been extinguished or removed from the cigarette holder

3.24
ambient air flow
air flow around the cigarettes during the smoking process

NOTE See Annex A.

4 Standard conditions

4.1 Machine pressure drop

The whole of the flow path between the butt end of the cigarette and the suction mechanism shall offer the least possible resistance, and its pressure drop (see 3.5) shall not exceed 300 Pa.

4.2 Puff duration

The standard puff duration (see 3.7) shall be (2,00 ± 0,02) s.

4.3 Puff volume

The standard puff volume (see 3.8) measured in series with a pressure drop device of 1 x (1 ± 5 %) kPa shall be (35,0 ± 0,3) ml. In one puff duration (see 3.7) not less than 95 % of the puff volume shall leave the butt end of the cigarette.

4.4 Puff frequency

The standard puff frequency (see 3.10) shall be one puff every (60 ± 0,5) s measured over 10 consecutive puffs.

4.5 Puff profile

The puff profile (see 3.12) shall be measured with an impedance of 1 x (1 ± 5 %) kPa as specified in 4.3. It shall be bell-shaped with a maximum between 0,8 s and 1,2 s from the start of the puff. The increasing and decreasing parts of the profile shall not have more than one point of inflection each. The maximum flow rate shall be between 25 ml/s and 30 ml/s (see Annex B). At no point shall the direction of flow be reversed.

NOTE Principles of suction mechanisms using a piston pump to obtain the puff profile are given in Annex B.

4.6 Restricted smoking

An analytical smoking machine shall be a restricted smoker [i.e. fulfil the conditions for restricted smoking (see 3.3)].

4.7 Puff number

Each individual puff shall be counted and recorded and the puff number (see 3.9) rounded off to the nearest one-tenth of a puff, based on the puff duration.

4.8 Cigarette holder

The design of the standard cigarette holder (see 3.14) is such that it shall cover 9,0 mm, with a range of 8,0 mm to 9,5 mm, from the butt end of a cigarette, and shall be impermeable to smoke components and to air. Labyrinth seals with dimensions appropriate for the diameter of the cigarettes under test shall be used in the cigarette holder.

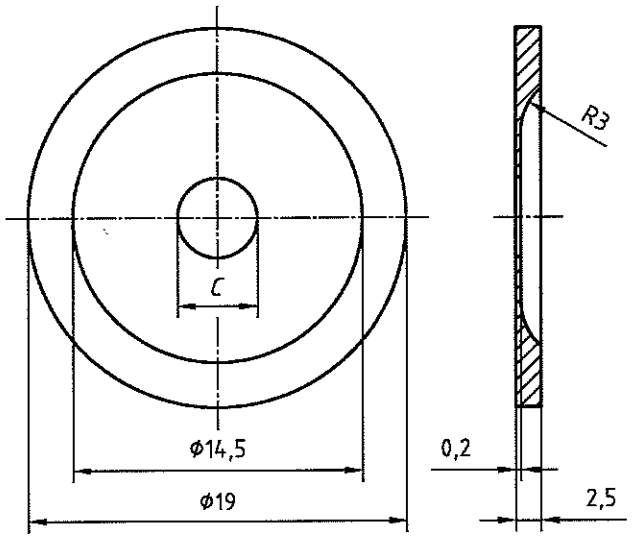
Either the cigarette holder or the smoke trap shall be equipped with a perforated disc (washer) of plain expanded synthetic rubber, closed-cell sponge grade, which partly obstructs the butt end of the cigarette. The synthetic rubber shall have a density of 150 kg/m^3, low swell oil resistance and compression-deflection range of 35 kPa to 63 kPa. Four labyrinth seals shall be used; the one closest to the butt end (back seal) shall be reversed. The dimensions of the washer and labyrinth seals are given in Figure 1. The washer shall be supported by a structure with a hole in its centre of 4 mm diameter.

The axis of the holder shall be within 0° to + 5° of the horizontal and the holder shall ensure that the cigarette is held within ± 5° of the holder axis.

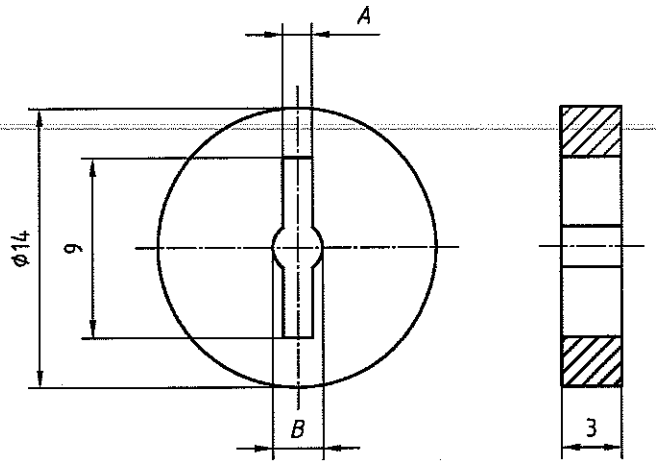
An example of a suitable assembly is given in Figure 2.

Manufacturing tolerances for the individual components of the cigarette holder result in an uneven tolerance about the specified 9 mm insertion depth.

Dimensions in millimetres



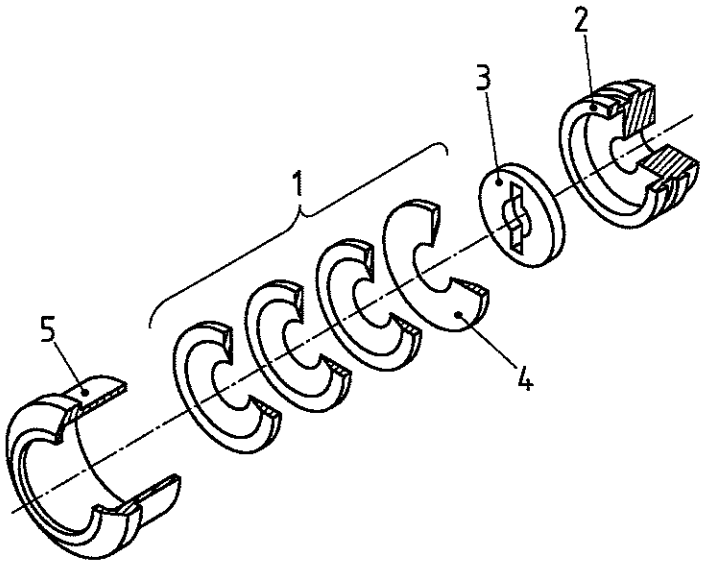
a) Labyrinth seal



b) Washer

Cigarette diameter	A	B	C
4,5 to 5,49	1,45	2,5	4
5,5 to 6,49	1,7	3	4,5
6,5 to 7,49	1,95	3,5	5,5
7,5 to 9	2,2	4	6,5

Figure 1 — Cigarette holder: Labyrinth seal and perforated disc (washer) (dimensional details)



Key

1 labyrinth seals
2 washer support
3 washer
4 labyrinth seal (reversed)
5 labyrinth cap

NOTE Washer support is for use where a central glass fibre smoke trap is used to trap smoke from more than one cigarette.

Figure 2 — Cigarette holder (schematic)

4.9 Cigarette position

The cigarette holders shall be arranged so that no cigarette influences the burning of any other cigarette.

The cigarette shall be positioned in the holder so that the butt end is in contact with the washer when inserted.

NOTE See 3.19.

4.10 Ashtray position

The ashtray shall be placed in a horizontal plane between 20 mm and 60 mm below the plane of the axes of the cigarettes.

NOTE See 3.22.

5 Specification for the routine analytical smoking machine

5.1 General

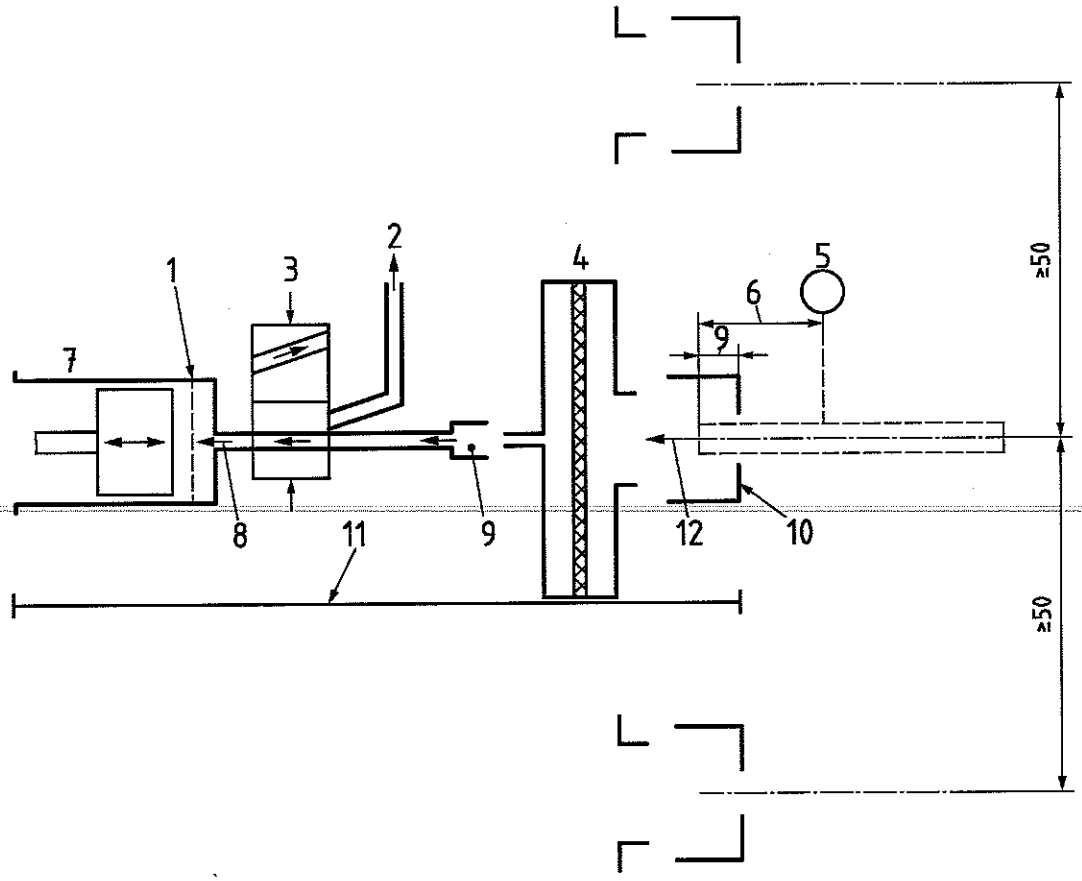
The smoking machine shall comply with the standard conditions (see 4.1 to 4.10) and the special conditions given in 5.2 to 5.8.

5.2 Operating principle and puff profile

5.2.1 The machine shall include a device to draw a fixed volume of air (puff) through a cigarette. A schematic diagram is shown in Figure 3.

5.2.2 The machine shall produce a bell-shaped puff profile (see 4.5).

Dimensions in millimetres



Key

1 piston top dead centre
2 gas phase out
3 control valve
4 smoke trap
5 puff termination device
6 butt length
7 piston pump
8 swept volume
9 port
10 cigarette holder
11 channel
12 puff volume

Figure 3 — Smoking machine schematics

5.2.3 The machine shall be a restricted smoker (i.e. fulfil the conditions for restricted smoking, see 3.3).

5.3 Reliability and compensation

5.3.1 The machine shall contain devices to control the puff volume, the puff duration, and the puff frequency.

5.3.2 The machine shall possess the mechanical and electrical reliability necessary to meet the standard conditions regarding these parameters (see 4.2 to 4.4) during the test for prolonged periods.

5.3.3 The machine shall be capable of sufficient compensation (see 3.18).

When the machine has initially been set to give a puff volume of 35 ml without a pressure drop device, a reduction of no more than 1,5 ml shall be observed when the machine is tested with a pressure drop device of 3 kPa.

5.3.4 The connecting piping between the smoke trap and the suction source shall offer the least possible resistance to flow. The pressure drop of the total flow path between the butt end of the cigarette and the suction source shall not exceed 300 Pa before smoking (see 4.1).

5.3.5 The total dead volume (see 3.13) shall be as small as possible and shall not exceed 100 ml.

5.4 Cigarette holders and smoke traps

5.4.1 The machine shall contain devices for holding the cigarette and for trapping the smoke produced.

5.4.2 The cigarette holders shall be capable of holding the butt end of the cigarette during smoking. Labyrinth seals shall be used for attaching cigarettes.

5.4.3 Devices shall be provided for attaching cigarette holders to the machine so that the cigarette holders are held rigidly.

A screwed fitting or "O" ring seal is recommended. Rubber tubing is considered to be unsatisfactory.

5.4.4 The cigarettes to be smoked shall be attached to the ports or the smoke traps by standard cigarette holders (see 4.8).

5.4.5 The machine shall be designed to hold the cigarettes in the standard position (see 4.9).

The system shall be designed to prevent losses of smoke components between the butt end of the cigarette and the smoke trap.

5.4.6 The cigarette holders shall be arranged so that the sidestream smoke does not affect cigarettes smoked in adjacent holders (see 4.9). The distance between the centres of adjacent burning zones shall be at least 50 mm.

5.4.7 When the smoking machine is used for collecting particulate matter, it shall be fitted with a glass fibre filter smoke trap, comprising the following.

a) Airtight filter holder and end caps made of a non-hygroscopic and chemically inert material, able to contain a filter disc of glass fibre material 1 mm to 2 mm thick. The rough filter surface shall face the oncoming smoke. Two examples are given in Figure 4.

   Different designs of smoke trap can meet this requirement. It is recommended that for smoking machines where 5 cigarettes are smoked per trap, the diameter of the glass fibre filter should be 44 mm. For machines where 20 cigarettes are smoked per trap, the diameter of the glass fibre filter should be 92 mm.

b) Filter material which shall retain at least 99,9 % of all particles having a diameter equal to or greater than 0,3 µm of a dioctyl phthalate aerosol at a linear air velocity of 140 mm/s. The pressure drop of the filter assembly shall not exceed 900 Pa at this air velocity. The content of binder shall not exceed 5 % as mass fraction. Polyacrylate and polyvinyl alcohol (PVA) have been found to be suitable binders for this material.

The filter assembly shall be capable of quantitatively retaining all of the particulate matter in the mainstream smoke produced by the cigarette without loss. In addition, the filter assembly shall be chosen so that the increase in pressure drop of the assembly does not exceed 250 Pa when measured after the smoking run.

5.4.8 Each channel shall have a puff-termination device linked to a butt length (mark) sensor and puff counter. When activated by the sensor, the device shall prevent any further drawing of air through the cigarette.

Examples of suitable sensors are as follows:

a) a micro-switch activated by the burning through of a 100 % cotton, (48 ± 4) tex thread, placed on the butt mark;

b) a specially shielded infrared detector. The shielding defines a detection border plane perpendicular to the cigarette. The crossing of that plane by the burning cone terminates the puff.

5.4.9 The machine shall be capable of smoking a wide range of cigarettes of different lengths, diameters and cross-sectional shapes while complying with the standard conditions regarding cigarette butt lengths.

5.4.10 The machine shall be capable of making one or more clearing puffs after the termination of smoking.

5.5 Test atmosphere

The test atmosphere shall be controlled to ensure that all the cigarettes are smoked under identical conditions with regard to ambient air flow.

The temperature and relative humidity of the test atmosphere shall correspond to those specified in ISO 3402:

— temperature (22 ± 2) °C;

— relative humidity (60 ± 5) %.

The design of the enclosure around the smoking machine and of the sidestream smoke extraction system should provide identical conditions with regard to air flow around the cigarettes for the different designs of smoking machine which conform to the specification in this International Standard (see Annex A).

5.6 Puff counting

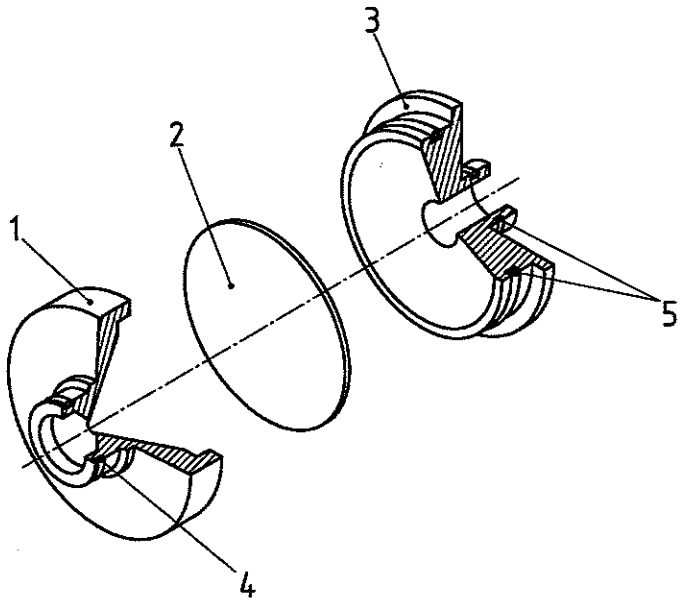
Each port shall have its own puff counter capable of counting to the nearest 0,1 puff (see 4.7).

5.7 Ignition

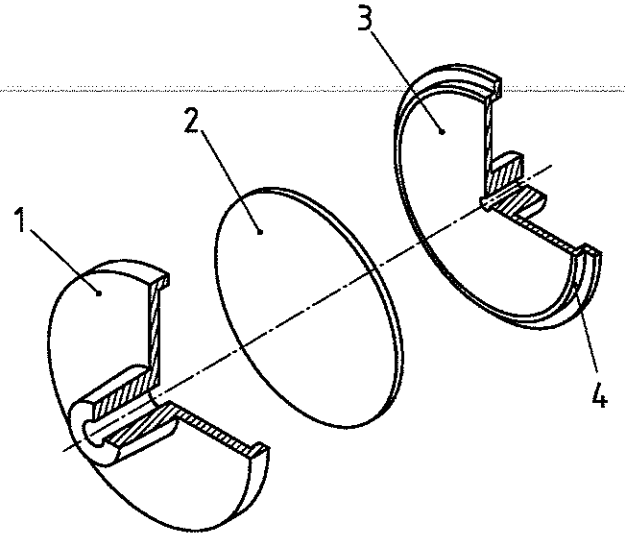
Flameless ignition shall be used. The lighters shall light the cigarettes at the first attempt without either touching or pre-charring the cigarette.

5.8 Smoking enclosure

The smoking process shall be carried out in an enclosure (see A.2), preferably transparent, which may be an integral part of the smoking machine, or a housing in which the machine can be sited. The enclosure shall be capable of being fitted with an air-extraction device to facilitate the controlled removal of sidestream smoke from the enclosure.



a)



b)

Key

1 GF holder front
2 filter disc
3 GF holder back
4 "O"-ring seal
5 "O"-ring seals

Figure 4 — Examples of glass fibre filter (GF) smoke traps (schematic)

Annex A
(normative)

Ambient air flow around cigarettes in routine analytical smoking machines: Control and monitoring

A.1 Scope

This annex specifies:

— the ambient air velocities surrounding cigarettes in an analytical smoking machine during the smoking process and the mechanical design of the enclosures immediately surrounding them; and

— the methods of air velocity measurement and the location where air velocity shall be measured.

NOTE The development of smoking machines has taken place since about 1960. However, because the mechanical configurations which can conform to this International Standard differ greatly, it has been found from the work of a special Task Force established by CORESTA that additional specification of the immediate smoking machine environment is necessary. This leads to better reproducibility in the international interlaboratory comparisons which are often required. It is doubtful if a general mechanical specification could be written to cover all types of smoking machines and so, as well as a general specification, it is necessary to provide examples for the designs most generally used.

There are two principal designs for smoking machines which satisfy the conditions specified in ISO 3308:

— type a) (see Figures A.1 and A.2) in which the position of the cigarette in its holder is fixed, i.e. adjustments are made by moving the puff termination device;

— type b) (see Figures A.3 to A.5) in which the position of the puff termination device is fixed, i.e. adjustments are made by moving the cigarette and its holder.

A.2 Examples of designs of smoking machine enclosures

A.2.1 Smoking machines, type a)

Figures A.1 and A.2 show schematic designs of the enclosure, including the features which shall be incorporated.

A.2.2 Smoking machines, type b)

Examples of this type of machine exist with 20 channels and 8 channels. Features common to both are shown in detail in Figure A.3. Figure A.4 shows a schematic design of the enclosure, including the features which shall be incorporated in 20-channel versions. Figure A.5 is appropriate to 8-channel versions.

A.3 Air velocity measurement locations

A.3.1 General

The reference points at which the measurement of air velocity has to be made shall be given. The required measurements shall be made such that the centre of the air velocity meter probe is within 2 mm in each plane of the specified position.

A.3.2 Smoking machines, type a)

The air velocity shall be measured, with the cigarette holders in place, at a point on the axis of the cigarette, 74 mm from the mouth end of the cigarette.

A.3.3 Smoking machines, type b)

A.3.3.1 The air velocity shall be measured with the cigarette holders in place at a point on the axis of the cigarette as held in its holder 40 mm towards the end of the cigarette which is to be lit measured from the position of the puff termination device.

NOTE Certain types of air velocity probe are mounted directly in a port, thereby replacing a filter and cigarette holder during measurement.

A.3.3.2 In order to check the uniformity of air flow across the smoking machine, measurements shall be made at a central port and at a port near each extreme. Additional measurements may be required upon installation or relocation of the machine.

A.4 Specification of the air velocity meter

An air velocity meter capable of an accuracy of not less than 20 mm/s at 200 mm/s air velocity shall be used. The air velocity measuring equipment shall be capable of integrating air velocity data over a minimum period of 10 s.

The value of a measurement of air velocity shall consist of the average of not less than 10 replications of 10 s integrations.

NOTE Advice on appropriate meters can be obtained from the suppliers of smoking machines.

A.5 Standard value of air velocity

The standard value of the air velocity shall be 200 mm/s.

Laboratory procedures should aim to ensure that the air velocity average during a smoking run lies in the range of 170 mm/s to 230 mm/s.

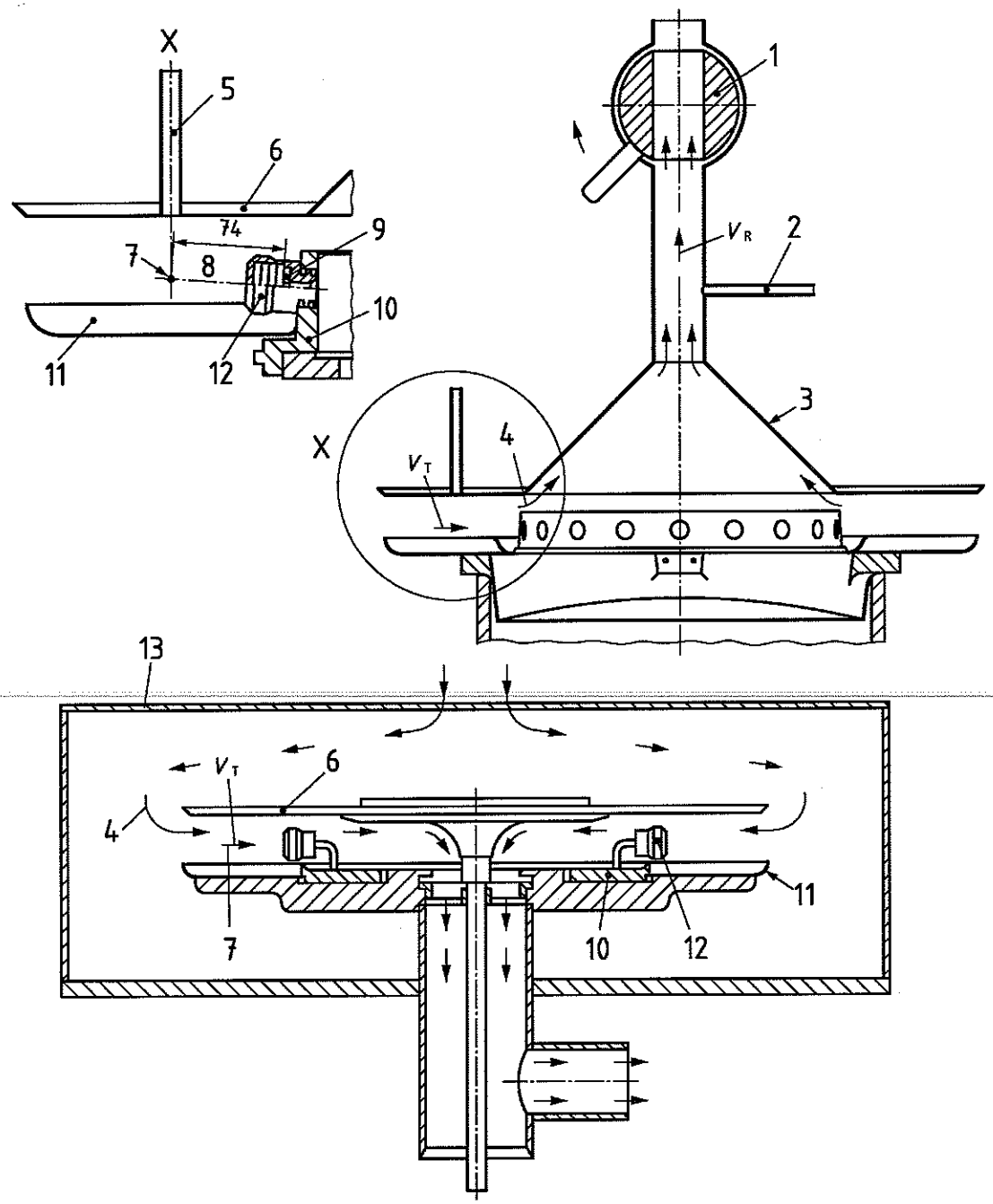
For smoking machines type b), the air velocity measured at an individual port should be within the range of 150 mm/s to 250 mm/s.

A.6 Setting and checking air velocity

Air velocity should be checked, and adjusted if necessary, when the machine is used.

Extreme atmospheric conditions, external to the test atmosphere, may affect air flow in smoking machine enclosures. In such circumstances, more frequent checks of air velocity should be made.

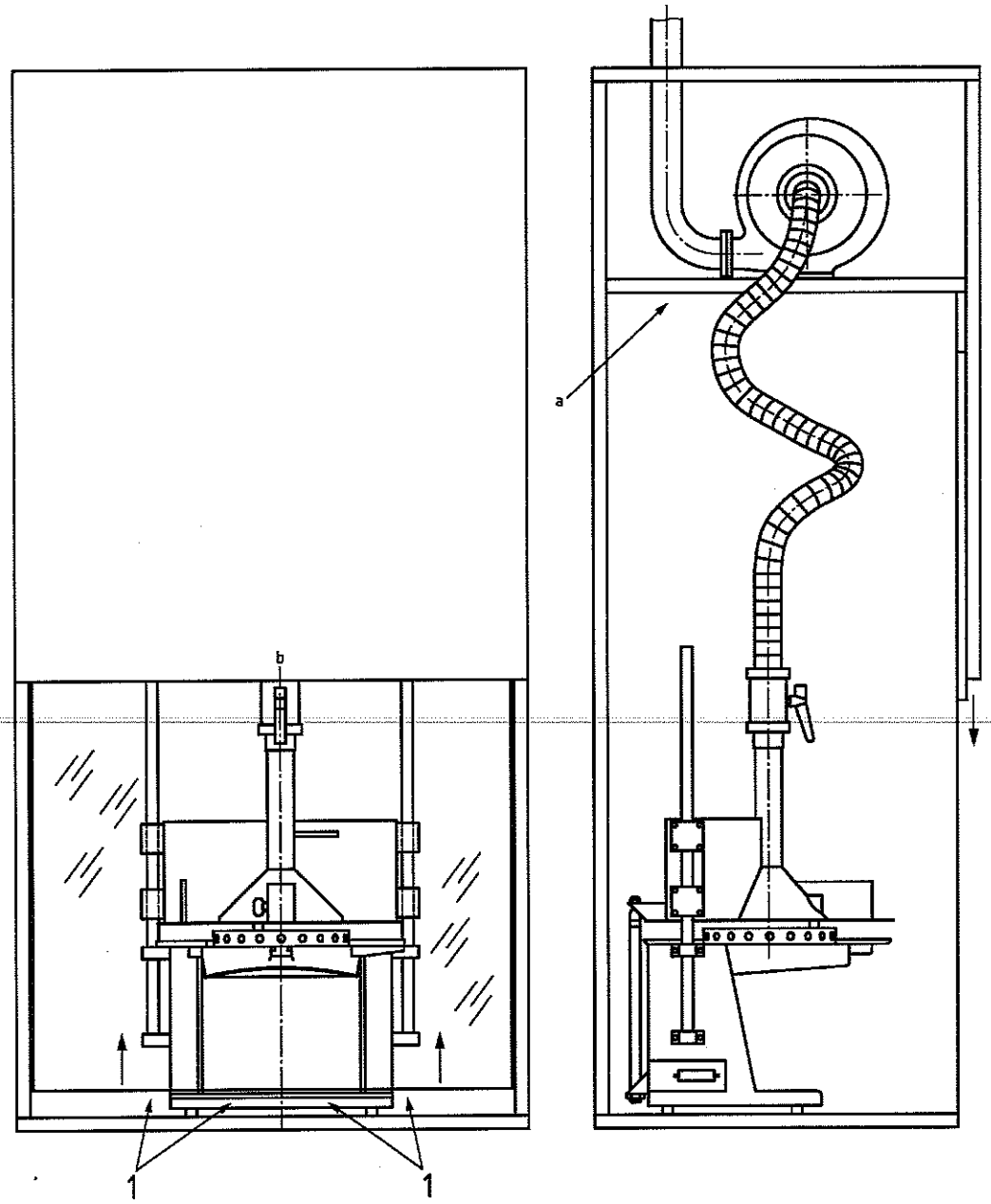
Dimensions in millimetres



Key

1	ball valve	8	cigarette centre
2	sensor guide pipe	9	polychloroprene washer
3	extraction hood	10	smoking ring
4	air flow (schematic)	11	ashtray
5	sensor guide pipe	12	cigarette holder
6	hood	13	enclosure
7	reference air velocity measurement position		
V_T	Air velocity around the cigarette	V_R	Air velocity in the extraction duct

Figure A.1 — Machines with fixed cigarette position: Rotary smoking machines type a)



Key

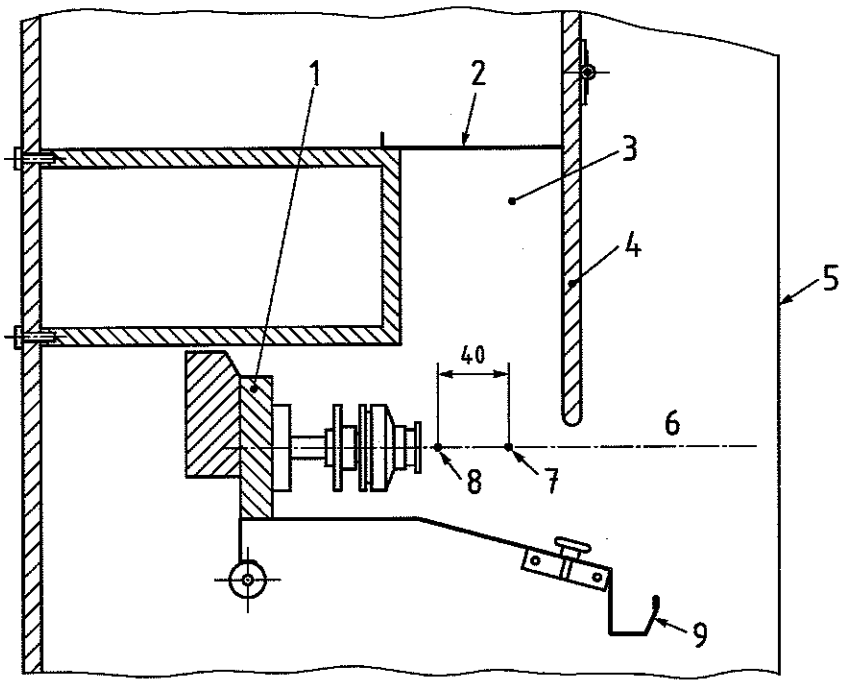
1 air

a The enclosure should not be open at the top.

b The front flap should be in the closed position during smoking.

Figure A.2 — Example of an enclosure for a rotary smoking machine type a) with hood

Dimensions in millimetres

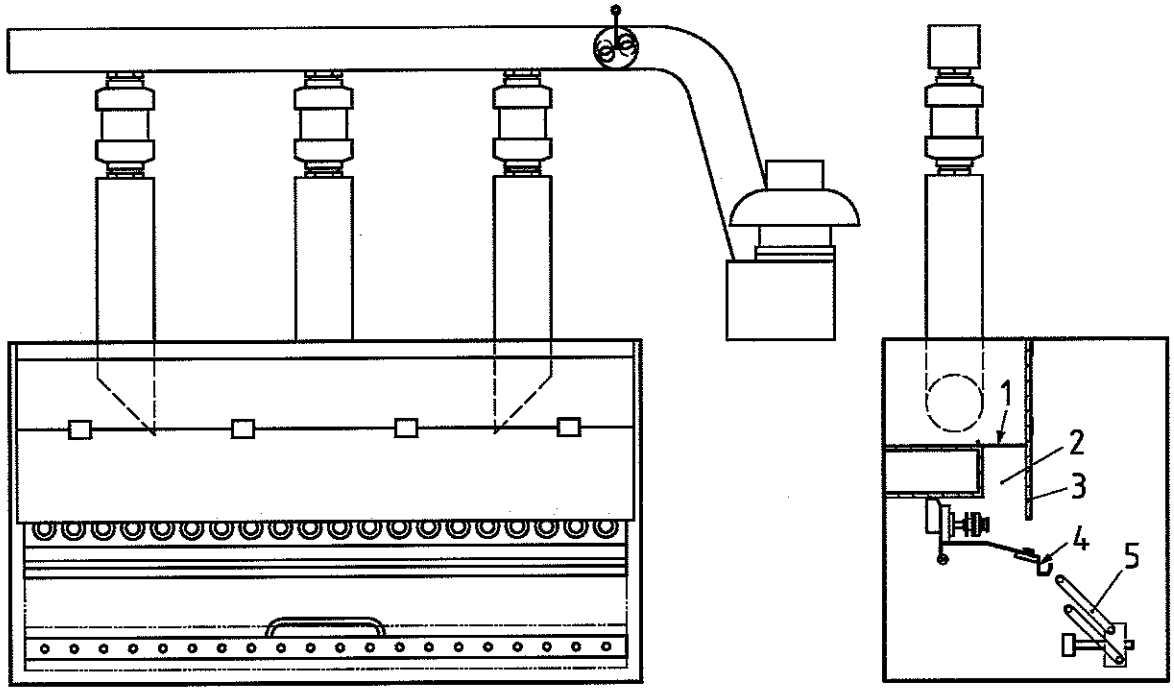


Key

1	smoking bar	6	nominal cigarette centre height
2	baffle plate	7	reference air velocity measurement position
3	internal duct	8	puff termination position
4	hinged front flap	9	ashtray
5	front of smoking machine		

NOTE This view shows the relationship between the puff termination position, smoking bar and ashtray.

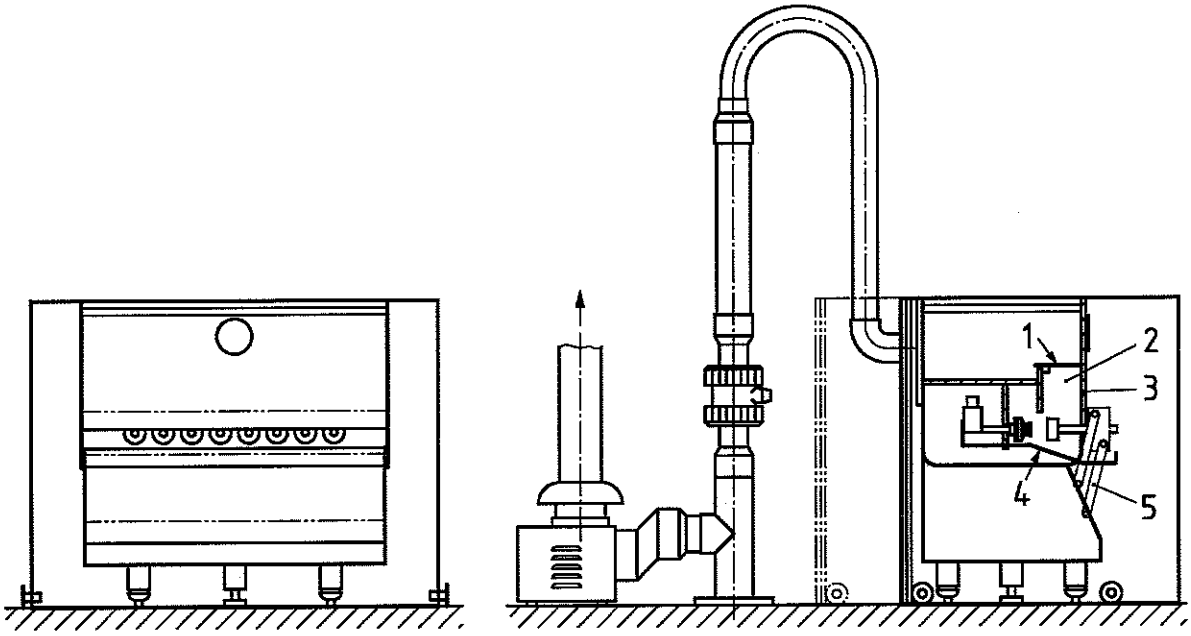
Figure A.3 — Smoking machine type b) with fixed puff-termination position



Key

1 baffle plate
2 internal duct
3 hinged front flap
4 ashtray
5 lighter bar

Figure A.4 — Schematic view of a 20-channel linear machine type b)



Key

1 baffle plate
2 internal duct
3 hinged front flap
4 ashtray
5 lighter bar

Figure A.5 — Schematic view of an 8-channel linear machine type b): General view without carbon monoxide collection

Annex B
(informative)

Description of the puffing mechanism of a piston-type smoking machine

B.1 Scope

This annex describes the use of the piston principle but it is not intended to preclude or restrict the future development of smoking machines.

B.2 Principle of the puffing mechanism

Examples of piston/crankshaft mechanisms are given in Figure B.1.

B.3 Special considerations

B.3.1 Total swept volume

The total swept volume is the volume of air displaced when the piston passes from the top dead centre to the bottom dead centre. It may be up to 3 % greater than the puff volume. A typical example of the resultant swept profile is given in Figure B.2.

B.3.2 Puff volume

The puff volume may be controlled by truncating the "skirt" or "tails" of the swept profile using a valve.

B.4 Design considerations of the puffing mechanism

B.4.1 It would appear that specifications of A, r, l and h (see Figure B.1) are the most important design considerations. Since 2 Ar equals swept volume, r is automatically fixed and l and h determine the shape of the puff. For reasons of symmetry, l and h should be as large as possible. Therefore, in the manufacture of a piston-type smoking machine, the recommendations given in B.4.2 to B.4.6 should apply.

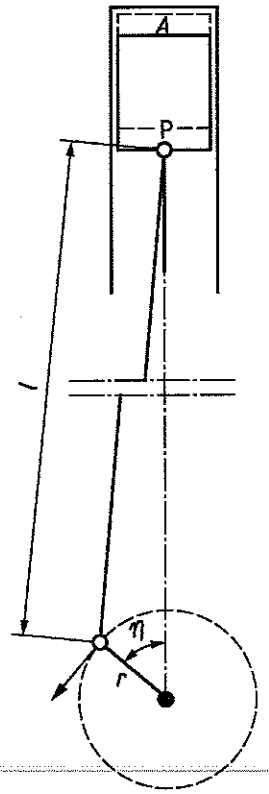
B.4.2 The speed of rotation of the shaft should be constant during puffing. It should be fully adjustable and should have a fine control.

B.4.3 It is desirable that pistons and cylinders be completely interchangeable.

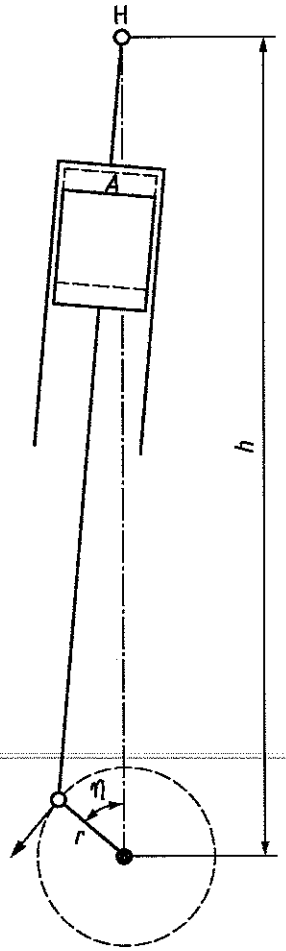
B.4.4 The distance l or h should be greater than 10 r.

B.4.5 The piping between the smoke trap and the cylinder should offer the least possible pressure drop (see 5.3.4).

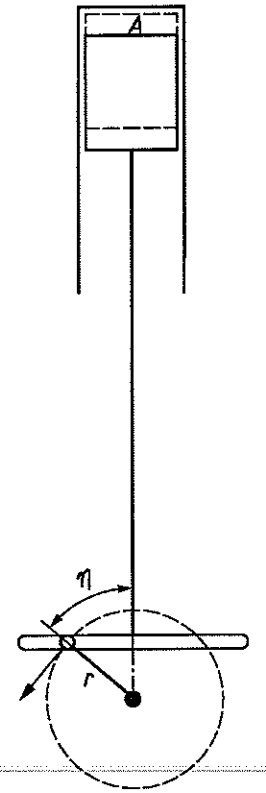
B.4.6 In order to ensure that the machine performs in accordance with the specification, incorporation of a mechanism to start or stop the piston at a definite point may be necessary.



a) Conventional crankshaft with connecting rod and small end



b) Rigid piston connected with cylinder pivoted at H

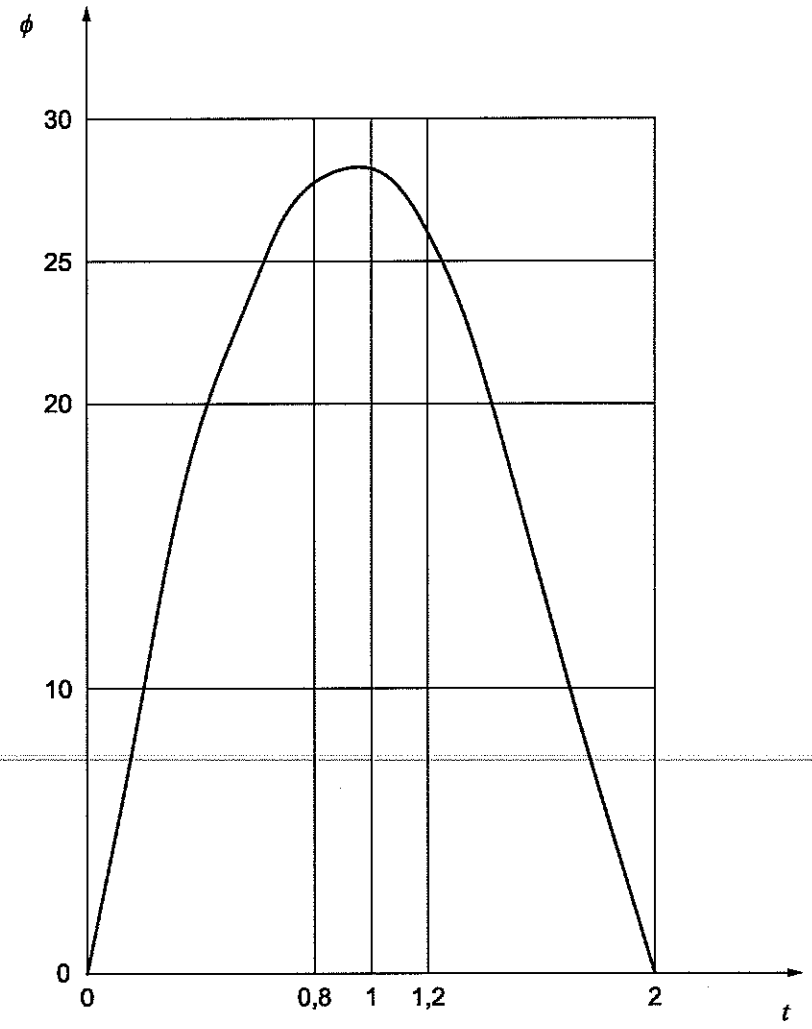


c) Symmetrical piston movement

Key

H, P pivots
A area of cross-section
l length of connection rod
h distance between crank and pivot H
r radius of crank
η angular displacement of the crankshaft

Figure B.1 — Examples of piston/crankshaft mechanisms



Key

Φ flow rate, ml/s

t time, s

Figure B.2 — Typical puff profile without cigarette (swept profile)

Annex C
(informative)

Diagrammatic representation of a puff profile

C.1 To illustrate certain definitions and certain standard conditions, an example of a puff profile is shown in Figure C.1.

At time $t = 0$, suction may be applied to the cigarette by means of a piston pump. The resulting flow rate Φ at the butt end of the cigarette varies to give a bell-shaped puff profile. The maximum flow rate Φ_m is reached at time t_m. The flow rate then decreases during the puff duration to reach the value Φ_d at time t_d when the puffing source ceases to apply suction, but a pressure differential still exists.

Finally, the flow rate decreases slowly to zero, a value reached at time t_e.

C.2 The standard puff profile has its maximum so that

$$25 \text{ ml/s} \leq \Phi_m \leq 30 \text{ ml/s}$$

at time t_m so that

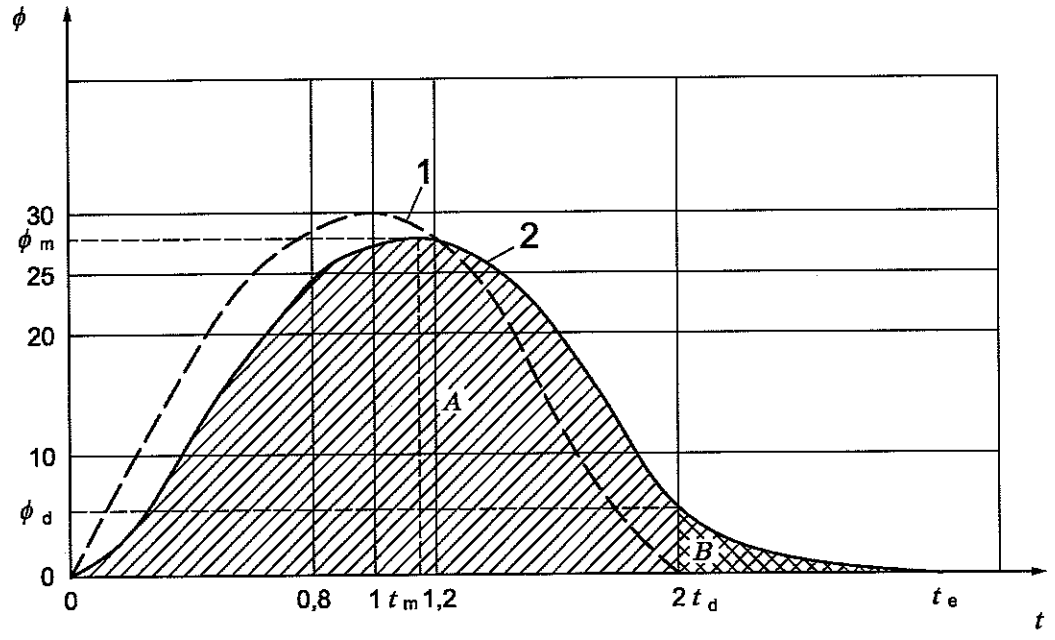
$$0{,}8 \text{ s} \leq t_m \leq 1{,}2 \text{ s}$$

The standard puff duration is t_d = 2 s and the time t_e is consequently limited by the standard puff frequency to t_e = 60 s.

The puff volume, V, may be calculated on the basis of the shaded area in Figure C.1 from the formula:

$$V = \int_0^{t_e} \Phi(t)\,dt = A + B = \int_0^{t_d} \Phi(t)\,dt + \int_{t_d}^{t_e} \Phi(t)\,dt$$

The consequence of the standard conditions is the following:



V=35 ml

$$A = \int_0^{t_d} \Phi(t)\,dt \geq 0{,}95\,V$$

Key

1 swept profile
2 puff profile
Φ flow rate, ml/s
t time, s

Figure C.1 — Diagrammatic representation of a puff profile with cigarette

Bibliography

[1] ISO 558:1980, *Conditioning and testing — Standard atmospheres — Definitions*

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

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

[4] ISO 7210, *Routine analytical cigarette-smoking machine — Additional test methods*

ISO 3308:2012(E)

ICS 65.160

Price based on 25 pages

Nederlandse

norm

NEN-ISO 4387 (en)

Sigaretten - Bepaling van het totale en nicotine-vrije droge rookcondensaat bij gebruik van een sigarettenrookmachine voor routinematig analytisch onderzoek (ISO 4387:2000,IDT)

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

mei 2000
ICS 65.160

Als Nederlandse norm is aanvaard:

- ISO 4387:2000,IDT

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INTERNATIONAL STANDARD

ISO 4387

Third edition
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Cigarettes — Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine

Cigarettes — Détermination de la matière particulaire totale et de la matière particulaire anhydre et exempte de nicotine au moyen d'une machine à fumer analytique de routine

Reference number
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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 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 International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 4387 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

This third edition cancels and replaces the second edition (ISO 4387:1991), which has been editorially revised.

Annex A of this International Standard is for information only.

Introduction

Cigarettes are manufactured to close tolerances using strict quality control procedures. However, all the constituents involved in the manufacture are derived from natural products (tobacco, cigarette paper, tipping, etc.) and this results in a final product which is intrinsically variable. The complexity does not finish here because the cigarette is converted during smoking to cigarette smoke.

Cigarette smoke is a complex mixture consisting of many individual chemical constituents. These compounds exist as gases, vapours and condensed aerosol particles. Additionally, various ageing processes, together with diffusional and intersolubility effects, start occurring immediately after the formation of the smoke which further complicate its composition.

The quantitative measurement of nicotine-free dry particulate matter (NFDPM, sometime referred to as "tar") is, therefore, dependent on its arbitrary definition.

From the time that scientists have attempted to determine a value for NFDPM, many different methods have been used. However, experience has shown some procedures to be more reliable and, with these factors in mind, during 1988 and 1989, collaborative studies by Task Forces composed of members of the Cooperation Centre for Scientific Research Relative to Tobacco (CORESTA) Smoke and Technology groups have been made on the repeatability and reproducibility of the determination of total and dry particulate matter from cigarettes.

The studies show that improvements in repeatability and reproducibility result when some restrictions are placed on the wide variety of methods and practices permitted by existing standard methods. Thus, this International Standard, and the others which together form a complete set for the sampling, conditioning and determination of nicotine, water and particulate matter from cigarettes, have been produced after much cooperation and collaborative experimentation by many laboratories in many countries.

CORESTA first published an International Standard for the machine smoking of cigarettes in 1968, and since that time many improvements in equipment as well as in procedure have been suggested.

This International Standard incorporates these improvements and consequently represents the state of the art on this subject and provides one set of procedures accepted as reference methods.

This method is a machine method and allows cigarettes to be smoked using a strictly controlled set of parameters. Thus, it enables the NFDPM and nicotine from cigarettes, when smoked by this procedure, to be compared and ranked on the basis of machine yield.

INTERNATIONAL STANDARD ISO 4387:2000(E)

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

1 Scope

This International Standard specifies methods for the determination of total particulate matter and for the subsequent determination of nicotine-free dry particulate matter present in the smoke from cigarettes generated and collected using a routine analytical smoking machine.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents 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 2971, *Cigarettes and filter rods — Determination of nominal diameter —Method using a laser beam measuring apparatus.*

ISO 3308:2000, *Routine analytical cigarette-smoking machine — Definitions and standard conditions.*

ISO 3402, *Tobacco and tobacco products — Atmosphere for conditioning and testing.*

ISO 6488-1, *Tobacco — Determination of water content — Part 1: Karl Fischer method.*

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

ISO 8243, *Cigarettes — Sampling.*

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 16055, *Tobacco and tobacco products — Monitor test piece — Requirements and application.*

3 Terms, definitions and abbreviated terms

For the purposes of this International Standard, the following terms, definitions and abbreviated terms apply.

3.1
total particulate matter
crude smoke condensate
TPM
that portion of the mainstream smoke which is trapped in the smoke trap, expressed as milligrams per cigarette

3.2
dry particulate matter
dry smoke condensate
DPM
total particulate matter after deduction of its water content, expressed as milligrams per cigarette

3.3
nicotine-free dry particulate matter
nicotine-free dry smoke condensate
NFDPM
dry particulate matter after deduction of its nicotine content, expressed as milligrams per cigarette

3.4
smoking process
use of a smoking machine to smoke cigarettes from lighting to final puff

3.5
smoking run
specific smoking process to produce such smoke from a sample of cigarettes as is necessary for the determination of the smoke components

3.6
laboratory sample
sample intended for laboratory inspection or testing and which is representative of the gross sample or the sub-period sample

3.7
test sample
cigarettes for test taken at random from the laboratory sample and which are representative of each of the increments making up the laboratory sample

3.8
conditioning sample
cigarettes selected from the test sample for conditioning prior to tests

3.9
test portion
group of cigarettes prepared for a single determination and which is a random sample from the test sample or conditioned sample, as appropriate

3.10
monitor test piece
cigarette taken from a batch specially fabricated under controlled manufacturing conditions

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

4 Principle

The test cigarettes are sampled then conditioned. The test cigarettes are smoked on an automatic smoking machine with simultaneous collection of total particulate matter in a glass fibre filter trap. If used, the consistency of the laboratory smoking process and subsequent analytical procedures are controlled by using monitor test pieces specified in ISO 16055. The mass of the total particulate matter so collected is determined gravimetrically. The total particulate matter is extracted from the trap for determination of the water and nicotine contents by gas chromatography.

NOTE In the 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 requirements of ISO 3308.

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

5.3 Apparatus for the determination of puff duration and frequency.

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

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

5.5 Conditioning enclosure, carefully maintained under the conditions specified in ISO 3402.

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

5.7 Device for the determination of diameter, in accordance with ISO 2971.

If such apparatus is not available, the diameter may be determined from the circumference by slitting the cigarette longitudinally, removing and flattening the paper then measuring its width.

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

5.9 Gloves, made of cotton, or the non-talc surgical type.

6 Sampling

A laboratory sample (3.6) shall be taken by a sampling scheme such as one of those given in ISO 8243.

This sample will normally contain cigarettes taken from different parts of the population. Make up the test sample (3.7) required for the test by randomly selecting cigarettes from the different parts of the population represented in the laboratory sample.

7 Determination of total particulate matter

7.1 Preparation of the cigarettes for smoking

7.1.1 General

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

The symbols used in this clause are as follows:

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

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

Q is the total number of cigarettes available (laboratory sample, see 3.6);

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

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

P is the total number of packets of cigarettes available.

NOTE The multiplier C is usually at least 1,2 to provide extra cigarettes in case some are damaged and for optional tests which may be required (see 7.5). If selection by mass or draw resistance (or any other parameter) is necessary, C will have to be much larger (experience suggests 2 to 4) depending on the selection process.

The precision normally required generally demands that $80 \leqslant \quad \leqslant 100$. This number may be considerably augmented if the variability of the sample is high; on the contrary, in certain comparisons made of homogeneous samples, this number may be reduced. It can also be reduced when N represents a sub-period sample. N shall never be less than 40 when 20 cigarettes are smoked per trap, or less than 20 when 5 cigarettes are smoked per trap.

It is necessary for 40 cigarettes to be smoked when 20 cigarettes are smoked per trap, thus providing a replicate analysis and data replication.

The N cigarettes to be smoked will be tested in $n = N/q$ determinations if q cigarettes 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.

7.1.2 Selection of test portions from a bulk of Q cigarettes

If the test sample is in the form of a single bulk, consisting of Q cigarettes, $C \times N$ cigarettes shall be selected at random so that every cigarette has an equal probability of being selected.

7.1.3 Selection of test portions from P packets

If the test sample consists of P packets, the selection procedure depends upon the number of cigarettes in each packet (Q/P) compared with q.

If $Q/P \geqslant C \times q$, select a test portion by choosing a single packet at random, then randomly select $C \times q$ cigarettes from that packet.

If $Q/P < C \times q$, select the smallest number of packets (k) such that

$$\frac{Q \times k}{P} \geqslant C \times q$$

and randomly choose an equal (or as near equal as possible) number of cigarettes from each packet to form the test portion of $C \times q$ cigarettes.

7.1.4 Duplicate test portions

Provided that the test sample is sufficiently large ($\geqslant 2C \times N$), a duplicate set of n test portions should be reserved. In this event the parallel selection of a test portion and its duplicate would seem sensible. In this case the two selection conditions of 7.1.3 would need to be changed to $Q/P \geqslant 2C \times q$ and $Q/P < 2C \times q$.

7.2 Marking the butt length

7.2.1 Standard butt length

The standard butt length to which cigarettes shall be marked shall be the greatest of the following three lengths:

— 23 mm,

— length of filter + 8 mm, or

— length of overwrap + 3 mm,

where the overwrap is defined as any wrapper applied to the mouth end of the cigarette, and the length of the filter is defined as the total length of the cigarette minus the length of the tobacco portion.

NOTE Butt length is defined in ISO 3308 as the length of unburnt cigarette remaining at the moment when smoking is stopped.

7.2.2 Measurement of length of filter

The length of filter as defined in 7.2.1 shall be the mean value of 10 cigarettes taken from the laboratory sample, measured to an accuracy of 0,5 mm. Express the mean to the nearest 0,5 mm.

NOTE In some instances it may be necessary to measure more than 10 cigarettes, but when the variation in filter length can be demonstrated to be well controlled, a smaller number of measurements may be sufficient.

7.2.3 Measurement of length of overwrap

The length of overwrap as defined in 7.2.1 shall be the mean value of 10 overwraps taken from the laboratory sample, measured to an accuracy of 0,5 mm. Express the mean to the nearest 0,5 mm.

NOTE In some instances it may be necessary to measure more than 10 cigarettes, but when the variation in overwrap length can be demonstrated to be well controlled, a smaller number of measurements may be sufficient.

7.2.4 Butt length to be marked on the cigarettes before conditioning

Draw a line, using a fine soft-tipped marker, at the standard butt length, to an accuracy of 0,5 mm, from the mouth end for the particular cigarette type.

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

If cigarettes are to be smoked on a smoking machine on which the butt length in accordance to 7.2.1 can be pre-set, it is not necessary to mark the butt lengths on the cigarettes themselves.

7.3 Selection of cigarettes

If a selection by mass or draw resistance (or any other parameter) is necessary because of the nature of the problem being studied, the selection shall not be considered as a method of reducing the number of cigarettes to be smoked.

7.4 Conditioning

Condition all the test portions in the conditioning atmosphere specified in ISO 3402 for a minimum of 48 h and a maximum of 10 days.

If for any reason test samples are to be kept for longer than 10 days before conditioning, store them in 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 also be in accordance with ISO 3402.

Transfer the test portions to the smoking location in airtight containers (just large enough to contain the portions) unless the smoking location and the conditioning location are adjoining and have identical atmospheres.

7.5 Preliminary tests before smoking

The following data may be required in the test report:

a) total length of the cigarette;

b) nominal diameter, determined in accordance with ISO 2971;

c) draw resistance of the cigarette, determined in accordance with ISO 6565;

d) average mass of the conditioned cigarettes selected for the smoking operation (in milligrams per cigarette);

e) water content (as a mass fraction) of the conditioned cigarettes, determined in accordance with ISO 6488-1.

7.6 Smoking and collection of particulate matter

7.6.1 Smoking plan

Choose a smoking plan; examples of plans are given in informative annex A.

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

The plan should include the use of a test portion of monitor test pieces. The test pieces are included in the plan as if they were a type of cigarette and prepared and smoked as in 7.6.4, 7.7, 7.8 and 7.9.

7.6.2 Preparation of smoke traps and cigarette holders

For all operations, the operator shall prevent contamination from the fingers by wearing gloves of a suitable material (5.9).

Insert filter discs which have been conditioned in the test atmosphere 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. If the smoke trap is designed to contain the perforated disc (washer), insert it and fit the sealing devices (end caps). If the cigarette holder is designed to contain a perforated disc, insert it into the cigarette holder before attaching the labyrinth seals (see ISO 3308:2000, 4.8). Weigh the assembled smoke traps to the nearest 0,1 mg.

Because of absorption of water by smoke traps and solvent, it is necessary to determine a value for the sample blank. Prepare sample blanks by treating additional smoke traps (at least 2 per 100 cigarettes) in the same manner as that used for smoke collection.

7.6.3 Setting up the smoking machine

7.6.3.1 General

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.

The puff volume should be checked if it is suspected that the smoking machine is subject to a large change in temperature during use.

7.6.3.2 Measurement of puff duration

A timer, working with reference to a crystal-controlled oscillator, 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. The accuracy of the timing device shall be such as to ensure that a 1 % error in the puff duration can be detected. The timer should be coupled directly to the triggering circuits.

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.

7.6.3.3 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. The timer used shall be suitable for measuring to the nearest 0,1 s and should, preferably, be coupled directly to the triggering circuits.

7.6.3.4 Measurement of puff volume

The displacement of the bubble in a soap bubble flow meter (5.2) gives a direct measurement of puff volume and also provides a check for leaks in the system. A suitable indicator graduated at 35 ml shall have a resolution of 0,1 ml. It shall be connected through a standard pressure drop device of 1 kPa ± 5 % to the cigarette holder of the smoking machine channel under test. Before use for a series of measurements, wet the instrument twice with detergent solution and then allow it to drain for a period of between 30 s and 45 s.

The bubble flow meter shall contain a mass fraction of 15 % aqueous solution of a surface active agent. Teepol L ® [1] has been found to be satisfactory. The concentration of Teepol as purchased must be known before carrying out further dilution.

Fit the prepared smoking trap or traps and cigarette holders onto the machine. Attach a plastic insert of an appropriate size for the labyrinth seals in the cigarette holder to the resistance in 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 cigarette holder in port 1 and determine the puff volume; adjust if necessary to (35,0 ± 0,3) ml. Repeat for all remaining ports in turn.

Repeat the determinations until the necessary precision of measurement is obtained. If the number of replicates exceeds three, continue until the correct precision is obtained but replace the pad before smoking, reweigh the smoke trap and recheck the puff volume with the new pad in place. Measure the temperature and relative humidity of the air surrounding the smoking machine and note the atmospheric pressure.

7.6.4 Procedure for smoking run

Insert the conditioned cigarettes from the test portion into the cigarette holders so that the butt end impinges upon the perforated disc (washer) fitted within the filter trap. Avoid any leaks or deformations. Any cigarettes found to have obvious defects, or which have been damaged during insertion, shall be discarded and replaced with spare, conditioned cigarettes.

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[1] Teepol L is the trade name of a product supplied by Shell. This information is given for the convenience of users of this International Standard 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.

Ensure that the cigarettes are positioned correctly so that the axes of the cigarettes coincide with the axes of the ports. Adjust the position of each cigarette so that when the burning coal reaches the butt mark, the puff termination device is activated. If the burning through of 100 % cotton thread of (48 ± 4) tex is used to terminate smoking at the butt mark, the cotton shall just touch the cigarette at the butt mark, without modifying the cigarette positioning.

Zero the puff counters and light each cigarette at the beginning of its first puff. Should it be necessary to relight a cigarette, a hand-held electrical lighter may be used. When each butt mark has been reached, remove the burning coal from the cigarette and note the final reading of the puff counters. After the smoking process is complete, leave the cigarette 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.

If required, new cigarettes shall be inserted immediately and the smoking process repeated until the predetermined number of cigarettes, in accordance with the smoking plan, has been smoked into the smoke trap. Immediately begin the determination of total particulate matter as described in 7.7.

7.7 Determination of total particulate matter

Remove the smoke traps from the smoking machine (gloves shall be worn). Where necessary, remove the cigarette holder from the smoke trap.

Cover the front and back apertures of the trap with the sealing devices (5.8).

It is recommended, particularly when plain cigarettes have been smoked, that the removal of the holder be conducted with the smoke trap held with its cigarette-facing side downwards to avoid any possible contaminants from the cigarette 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 pad damage. Discard any disc showing such stains or damage.

Glass fibre filter pads of 44 mm diameter are capable of retaining up to 150 mg of total particulate matter and pads of 92 mm diameter are capable of retaining 600 mg of TPM. If, during smoking, this mass is exceeded, the number of cigarettes shall be reduced and a calculation made to allow for the reduced number of cigarettes smoked.

7.8 Calculation of total particulate matter

The TPM content, m_{TPM}, for each channel, expressed in milligrams per cigarette, is given by the equation:

$$m_{TPM} = \frac{m_1 - m_0}{q}$$

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 cigarettes smoked into the trap.

7.9 Treatment of total particulate matter

7.9.1 Extraction procedure

Remove the sealing devices from the smoke trap (gloves 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.

7.9.2 Determination of water

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

The DPM content, m_{DPM}, for each trap, expressed in milligrams per cigarette, is given by the equation:

$$m_{DPM} = m_{TPM} - m_W$$

where

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

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

7.9.3 Determination of nicotine

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

The NFDPM content, m_{NFDPM}, for each trap, expressed in milligrams per cigarette, is given by the equation:

$$m_{NFDPM} = m_{DPM} - m_N$$

where

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

m_N is the nicotine content in the TPM, in milligrams per cigarette.

8 Test report

The test report shall show the method used and the results obtained. It shall also mention any operating conditions not specified in this International Standard, 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 below in a) to d) shall be recorded.

a) Characteristic data about the cigarette

All details necessary for the identification of the cigarettes smoked shall be given. In the case of commercial cigarettes this should include:

— name of manufacturer and country of manufacture,

— product name,

— packet number (of the product sampled that day),

— marks on any tax stamp,

— printed smoke yields (if any),

— length of cigarette,

— length of filter,

— length of overwrap.

b) Data about sampling

— type of sampling procedure,

— date of sampling,

— place of purchase or sampling,

— kind of sampling point,

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

— number of cigarettes in laboratory sample.

c) Description of test

— reference to this International Standard,

— date of test,

— type of smoking machine used,

— type of smoke trap used,

— total number of cigarettes smoked,

— number of cigarettes smoked into each smoke trap,

— butt length,

— room temperature (in degrees Celsius) during smoking operation,

— relative humidity (in percent) during smoking operation,

— atmospheric pressure (in kilopascals) during smoking operation.

d) 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. Details should include the following:

— average length of the cigarettes to the nearest 0,1 mm,

— average length of the filter to the nearest 0,5 mm,

— average length of the overwrap to the nearest 0,5 mm,

— butt length to which cigarettes were smoked,

— average diameter of the cigarettes to the nearest 0,01 mm,

— average number of puffs per cigarette for each channel to the nearest 0,1 puff,

— TPM content (in milligrams per cigarette) for each channel to the nearest 0,1 mg, and the average per cigarette to the nearest 1 mg,

— DPM content (in milligrams per cigarette) for each channel to the nearest 0,1 mg, and the average per cigarette to the nearest 1 mg,

— NFDPM content (in milligrams per cigarette) for each channel to the nearest 0,1 mg, and the average per cigarette to the nearest 1 mg.

9 Repeatability and reproducibility

A major international collaborative study involving 30 laboratories and 6 samples conducted in 1990 showed the following values for the repeatability limits (r) and the reproducibility limits (R) of this method.

The difference between two single results found on matched cigarette samples by one 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 cigarette 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 Table 1.

Table 1 — Estimates given by data analysis

Values in milligrams per cigarette

Mean value m_{NFDPM}	Repeatability limit r	Reproducibility limit R
0,82	0,40	0,60
1,61	0,52	0,74
3,31	0,52	0,90
7,70	0,88	1,51
12,61	1,06	1,70
17,40	1,19	1,84

For the purpose of calculating r and R, one test result was defined as the mean yield obtained from smoking 20 cigarettes in a single run.

For further details of the interaction of r and R with other factors, see CORESTA Report 91/1.

The subject of tolerances due to sampling is dealt with in ISO 8243.

Annex A
(informative)

Smoking plans

In the majority of cases, the results of mechanical smoking permit a comparison of types of cigarettes (treatments). This comparison should be made according to a smoking plan established in advance; the smoking plan should take account of the following:

a) the capacity and the variability of the smoking machine: number of channels;

b) the capacity of the smoke traps: this determines the number of cigarettes to be smoked in each channel;

c) the nature of the cigarettes: for those of high condensate yield it is prudent to reduce the number to be smoked in each channel;

d) required precision: the results of smoking always give a certain variability; the distribution of the treatments in each smoking run and of the smoking runs in time should reduce the effects of uncontrolled or badly controlled factors (mechanical or personal); in general, the larger the test portion, the greater the precision.

The order of magnitude of the number N of cigarettes in a test portion is fixed for each type as a function of various factors, in particular:

— the precision sought;

— the time necessary for the smoking processes, which is itself related to the capacity of the machine.

The exact value to be selected for N, chosen in the ranges above (see 7.1), taking into account the preceding factors, is determined by calculation for each experiment taking into account the parameters which characterize it.

The different parameters are related by the equation

$$t \times N = s \times c \times q$$

where

t is the number of types to be compared (treatments);

s is the number of smoking runs to be carried out;

c is the number of channels on the machine;

q is the number of cigarettes smoked into the same trap.

The examples of smoking plans proposed below illustrate the preceding remarks. They could correspond to the following objectives.

a) EXAMPLE 1: Comparison of two types of cigarettes on one single-channel smoking machine. The smoke trap can collect the condensate of five cigarettes.

b) EXAMPLE 2: Comparison of three types of cigarettes on one single-channel smoking machine. The smoke trap can collect the condensate of 20 cigarettes.

c) EXAMPLE 3: Comparison of two types of cigarettes on one four-channel smoking machine. The smoke trap can collect the condensate of five normal cigarettes. As the test cigarettes have high condensate yield (e. g. above 30 mg per cigarette) the number smoked should be reduced to three.

d) EXAMPLE 4: Comparison of 20 types of cigarettes on one 20-channel smoking machine. The smoke trap can collect the condensate of five normal cigarettes. Higher precision required.

e) EXAMPLE 5: Comparison of five types of cigarettes on one 20-channel smoking machine. The smoke trap can collect the condensate of five normal cigarettes. Higher precision required.

EXAMPLE 1: Comparison of two types of cigarettes on one single-channel smoking machine

Number of treatments	$t = 2$ (A, B)
Number of cigarettes in the test sample	$N = 40$
Number of cigarettes per channel	$q = 5$
Number of channels	$c = 1$
Number of smoking runs	$s = 16$ (1, 2, ... 16)
Thus testing 80 cigarettes	$2 \times 40 = 16 \times 1 \times 5$

The number N of cigarettes to be smoked is limited to 40 of each type, so that the duration of the smoking process is not too long. Each smoking run carries only one treatment. Distribute the runs in time while repeating four times the sequence shown in Table A.1 (k represents successive values 0, 4, 8 and 12):

Table A.1

Run	Treatment
$1 + k$	A
$2 + k$	B
$3 + k$	B
$4 + k$	A

EXAMPLE 2: Comparison of three types of cigarettes on one single-channel smoking machine

Number of treatments	$t = 3$ (A, B, C)
Number of cigarettes in the test sample	$N = 60$
Number of cigarettes per channel	$q = 20$
Number of channels	$c = 1$
Number of smoking runs	$s = 9$ (1, 2, ... 9)
Thus testing 180 cigarettes	$3 \times 60 = 9 \times 1 \times 20$

Each smoking run carries only one treatment. The runs are distributed in time in an ordered fashion, e.g. by means of a matrix of the following type:

B	A	C
C	B	A
A	C	B

Run	1	2	3	4	5	6	7	8	9
Treatment	B	A	C	C	B	A	A	C	B

EXAMPLE 3: Comparison of two types of cigarettes on one four-channel smoking machine

Number of treatments	$t = 2$ (A, B)
Number of cigarettes in the test sample	$N = 48$
Number of cigarettes per channel	$q = 3$
Number of channels	$c = 4$ (a, b, c, d)
Number of smoking runs	$s = 8$ (1, 2, ... 8)
Thus testing 96 cigarettes	$2 \times 48 = 8 \times 4 \times 3$

Allocate the smoking channels to the two treatments utilizing the matrix below, which is constructed for four treatments but which is easily adapted to the case of two treatments by identifying A with C on the one hand and B with D on the other. (In general, all matrices of dimensions g can be utilized for a number of treatments which are sub-multiples of g.)

A	B	C	D
D	C	A	B
B	A	D	C
C	D	B	A

Channel / Run	a	b	c	d
1	A	B	A	B
2	B	A	A	B
3	B	A	B	A
4	A	B	B	A
5	A	B	A	B
6	B	A	A	B
7	B	A	B	A
8	A	B	B	A

In each smoking run, two channels are allocated to each treatment. For example, in run 6:

— cigarette A is smoked in channels b and c,

— cigarette B is smoked in channels a and d.

Each type is smoked four times in each of the four channels.

EXAMPLE 4: Comparison of 20 types of cigarettes on one 20-channel smoking machine

Number of treatments	$t = 20$ (A, B, ... T)
Number of cigarettes in the test sample	$N = 100$
Number of cigarettes per channel	$q = 5$
Number of channels	$c = 20$ (a, b, ... t)
Number of smoking runs	$s = 20$ (1, 2, ... 20)
Thus testing 200 cigarettes	$20 \times 100 = 20 \times 20 \times 5$

Allocate the smoking channels to the 20 treatments utilizing the matrix below:

Channel / Run	a	b	c	d	e	f	g	h	i	j	k	l	m	n	o	p	q	r	s	t
1	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	T
2	D	N	B	J	A	R	H	L	C	O	Q	F	S	K	T	I	E	P	M	G
3	I	A	M	E	K	Q	O	F	H	B	R	J	G	P	C	N	L	S	T	D
4	K	C	I	N	Q	H	M	A	J	F	S	R	B	O	G	L	D	T	P	E
5	B	J	H	S	F	M	P	K	N	A	T	C	R	Q	E	O	G	L	D	I
6	H	D	Q	M	C	S	F	P	T	G	O	E	K	A	I	J	B	N	R	L
7	E	L	G	Q	D	P	K	T	M	S	A	I	N	F	R	C	O	H	J	B
8	M	H	D	P	L	G	S	C	K	T	F	O	J	R	B	Q	I	E	N	A
9	L	Q	F	B	J	O	N	G	R	C	P	K	H	S	D	T	A	I	E	M
10	G	R	L	T	N	D	A	J	Q	H	E	B	O	M	K	F	S	C	I	P
11	N	E	T	I	O	B	J	R	F	K	C	G	L	D	H	M	P	Q	A	S
12	C	O	K	F	B	J	Q	N	A	P	M	S	I	E	L	H	T	D	G	R
13	F	P	A	O	G	C	B	M	S	D	L	N	T	I	J	E	R	K	H	Q
14	P	T	R	H	S	N	D	E	G	I	J	M	F	L	Q	B	K	A	O	C
15	R	K	P	G	T	E	I	O	L	N	H	D	Q	C	S	A	J	M	B	F
16	T	G	E	C	I	K	L	S	O	M	D	Q	P	H	A	R	N	B	F	J
17	S	F	N	R	H	L	T	B	E	Q	I	A	C	J	P	D	M	G	K	O
18	Q	M	O	L	P	T	E	I	D	R	G	H	A	B	N	S	F	J	C	K
19	O	S	J	A	R	I	C	Q	P	E	B	T	D	G	M	K	H	F	L	N
20	J	I	S	K	M	A	R	D	B	L	N	P	E	T	F	G	C	O	Q	H

All the treatments are represented in each smoking run. Overall, each treatment is smoked once in each of the 20 channels.

EXAMPLE 5: Comparison of five types of cigarettes on one 20-channel smoking machine

Number of treatments $t = 5$ (A, B, C, D, E)

Number of cigarettes in the test sample $N = 200$

Number of cigarettes per channel $q = 5$

Number of channels $c = 20$ (a, b ... t)

Number of smoking runs $s = 10$ (1, 2, ... 10)

Thus testing 1 000 cigarettes $5 \times 200 = 10 \times 20 \times 5$

Allocate the smoking channels to five treatments utilizing the matrix below:

D	B	E	A	C
A	D	B	C	E
B	A	C	E	D
C	E	D	B	A
E	C	A	D	B

Channel	a	b	c	d	e	f	g	h	i	j	k	l	m	n	o	p	q	r	s	t
Run																				
1	D	B	E	A	C	C	E	D	A	B	E	C	B	A	D	B	D	A	C	E
2	A	D	B	C	E	A	C	E	B	D	C	E	A	D	B	A	B	D	E	C
3	B	A	C	E	D	E	B	C	D	A	A	D	C	B	E	D	E	C	A	B
4	C	E	D	B	A	B	D	A	E	C	D	B	E	C	A	E	C	B	D	A
5	E	C	A	D	B	D	A	B	C	E	B	A	D	E	C	C	A	E	B	D
6	C	A	E	B	D	B	A	D	E	C	D	A	B	C	E	E	C	A	D	B
7	E	C	B	D	A	D	B	E	C	A	B	D	A	E	C	C	E	D	B	A
8	D	E	C	A	B	A	D	C	B	E	E	B	C	D	A	B	A	C	E	D
9	A	B	D	E	C	C	E	A	D	B	A	C	E	B	D	A	D	B	C	E
10	B	D	A	C	E	E	C	B	A	D	C	E	D	A	B	D	B	E	A	C

In each smoking run, each treatment is smoked in four channels. For example, in run 7:

— cigarette A is smoked in channels e, j, m, t,

— cigarette B is smoked in channels c, g, k, s,

— cigarette C is smoked in channels b, i, o, p,

— cigarette D is smoked in channels d, f, l, r,

— cigarette E is smoked in channels a, h, n, q.

Overall, each treatment is smoked twice in each of the 20 channels.

NOTE It is not always possible to smoke each treatment equally in each of the channels. In the present case, if the number of cigarettes in the test sample were 160 it would be necessary to smoke 8 runs. One could distribute the cigarettes as seen above in runs 1 to 8. Then each type would be smoked once or twice in each of the 20 channels.

Bibliography

[1] ISO 3400, *Cigarettes — Determination of alkaloids in smoke condensates — Spectrometric method.*

[2] ISO 10362-2, *Cigarettes — Determination of water in smoke condensates — Part 2: Karl Fischer method.*

[3] CORESTA Report 91/1; *Information Bulletin of Cooperation Centre for scientific research relative to tobacco,* 1991-1, ISSN 0525-6240.

ISO 4387:2000(E)

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Nederlandse norm

NEN-ISO 4387/A1

(en)

Cigarettes - Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine (ISO 4387:2000/Amd 1:2008,IDT)

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Als Nederlandse norm is aanvaard:

- ISO 4387:2000/Amd 1:2008,IDT

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INTERNATIONAL STANDARD

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Third edition
2000-04-01

AMENDMENT 1
2008-09-15

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

AMENDMENT 1

Cigarettes — Détermination de la matière particulaire totale et de la matière particulaire anhydre et exempte de nicotine au moyen d'une machine à fumer analytique de routine

AMENDEMENT 1

Reference number
ISO 4387:2000/Amd.1:2008(E)

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Foreword

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Amendment 1 to ISO 4387:2000 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

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

AMENDMENT 1

Page v, Introduction

Add the following text at the end of the existing text:

No machine smoking regime can represent all human smoking behaviours:

— it is recommended that cigarettes also be tested under conditions of a different intensity of machine smoking than those specified in this International Standard;

— machine smoking testing is useful to characterize cigarette emissions for design and regulatory purposes, but communication of machine measurements to smokers can result in misunderstandings about differences in exposure and risk across brands;

— smoke emission data from machine measurements may be used as inputs for product hazard assessment, but they are not intended to be nor are they valid as measures of human exposure or risks. Communicating differences between products in machine measurements as differences in exposure or risk is a misuse of testing using ISO standards.

ISO 4387:2000/Amd.1:2008(E)

ICS 65.160

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Nederlandse norm

NEN-ISO 4387/A2

(en)

Sigaretten - Bepaling van het totale en nicotine-vrije droge rookcondensaat bij gebruik van een rookmachine voor routinematig analytisch onderzoek van sigaretten (ISO 4387:2000/Amd 2:2017,IDT)

Cigarettes - Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine (ISO 4387:2000/Amd 2:2017,IDT)

ICS 65.160

oktober 2017

Als Nederlands wijzigingsblad is aanvaard:

- ISO 4387:2000/Amd 2:2017,IDT

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INTERNATIONAL STANDARD

ISO 4387

Third edition
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AMENDMENT 2
2017-09

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

AMENDMENT 2

Cigarettes — Détermination de la matière particulaire totale et de la matière particulaire anhydre et exempte de nicotine au moyen d'une machine à fumer analytique de routine

AMENDEMENT 2

Reference number
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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

ISO 4387:2000/Amd.2:2017(E)

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

AMENDMENT 2

7.6.3.4, second paragraph

Replace the paragraph with the following:

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

Delete the following footnote:

> 1) Teepol L is the trade name of a product supplied by Shell. This information is given for the convenience of users of this International Standard 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.

ISO 4387:2000/Amd.2:2017(E)

ICS 65.160

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Waarom betaalt u voor een norm?
Normen zijn afspraken voor en door de markt, zo ook deze norm. NEN begeleidt het gehele normalisatieproces. Van het bijeenbrengen van partijen, het maken en vastleggen van de afspraken en het bieden van hulp bij de toepassing van de normen. Om deze diensten te kunnen bekostigen betalen alle belanghebbende partijen die aan tafel zitten voor het normalisatieproces, en u als gebruiker voor normen en trainingen. NEN is een stichting en heeft geen winstoogmerk.

Wat is nu precies de toegevoegde waarde van normen?
Stelt u zich eens voor ... u wilt in het buitenland geld pinnen, maar uw bankpas past niet. Of uw nieuwe telefoon herkent uw simkaart niet. De samenstelling van de benzine over de grens is anders waardoor u niet kunt tanken. Het dagelijks leven zou zonder goede afspraken over producten, processen en diensten een stuk complexer zijn.

Het maken en vastleggen van afspraken door belanghebbende partijen noemen we het normalisatieproces. Normalisatie had vanouds betrekking op techniek en producten. Nu worden steeds vaker normen voor diensten ontwikkeld. Zo zijn er afspraken op het gebied van gezondheidszorg, schuldhulpverlening, kennisintensieve dienstverlening, externe veiligheid en MVO.

Normen zorgen voor verbetering van producten, diensten en processen; qua veiligheid, gezondheid, efficiëntie, kwaliteit en duurzaamheid. Dit ziet u op de werkvloer, in de omgang met elkaar en in de samenleving als geheel. Organisaties die normalisatie onderdeel van hun strategie maken, vergroten hun professionaliteit, betrouwbaarheid en concurrentiekracht.

Wat doet NEN?
NEN ondersteunt in Nederland het normalisatieproces. Als een partij zich tot NEN richt met de vraag om een afspraak tot stand te brengen, gaan wij aan de slag. We onderzoeken in hoeverre normalisatie mogelijk is en er interesse voor bestaat. Wij nodigen vervolgens alle belanghebbende partijen uit om deel te nemen. Een breed draagvlak is een randvoorwaarde. De afspraken komen op basis van consensus tot stand en worden vastgelegd in een document. Dit is meestal een norm. Afspraken die in een NEN-norm zijn vastgelegd mogen niet conflicteren met andere geldige NEN-normen. NEN-normen vormen samen een coherent geheel. Een belanghebbende partij kan een producent, ondernemer, dienstverlener, gebruiker, maar ook de overheid of een consumenten- of onderzoeksorganisatie zijn. De vraag is niet altijd om een norm te ontwikkelen. Vanuit de overheid komt regelmatig het verzoek om te onderzoeken of er binnen een bepaalde sector of op een bepaald terrein normalisatie mogelijk is. NEN doet dan onderzoek en start afhankelijk van de uitkomsten een project. Deelname staat open voor alle belanghebbende partijen. NEN beheert ruim 30.000 normen. Dit zijn de in Nederland aanvaarde internationale (ISO, IEC), Europese (EN) en nationale normen (NEN). In totaal zijn er ruim 800 normcommissies actief met in totaal bijna 5.000 normcommissieleden. Een goed beheer van de omvangrijke normencollectie en de afstemming tussen nationale, Europese en internationale normcommissies vereisen dan ook een zeer goede infrastructuur.

Betalen kleine organisaties net zoveel als grote organisaties?
Het uitgangspunt is dat alle partijen die deelnemen aan het normalisatieproces een evenredig deel betalen. De normcommissieleden kunnen onderling andere afspraken maken. Zo worden er wel eens afspraken gemaakt dat de grote partijen een groter deel betalen dan de kleinere bedrijven. De prijzen voor normen zijn voor iedereen gelijk. De kosten voor licenties zijn afhankelijk van de omvang van een organisatie en het aantal gebruikers.

Voordelen van normalisatie en normen
Gegarandeerde kwaliteit | Veiligheid geborgd | Bevordert duurzaamheid | Opschalen en vermarkten van nieuwe innovatieve producten | Meer (internationale) handelsmogelijkheden | Verhoogde effectiviteit en efficiëntie | Onderscheidend in de markt.

Voordelen van deelname
Invloed op de (internationale en Europese) afspraken | Als eerste op de hoogte van veranderingen | Netwerk; ook op Europees en internationaal niveau | Kennisvergroting.

Nederlandse norm

NEN-ISO 8454

(en)

Cigarettes - Determination of carbon monoxide in the vapour phase of cigarette smoke - NDIR method (ISO 8454:2007,IDT)

Vervangt NEN-ISO 8454:1996

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Als Nederlandse norm is aanvaard:

- ISO 8454:2007,IDT

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INTERNATIONAL STANDARD

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Cigarettes — Determination of carbon monoxide in the vapour phase of cigarette smoke — NDIR method

Cigarettes — Dosage du monoxyde de carbone dans la phase gazeuse de la fumée de cigarette — Méthode IRND

Reference number
ISO 8454:2007(E)

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ISO 8454 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

This third edition cancels and replaces the second edition (ISO 8454:1995), which has been technically revised.

INTERNATIONAL STANDARD ISO 8454:2007(E)

Cigarettes — Determination of carbon monoxide in the vapour phase of cigarette smoke — NDIR method

1 Scope

This International Standard specifies a method for the determination of carbon monoxide (CO) in the vapour phase of cigarette smoke.

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

ISO 3402, *Tobacco and tobacco products — Atmosphere for conditioning and testing*

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

3 Terms and definitions

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

3.1
vapour phase
portion of smoke, which passes the particulate phase trap during smoking in accordance with ISO 4387 using a machine conforming to ISO 3308

3.2
clearing puff
any puff taken after a cigarette has been extinguished or removed from the cigarette holder

4 Principle

Smoking of cigarettes in accordance with the procedures given in ISO 4387. Collection of the vapour phase of the cigarette smoke and measurement of the carbon monoxide using a non-dispersive infrared (NDIR) analyser calibrated for carbon monoxide. Calculation of the amount of carbon monoxide per cigarette.

5 Apparatus

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

5.1 Conditioning enclosure, maintained accurately in accordance with the conditions specified in ISO 3402, for conditioning the cigarette sample prior to smoking (see also 7.1).

5.2 Routine analytical cigarette-smoking machine and accessories, complying with the requirements of ISO 3308.

5.3 Vapour-phase collection system, which can be fitted to one or more of the smoking machine channels. The use of the system shall ensure collection of all the vapour phase (normally vented to atmosphere) to be stored in a previously evacuated container for subsequent sampling through an NDIR analyser.

The collection system shall not cause interference with the normal performance of the smoking machine and the consequent determination of total particulate matter and nicotine.

The impermeability of the gas-collecting device to a vapour phase shall be checked with a vapour phase containing a volume fraction of 4 % to 6 % of CO. The CO concentration shall be measured directly after filling the previously evacuated gas-collecting device. After a period of not less than 2 h, the measured value of CO concentration in the vapour phase in the device shall not differ by more than a volume fraction of 0,2 % from the value expected from the first determination.

When a bag is used as the gas-collecting device, it shall be large enough to avoid the final pressure of its contents exceeding the ambient atmospheric pressure. The volume of the bag should also be no greater than twice the volume of the gas content collected at atmospheric pressure. In practice, the collection of the vapour phase from 5 cigarettes requires a volume of 3 l and the collection of the vapour phase from 20 cigarettes requires a volume of 10 l.

5.4 Non-dispersive infrared (NDIR) analyser, selective and calibrated for the measurement of carbon monoxide in vapours and gases.

Analysers are available from several manufacturers and should have a preferred working range of a volume fraction of 0 % to 10 % CO and a sampling rate of between 0,5 l/min and 5 l/min. The analyser shall have a precision of 1 % of full scale, a linearity of 1 % of full scale and a repeatability of 0,2 % of full scale, under conditions of constant temperature and pressure. In terms of volume fractions its response to 10 % CO_2 shall not exceed 0,05 % as CO. Its response to 2 % water vapour shall not exceed 0,05 % as CO.

5.5 Ignition device, effecting flameless ignition. Experience has shown that the lighting process can influence the CO yield considerably. The lighters shall light the cigarettes at the first attempt without either touching or pre-charring the cigarettes. The CO yields are increased by higher lighting intensity.

5.6 Barometer, capable of measuring atmospheric pressures to the nearest 0,1 kPa.

5.7 Thermometer, capable of measuring temperature to the nearest 0,1 °C.

6 Standard gas mixtures

Make-up gas shall be nitrogen as other gases can change the detected response of carbon monoxide. Gases used should be of high purity (with low content of carbon dioxide) and used within the manufacturer's time limits.

The NDIR analyser should be calibrated with at least three standard gas mixtures of accurately known concentrations within a relative error of 2 % covering the expected range in such a way as to avoid extrapolation of the calibration curve. Typically volume fractions of about 1 %, 3 % and 5 % of CO in nitrogen are appropriate.

7 Procedure

7.1 Conditioning

Condition the test portion taken from and representative of the laboratory sample in accordance with ISO 3402. Verify that equilibrium has been properly attained as described in ISO 3402.

The atmosphere in the laboratory where the smoking is to be carried out shall also be in accordance with ISO 3402. Place the conditioned test portion in an airtight container (just large enough to contain the portion) and remove each cigarette from the container just before smoking.

7.2 Calibration of the NDIR analyser

7.2.1 Warm up the instrument according to the manufacturer's recommendations, purge the instrument with air and adjust to read zero.

7.2.2 Fill a previously evacuated vapour-phase collection container with the standard gas mixture of a volume fraction of about 5 % CO, re-evacuate and refill with gas. Ensure that the gas in the container is at ambient temperature and pressure. Introduce the gas into the measuring cell using the system sampling pump allowing 5 s to 10 s for equilibration of pressure of the analyser. Note the reading on the analyser concentration display when a steady value has been obtained.

If necessary, adjust the analyser reading to agree with the certified value of the standard gas.

7.2.3 Repeat the procedure as specified in 7.2.2 for at least two other standard gas mixtures. If there is a difference of greater than a volume fraction of 0,2 % CO between the observed and expected values, attention should be given to the analyser linearity.

7.2.4 Recalibrate the instrument at least once a week, using the standard gases. The calibration shall be linear within the limits reported in 5.4.

7.2.5 Check the calibration prior to the measurement using the standard gas containing a volume fraction of about 5 % carbon monoxide. If there is a difference greater than a volume fraction of 0,2 % CO between observed and expected values, repeat the full calibration.

7.3 Smoking and collection of vapour phase

7.3.1 Preparation of vapour-phase collection system

Prepare the system using the instructions pertinent to the equipment fitted.

Ensure that the vapour-phase collecting device has been completely flushed with ambient air and evacuated before the start of the smoking process. There shall not be any residual vacuum upstream of the collection device before smoking.

7.3.2 Smoking procedure

7.3.2.1 Smoke the cigarettes in accordance with the procedure stated in ISO 4387.

7.3.2.2 For linear smoking machines: after completion of smoking each of the first four cigarettes, remove the cigarette butt and take one clearing puff for each trap. After completion of the smoking of all five cigarettes five clearing puffs shall be taken.

7.3.2.3 For rotary smoking machines: after completion of the smoking run, remove the cigarette butts and take five clearing puffs.

7.3.2.4 Record the total number of puffs taken on each channel, i.e. smoking puffs plus clearing puffs.

7.4 Measurement of carbon monoxide volume concentration

7.4.1 Recheck the calibration of the analyser (see 7.2.5) and introduce the vapour phase into the measuring cell of the analyser under the same conditions of ambient temperature and pressure as for sampling and the same gas flow rate as used during calibration. Read the analyser display giving the carbon monoxide concentration. Recalibration may be necessary when the barometric pressure has changed for more than 10 kPa and the CO analyser has no internal compensation.

7.4.2 At the end of each smoking, the vapour-phase collection container shall be emptied. The apparatus is then ready for the next smoking starting at step 7.3.2.1.

8 Expression of results

8.1 Calculation of the average volume of carbon monoxide per cigarette

The average volume of carbon monoxide per cigarette is given by Equation (1):

$$V_{as} = \frac{C \times V \times N \times p \times T_0}{S \times 100 \times p_0 \times (t + T_0)} \quad (1)$$

where

V_{as} is the average volume of carbon monoxide per cigarette, in millilitres;

C is the percentage by volume of carbon monoxide observed;

V is the puff volume, in millilitres;

N is the number of puffs in the measured sample (including clearing puffs);

p is the ambient pressure, in kilopascals;

p_0 is the standard atmospheric pressure, in kilopascals;

S is the number of cigarettes smoked;

T_0 is the temperature for the triple point of water, in Kelvin;

t is the ambient temperature, in degrees centigrade.

In the calculation the following values can be used:

V = 35 ml and rounded values of p_0 (101,3 kPa) and T_0 (273 K).

8.2 Calculation of the average mass of carbon monoxide per cigarette

The average mass of carbon monoxide per cigarette is given by Equation (2):

$$m_{cig} = V_{as} \times \frac{M_{CO}}{V_m} \quad (2)$$

where

m_{cig} is the average mass of carbon monoxide per cigarette, in milligrams;

M_{CO} is the molar mass of carbon monoxide, in grams per mole;

V_m is the molar volume of an ideal gas, in litres per mole.

In the calculation the following values can be used:

Rounded values of M_{CO} (28 g/mol) and V_m (22,4 l/mol).

9 Repeatability and reproducibility

An international collaborative study [1] conducted in 2003, involving 58 laboratories and eight samples (seven commercial brands and the CORESTA monitor test piece CM4), gave the following values for this method.

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

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

The test results were subjected to statistical analysis in accordance with ISO 5725-1 and ISO 5725-2 to give the precision data shown in Table 1.

Table 1 — Estimates given by data analysis

Values in milligrams per cigarette

Mean value m_{cig}	Repeatability limit r	Reproducibility limit R
4,12	0,77	1,11
6,79	0,94	1,49
8,31	0,99	1,75
9,55	0,93	1,71
10,99	1,36	2,25
11,45	1,45	2,13
11,85	1,39	2,36
13,23	1,46	2,31

For the purpose of calculating r and R, one test result was defined as the mean yield obtained from smoking 20 cigarettes in a single run.

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 International Standard 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 listed in 10.2 to 10.5 shall be recorded.

10.2 Characteristic data about the cigarette and cigarette identification

All necessary details to describe the sample fully such as:

a) name of manufacturer;

b) country of manufacture;

c) product name;

d) date of sampling;

e) place of purchase or sampling;

f) kind of sampling point;

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

h) packet number (of that product sampled that day);

i) marks on any tax stamp;

j) printed smoke yields (if any);

k) length of cigarette;

l) length of filter;

m) length of overwrap.

10.3 Sampling

All necessary details to describe the sampling fully such as:

a) type of sampling procedure;

b) number of cigarettes in laboratory sample;

c) date and location of purchase or sampling at manufacturers' premises.

10.4 Description of test

All necessary details to describe the test fully such as:

a) reference to this International Standard, i.e. ISO 8454:2007;

b) date of test;

c) type of smoking machine used;

d) type of analyser used;

e) total number of cigarettes smoked in the entire determination on that cigarette type;

f) number of cigarettes smoked into each collection device;

g) butt length;

h) room temperature (°C) during smoking operation and analysis;

i) relative humidity (%) during smoking operation;

j) atmospheric pressure (kPa) during smoking operation and analysis.

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:

— average length of the cigarettes, average length of the filters, average length of the overwrap, average butt length to which the cigarettes were smoked, average length of tobacco portion smoked, all to the nearest 0,1 mm;

— average diameter of the cigarettes, in millimetres;

— average draw resistance of the conditioned cigarettes;

— average mass, in milligrams per cigarette, of the conditioned cigarettes selected for the smoking operation;

— average number of puffs per cigarette for each channel, to the nearest 0,1 puff;

— average number of total puffs taken for each channel/collection device, including final five clearing puffs, to the nearest 0,1 puff;

— observed carbon monoxide concentration, expressed as a percentage by volume, for each channel, to the nearest 0,01 %, and the average per cigarette, to the nearest 0,1 %;

— amount of carbon monoxide determined, in milligrams per cigarette for each channel, to the nearest 0,1 mg, and the average per cigarette, to the nearest 1 mg.

Bibliography

[1] CORESTA Report, *CORESTA study for the determination of repeatability and reproducibility of the measurement of nicotine-free particulate matter, nicotine and CO in smoke using the ISO smoking methods*; October 2003

[2] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[3] 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*

ISO 8454:2007(E)

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Nederlandse norm

NEN-ISO 8454/A1

(en)

Cigarettes - Determination of carbon monoxide in the vapour phase of cigarette smoke - NDIR method (ISO 8454:2007/Amd 1:2009,IDT)

ICS 65.160

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Als Nederlands wijzigingsblad is aanvaard:

- ISO 8454:2007/Amd 1:2009,IDT

Normcommissie 370126 "Tabak en tabakproducten"

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Third edition
2007-06-01

AMENDMENT 1
2009-10-15

Cigarettes — Determination of carbon monoxide in the vapour phase of cigarette smoke — NDIR method

AMENDMENT 1

Cigarettes — Dosage du monoxyde de carbone dans la phase gazeuse de la fumée de cigarette — Méthode IRND

AMENDEMENT 1

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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.

Amendment 1 to ISO 8454:2007 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

Cigarettes — Determination of carbon monoxide in the vapour phase of cigarette smoke — NDIR method

AMENDMENT 1

New page iv

Introduction

No machine smoking regime can represent all human smoking behaviour:

— it is recommended that cigarettes also be tested under conditions of a different intensity of machine smoking than those specified in this International Standard;

— machine smoking testing is useful to characterize cigarette emissions for design and regulatory purposes, but communication of machine measurements to smokers can result in misunderstandings about differences in exposure and risk across brands;

— smoke emission data from machine measurements may be used as inputs for product hazard assessment, but they are not intended to be nor are they valid as measures of human exposure or risks. Communicating differences between products in machine measurements as differences in exposure or risk is a misuse of testing using ISO standards.

ISO 8454:2007/Amd.1:2009(E)

ICS 65.160

Price based on 1 page

Nederlandse norm

NEN-ISO 10315

(en)

Sigaretten - Bepaling van het gehalte aan nicotine in rookcondensaten - Gaschromatografische methode (ISO 10315:2014(Cor.2014-11),IDT)

Cigarettes - Determination of nicotine in smoke condensates - Gas-chromatographic method (ISO 10315:2014(Cor.2014-11),IDT)

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Cigarettes — Determination of nicotine in smoke condensates — Gas-chromatographic method

Cigarettes — Dosage de la nicotine dans les condensats de fumée — Méthode par chromatographie en phase gazeuse

Reference number
ISO 10315:2013(E)

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Foreword

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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 10315 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

This third edition cancels and replaces the second edition (ISO 10315:2000), which has been technically revised. It also incorporates the Amendment ISO 10315:2000/Amd 1:2011 and the Technical Corrigendum ISO 10315:2000/Cor 1:2000.

This corrected version of ISO 10315:2013 incorporates the following correction:

— the wording of 4.2 has been clarified.

Introduction

This International Standard may be considered as part of a set produced by ISO/TC 126 which describes the determination of total and nicotine-free dry particulate matter (NFDPM) in cigarette smoke condensates. The set comprises:

ISO 3308, ISO 3402, ISO 4387, ISO 8243, ISO 10315, and ISO 10362-1.

A related International Standard, ISO 3400, determines total alkaloids, whereas this International Standard determines only nicotine by virtue of the gas-chromatographic separation. Occasionally, differences can occur because of minor amounts of alkaloids other than nicotine in some types of tobacco.

Annex A provides information about the use of this method in conjunction with or simultaneously with the gas-chromatographic method of water determination specified in ISO 10362-1.

A bibliography is provided.

No machine smoking regime can represent all human smoking behaviour:

— it is recommended that cigarettes also be tested under conditions of a different intensity of machine smoking than those specified in this International Standard;

— machine smoking testing is useful to characterize cigarette emissions for design and regulatory purposes, but communication of machine measurements to smokers can result in misunderstandings about differences in exposure and risk across brands;

— smoke emission data from machine measurements may be used as inputs for product hazard assessment, but they are not intended to be nor are they valid as measures of human exposure or risks. Communicating differences between products in machine measurements as differences in exposure or risk is a misuse of testing using ISO standards.

INTERNATIONAL STANDARD ISO 10315:2013(E)

Cigarettes — Determination of nicotine in smoke condensates — Gas-chromatographic method

WARNING — The use of this International Standard can involve hazardous materials, operations, and equipment. This International Standard does not purport to address all the safety problems associated with its use. It is the responsibility of the user of this International Standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

1 Scope

This International Standard specifies a method for the gas-chromatographic determination of nicotine in cigarette smoke condensates. The smoking of cigarettes and the collection of mainstream smoke are normally carried out in accordance with ISO 4387.

NOTE 1 The method specified in this International Standard is also applicable to the determination of nicotine in cigarette smoke condensates obtained by non-standard smoking.

NOTE 2 In countries not in a position to use the gas-chromatographic method, reference should be made to ISO 3400 for the determination of total nicotine alkaloids. In such cases, values obtained using the method described in ISO 3400 may be used with the addition of a note in the expression of results.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

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

3 Principle

The smoke condensate from the mainstream smoke is dissolved in a solvent containing an internal standard. The nicotine content of an aliquot of the solution is determined by gas chromatography, and the nicotine content of the whole of the smoke condensate is calculated.

4 Reagents

Use only reagents of recognized analytical reagent grade.

4.1 Carrier gas: helium (CAS: 7440-59-7) or nitrogen (CAS: 7727-37-9) of high purity (at least 99,999 %).

4.2 Auxiliary gases: hydrogen (CAS: 1333-74-0) of high purity (at least 99,999 %) and air for the flame ionization detector.

4.3 Propan-2-ol (CAS: 67-63-0), with maximum water content of 1,0 mg/ml.

4.4 Internal standard: *n*-heptadecane (CAS: 629-78-7) or quinaldine (CAS: 91-63-4) of purity not less than 99 %.

Carvone (CAS: 99-49-0), *n*-octadecane (CAS: 593-45-3), or other appropriate internal standards may be used after assessment of their purity and determination that the internal standard does not co-elute with other components in the smoke extract. The peak area of the internal standard on samples should be monitored for consistency. In cases where inconsistencies are found, analysis of an extraction of a smoke sample without the internal standard in the extraction solution should be performed to confirm the absence of a peak in the smoke extract eluting at the same time as the internal standard (see Clause 9).

4.5 Extraction solvent: propan-2-ol (4.3) containing an appropriate concentration of internal standard (4.4); this is normally in the range of 0,2 mg/ml to 0,5 mg/ml.

Solvent not stored in a temperature-controlled laboratory shall be allowed to equilibrate to (22 ± 2) °C before use.

4.6 Reference substance: nicotine (CAS: 54-11-5) of known purity not less than 98 %.

Store this at between 0 °C and 4 °C and exclude light.

Nicotine salicylate (CAS: 29790-52-1) of known purity not less than 98 % may also be used.

NOTE The purity of the nicotine or nicotine salicylate may be verified in accordance with ISO 13276 or by any other validated method.

4.7 Calibration solutions

Dissolve the nicotine (4.6) in the solvent (4.5) to produce a series of at least four calibration solutions with concentrations that cover the range expected to be found in the test portion (usually 0,02 mg/ml to 2,0 mg/ml). Store these solutions at between 0 °C and 4 °C and exclude light.

Solvent and solutions stored at low temperatures shall be allowed to equilibrate to (22 ± 2) °C before use.

5 Apparatus

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

5.1 Gas-chromatograph, equipped with a flame ionization detector, recorder, and integrator or other suitable data handling instrument (see Clause 9).

5.2 Column, of internal diameter between 2 mm and 4 mm and preferably of length 1,5 m to 2 m.

The column is preferably made of glass, but other materials such as deactivated stainless steel or nickel may be used. Stationary phase: 10 % poly(ethylene glycol) (PEG) 20 000 plus 2 % potassium hydroxide on an acid-washed silanized support material, 150 µm (100 mesh) to 190 µm (80 mesh) (see also Clause 9).

6 Procedure

6.1 Test portion

Prepare the test portion by dissolving the smoke condensate obtained by the machine smoking of a known number of cigarettes in a fixed volume of the solvent (4.5) of 20 ml for 44 mm discs, or 50 ml for 92 mm discs, ensuring that the disc is fully covered. The volume may be adjusted to give a concentration of nicotine appropriate for the calibration graph (see 6.3) provided that there is adequate volume for effective extraction of the smoke condensate. Analysis should be performed as soon as possible, but if storage is inevitable then store the sample at between 0 °C and 4 °C and exclude light. For standard smoking, refer to ISO 4387.

6.2 Setting up the apparatus

Set up the apparatus and operate the gas chromatograph (5.1) in accordance with the manufacturer's instructions. Ensure that the peaks for solvent, internal standard, nicotine, and other smoke component peaks, especially neophytadiene (which can appear on the tail of the nicotine peak under certain circumstances), are well resolved (see also Clause 9).

Suitable operating conditions are as follows:

— column temperature, 170 °C (isothermal);

— injection temperature, 250 °C;

— detector temperature, 250 °C;

— carrier gas, helium, or nitrogen at a flow rate of about 30 ml/min;

— injection volume, 2 µl.

Using the above conditions, the analysis time is about 6 min to 8 min (see also Clause 9).

6.3 Calibration of the gas chromatograph

Inject an aliquot (2 µl) of each of the calibration solutions (4.7) into the gas chromatograph. Record the peak areas (or heights) of the nicotine and internal standard (4.4). Carry out the determination at least twice.

Calculate the ratio of the nicotine peak to the internal standard peak from the peak area (or height) data for each of the calibration solutions. Plot the graph of the nicotine concentrations in accordance with the area ratios, and calculate a linear regression equation (concentration of nicotine according to the area ratios) from these data. The graph should be linear and the regression line should pass through the origin. Use the slope of the regression equation.

Perform this full calibration procedure daily. In addition, inject an aliquot of an intermediate concentration standard after every 20 sample determinations. If the calculated concentration for this solution differs by more than 3 % from the original value, repeat the full calibration procedure.

6.4 Determination

Inject aliquots (2 µl) of the test portion (see 6.1) into the gas chromatograph. Calculate the ratio of the nicotine peak/internal standard peak from the peak area (or height) data.

Carry out two determinations on the same test portion (see 6.1).

Calculate the mean value of the ratio from the two determinations.

Where results are obtained from a number of separate channels of smoking and where an auto-sampler is used, a single aliquot portion from the smoke traps is considered adequate.

7 Expression of results

Calculate the concentration of nicotine in the test portion using the graph or linear regression equation prepared in 6.3. From the concentration of nicotine in the test portion, calculate the amount of nicotine in the smoke condensate. Deduce the amount in the cigarettes smoked. Express the test results in milligrams per cigarette, m_N, for each channel to the nearest 0,01 mg and the average per cigarette to the nearest 0,1 mg.

8 Repeatability and reproducibility

A major international collaborative study involving 30 laboratories and 6 samples, conducted in 1990, showed that when cigarettes are smoked in accordance with ISO 4387 and the resulting smoke solutions

are analysed by this method, the following values for the repeatability limits (r) and the reproducibility limits (R) are obtained.

The difference between two single results found on matched cigarette samples by one 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 cigarette samples reported by two laboratories will differ by more than the reproducibility limit (R) on average not more than one in 20 cases in the normal and correct operation of the method.

Data analysis gave the estimates as summarized in Table 1.

Table 1 — Estimates given by data analysis

Mean value m_N mg per cigarette	Repeatability limit r mg per cigarette	Reproducibility limit R mg per cigarette
0,091	0,040	0,069
0,179	0,046	0,069
0,326	0,050	0,076
0,673	0,077	0,109
0,835	0,079	0,142
1,412	0,107	0,195

For the purpose of calculating r and R, one test result was defined as the mean yield obtained from smoking 20 cigarettes in a single run.

For further details of the interaction of r and R with other factors, see CORESTA Report 91/1.

The subject of tolerances due to sampling is dealt with in ISO 8243.

9 Alternative gas-chromatographic procedures and analysis precautions

9.1 General

Alternative gas-chromatographic columns, both packed and capillary, have been found suitable for the determination of nicotine in smoke condensate. If these are used, it is necessary to ensure that the peaks due to nicotine and the internal standard are well resolved from peaks due to other smoke components and the solvent.

The data in Clause 8 refer to the reference column. Appropriate data for these alternative procedures are not yet available.

9.2 Alternative columns

9.2.1 Packed columns

The following may be used as alternative stationary phases in the column described in 5.2:

— 2 % Versamid 900[1)] plus 1 % potassium hydroxide, or

— 7 % PEG 20 000 plus 3 % polyphenyl ether (6 rings), or

1) These are trade names of examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

— lower loadings of PEG 20 000 (with or without potassium hydroxide).

9.2.2 Capillary columns

Fused silica capillary columns (0,2 mm to 0,53 mm ID) with a thin film thickness equal to or less than 1 µm, capable of analysing polar compounds, may be used.

Base-deactivated PEG stationary phases, such as CAM (J and W Scientific)[2], Carbowax-amine (Supelco)[2], Stabilowax-DB (Restek)[2], and CP WAX-51 (Chrompack)[2], give similar data to the PEG 20 000 plus potassium hydroxide packed column in 9.2.1.

NOTE Hydrogen is an alternative carrier gas if a capillary column is used.

9.3 Injection systems

The alternative columns described in 9.2.1 and 9.2.2 require the use of purpose-made injection systems. Suitable operating conditions may vary depending on the type of column used and they may need to be optimized following the manufacturer's instructions. Isothermal oven temperature or oven temperature programming, hold times, carrier gas and linear velocity and split ratio shall be set for the type of capillary column used. For example, for a 15 m, 0,32 mm ID, 0,25 µm film thickness capillary column, typical conditions might be as follows:

— oven temperature 160 °C (hold 4,5 min) rising to 200 °C at 30 °C/min (hold 1,5 min);

— carrier gas helium at a linear flow rate of about 25 cm/s;

— split ratio 20:1.

Using the above conditions, the analysis time is about 7 min to 8 min.

9.4 Alternative internal standards

Alternative internal standards have also been evaluated. These are carvone, quinaldine, and *n*-octadecane. These may be used after assessment of their purity and a check to ensure that they do not co-elute with other smoke components in the smoke extract being analysed. The peak area of the internal standard on samples should be monitored for consistency.

Where inconsistencies are found, analysis of a smoke sample without an internal standard in the extraction solution should be performed to confirm the absence of a peak in the smoke extract eluting at the same time as the internal standard.

10 Test report

The test report shall state the yield of nicotine per cigarette smoked and the method used and shall include all conditions which may affect the result (e.g. atmospheric test conditions during smoking). It shall also give all details necessary for the identification of the cigarettes smoked.

2) These are trade names of examples of suitable products available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of these products.

Annex A
(informative)

Use of this method with the gas-chromatographic determination of water

This method may be used in conjunction with, or simultaneously with, the gas-chromatographic method of water determination in smoke condensates specified in ISO 10362-1. This may be carried out by:

— the addition of an appropriate quantity of the internal standard specified for the water determination in the solvent described in 4.5;

— the use of helium, preferably, as the carrier gas;

— injection of an aliquot of the smoke condensate solution onto a column for water analysis, which is connected to a thermal conductivity detector, as well as onto the nicotine column and detector described in this method.

A simultaneous automated analysis of nicotine and water may be achieved by using a splitting system or an auto-sampler with two injection positions. When determining nicotine and water from the same sample sequentially, the water determination is performed first to prevent absorption of water by the sample affecting the final result.

NOTE When simultaneous determination of nicotine and water is performed, the described shelf lives of calibration solutions and test portions can be affected. The shortest shelf life mentioned in one of the methods used should be applied.

Bibliography

[1] ISO 3308, *Routine analytical cigarette-smoking machine — Definitions and standard conditions*

[2] ISO 3400, *Cigarettes — Determination of alkaloids in smoke condensates — Spectrometric method*

[3] ISO 3402, *Tobacco and tobacco products — Atmosphere for conditioning and testing*

[4] ISO 8243, *Cigarettes — Sampling*

[5] ISO 10362-1, *Cigarettes — Determination of water in smoke condensates — Part 1: Gas-chromatographic method*

[6] ISO 13276, *Tobacco and tobacco products — Determination of nicotine purity — Gravimetric method using tungstosilicic acid*

[7] REPORT CORESTA 91/1, *Information Bulletin of Cooperation Centre for scientific research relative to tobacco,* 1991-1 (ISSN-0525-6240)

ISO 10315:2013(E)

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Normen zijn afspraken voor en door de markt, zo ook deze norm. NEN begeleidt het gehele normalisatieproces. Van het bijeenbrengen van partijen, het maken en vastleggen van de afspraken en het bieden van hulp bij de toepassing van de normen. Om deze diensten te kunnen bekostigen betalen alle belanghebbende partijen die aan tafel zitten voor het normalisatieproces, en u als gebruiker voor normen en trainingen. NEN is een stichting en heeft geen winstoogmerk.

Wat is nu precies de toegevoegde waarde van normen?
Stelt u zich eens voor ... u wilt in het buitenland geld pinnen, maar uw bankpas past niet. Of uw nieuwe telefoon herkent uw simkaart niet. De samenstelling van de benzine over de grens is anders waardoor u niet kunt tanken. Het dagelijks leven zou zonder goede afspraken over producten, processen en diensten een stuk complexer zijn.

Het maken en vastleggen van afspraken door belanghebbende partijen noemen we het normalisatieproces. Normalisatie had vanouds betrekking op techniek en producten. Nu worden steeds vaker normen voor diensten ontwikkeld. Zo zijn er afspraken op het gebied van gezondheidszorg, schuldhulpverlening, kennisintensieve dienstverlening, externe veiligheid en MVO.

Normen zorgen voor verbetering van producten, diensten en processen; qua veiligheid, gezondheid, efficiëntie, kwaliteit en duurzaamheid. Dit ziet u op de werkvloer, in de omgang met elkaar en in de samenleving als geheel. Organisaties die normalisatie onderdeel van hun strategie maken, vergroten hun professionaliteit, betrouwbaarheid en concurrentiekracht.

Wat doet NEN?
NEN ondersteunt in Nederland het normalisatieproces. Als een partij zich tot NEN richt met de vraag om een afspraak tot stand te brengen, gaan wij aan de slag. We onderzoeken in hoeverre normalisatie mogelijk is en er interesse voor bestaat. Wij nodigen vervolgens alle belanghebbende partijen uit om deel te nemen. Een breed draagvlak is een randvoorwaarde. De afspraken komen op basis van consensus tot stand en worden vastgelegd in een document. Dit is meestal een norm. Afspraken die in een NEN-norm zijn vastgelegd mogen niet conflicteren met andere geldige NEN-normen. NEN-normen vormen samen een coherent geheel. Een belanghebbende partij kan een producent, ondernemer, dienstverlener, gebruiker, maar ook de overheid of een consumenten- of onderzoeksorganisatie zijn. De vraag is niet altijd om een norm te ontwikkelen. Vanuit de overheid komt regelmatig het verzoek om te onderzoeken of er binnen een bepaalde sector of op een bepaald terrein normalisatie mogelijk is. NEN doet dan onderzoek en start afhankelijk van de uitkomsten een project. Deelname staat open voor alle belanghebbende partijen. NEN beheert ruim 30.000 normen. Dit zijn de in Nederland aanvaarde internationale (ISO, IEC), Europese (EN) en nationale normen (NEN). In totaal zijn er ruim 800 normcommissies actief met in totaal bijna 5.000 normcommissieleden. Een goed beheer van de omvangrijke normencollectie en de afstemming tussen nationale, Europese en internationale normcommissies vereisen dan ook een zeer goede infrastructuur.

Betalen kleine organisaties net zoveel als grote organisaties?
Het uitgangspunt is dat alle partijen die deelnemen aan het normalisatieproces een evenredig deel betalen. De normcommissieleden kunnen onderling andere afspraken maken. Zo worden er wel eens afspraken gemaakt dat de grote partijen een groter deel betalen dan de kleinere bedrijven. De prijzen voor normen zijn voor iedereen gelijk. De kosten voor licenties zijn afhankelijk van de omvang van een organisatie en het aantal gebruikers.

Voordelen van normalisatie en normen
Gegarandeerde kwaliteit | Veiligheid geborgd | Bevordert duurzaamheid | Opschalen en vermarkten van nieuwe innovatieve producten | Meer (internationale) handelsmogelijkheden | Verhoogde effectiviteit en efficiëntie | Onderscheidend in de markt.

Voordelen van deelname
Invloed op de (internationale en Europese) afspraken | Als eerste op de hoogte van veranderingen | Netwerk; ook op Europees en internationaal niveau | Kennisvergroting.