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Cigarettes — Determination of nicotine-free dry particulate matter and nicotine in sidestream smoke — Method using a routine analytical linear smoking machine equipped with a fishtail chimney

Cigarettes — Détermination de la matière particulaire anhydre et exempte de nicotine et de la nicotine dans le courant secondaire de fumée — Méthode utilisant une machine à fumer analytique de routine linéaire équipée de cheminées individuelles en forme de queue de poisson

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Foreword

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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 20773 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

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 and this results in a final product which is intrinsically variable. Further complexity arises as the cigarette is converted to cigarette smoke during smoking.

The quantitative measurement of nicotine, of particulate matter and of nicotine-free dry particulate matter (NFDPM, sometimes referred to as “tar”) is therefore dependent on the arbitrary definition of the means used to generate and collect the smoke. In particular, the ambient conditions (e.g. temperature, humidity, air movement within the laboratory) under which the test pieces are conditioned and smoke is collected, play a critical role in the accuracy of the measurement.

Sidestream smoke in this International Standard is understood to be the smoke that is evolved from the cigarette during the smoking run other than from the mouth end.

NOTE This is distinguished from environmental tobacco smoke (ETS), which is a mixture of aged and diluted exhaled mainstream smoke and aged and diluted sidestream smoke, and for the assessment of which the present method does not apply.

From the time that scientists first attempted to determine nicotine and total and dry particulate matter yields in sidestream smoke, many different methods have been adopted. However, experience has shown some procedures to be more reliable and more amenable to handling of large numbers of samples. With these factors in mind, during the 1999–2002 period, collaborative studies by a task force composed of CORESTA members have shown that improvements in repeatability and reproducibility result when some restrictions are placed upon the wide variety of methods and practices described in existing methods.

This International Standard, produced after much collaborative experimentation by many laboratories in many countries, reflects the results of the optimization proposed and validated by the task force and provides one set of procedures that are the accepted reference procedures and for which repeatability and reproducibility of the determinations were assessed. Experience in the task force has shown how strict adherence to the detailed set up and conditions of the method, as well as the degree of proficiency of the operator, affect the precision of the results.

Further, it is preferable that the selected method be compatible with different modes of cigarette equilibration or puffing parameters for the smoking of the tested pieces. The standards defined by ISO for the determination of mainstream yields were, however, followed to the largest possible extent, although the machines used by the different laboratories were all of a linear type.

This method is a machine method and it allows cigarettes to be smoked using a strictly controlled set of parameters. Thus it enables the sidestream NFDPM and nicotine from cigarettes, when smoked by this procedure, to be compared and ranked. In the course of its studies, the task force demonstrated the value of comparing the analytical processes and their stability by use of the CORESTA monitor test piece for determining NFDPM and nicotine yields.

Since the determinations of NFDPM and nicotine in sidestream smoke are by nature more complex and delicate than their counterparts performed on mainstream smoke, it is highly recommended to include a control test piece in the smoking plans, as is done in mainstream determinations. It is possible to use the CORESTA monitor or any other internally designed control cigarette for this purpose. The use of an internationally recognized monitor test piece is recommended.

Cigarettes — Determination of nicotine-free dry particulate matter and nicotine in sidestream smoke — Method using a routine analytical linear smoking machine equipped with a fishtail chimney

1 Scope

This International Standard is applicable to the determination of total particulate matter and to the subsequent determination of nicotine and nicotine-free dry particulate matter present in the sidestream smoke from cigarettes. The described method is specified using the ISO 3308 smoking parameters (puff volume, duration and frequency) and butt length, but it is technically compatible with other smoking regimes.

NOTE The method may not be directly applicable to other sidestream smoke analytes.

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 2971, *Cigarettes and filter rods — Determination of nominal diameter — Method using a laser beam measuring apparatus*

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*

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

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

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

3 Terms and definitions

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

3.1
total sidestream particulate matter
crude sidestream smoke condensate
portion of the sidestream smoke which is trapped on the sidestream pad, together with that portion of the sidestream smoke which condenses on the wall of the fishtail chimney, expressed as milligrams per cigarette

3.2
dry sidestream particulate matter
dry sidestream smoke condensate
dry sidestream particulate matter, expressed as milligrams per cigarette, and composed of the sum of the total particulate matter trapped on the sidestream pad after deduction of its water content, plus the estimated nicotine-free dry particulate matter condensed on the walls of the sidestream chimney (this estimate being obtained by a UV absorption method described below), plus the nicotine condensed on the walls of the sidestream chimney

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

3.4
sidestream nicotine
sum of the nicotine condensed on the walls of the fishtail chimney, the nicotine collected on the sidestream pad and the nicotine collected in the impinger trap, expressed as milligrams per cigarette

3.5
smoking process
use of a smoking machine to smoke cigarettes from lighting to final puff

3.6
smoking run
specific smoking process to produce such sidestream smoke from a sample of cigarettes as is necessary for the determination of the smoke components

3.7
laboratory sample
sample intended for laboratory inspection or testing and which is representative of the gross sample or the sub-period sample

3.8
conditioning sample
cigarettes selected from the test sample for conditioning prior to tests for sidestream particulate matter and nicotine yield

3.9
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

3.10
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

3.11
conditioned sample
conditioned cigarettes smoked for sidestream particulate matter and nicotine tests

4 Principle

— Sampling of the test cigarettes.

— Conditioning of the test cigarettes.

— Smoking of the test cigarettes on a smoking machine in accordance with ISO 3308, with the exception of the specifications on air velocity control, and equipped with a fishtail chimney, a glass-fibre filter pad and an impinger trap for each channel.

— Simultaneous collection of total sidestream particulate matter on the walls of the fishtail chimney and in a glass-fibre filter pad, and collection of vapour phase sidestream nicotine in an impinger trap.

— Gravimetric determination of the mass of total sidestream particulate matter collected on the glass-fibre filter pad.

— Extraction of the total sidestream particulate matter from the glass-fibre filter pad for the determination of water and nicotine contents by gas chromatography.

— Estimation of the nicotine-free dry sidestream particulate matter condensed on the walls of the fishtail chimney by a UV absorbance method.

— Analytical determination by gas chromatography of the water collected in the glass-fibre filter pad.

— Analytical determination by gas chromatography of the nicotine collected in the fishtail chimney, the glass-fibre filter pad and the impinger trap.

NOTE In the countries that are not in a position to use the gas-chromatographic methods, reference should be made to ISO 3400 for the determination of total alkaloids, and the determination of water in smoke condensate should be performed by the Karl Fischer method given in ISO 10362-2. In such cases values obtained for nicotine and water in smoke condensate can be used with the addition of a note made in the expression of the result.

5 Apparatus

Usual laboratory apparatus and in particular the following items.

5.1 Fishtail chimney[1)], manufactured in glass, of design and dimensions shown in Figure 1.

Dimensions in millimetres

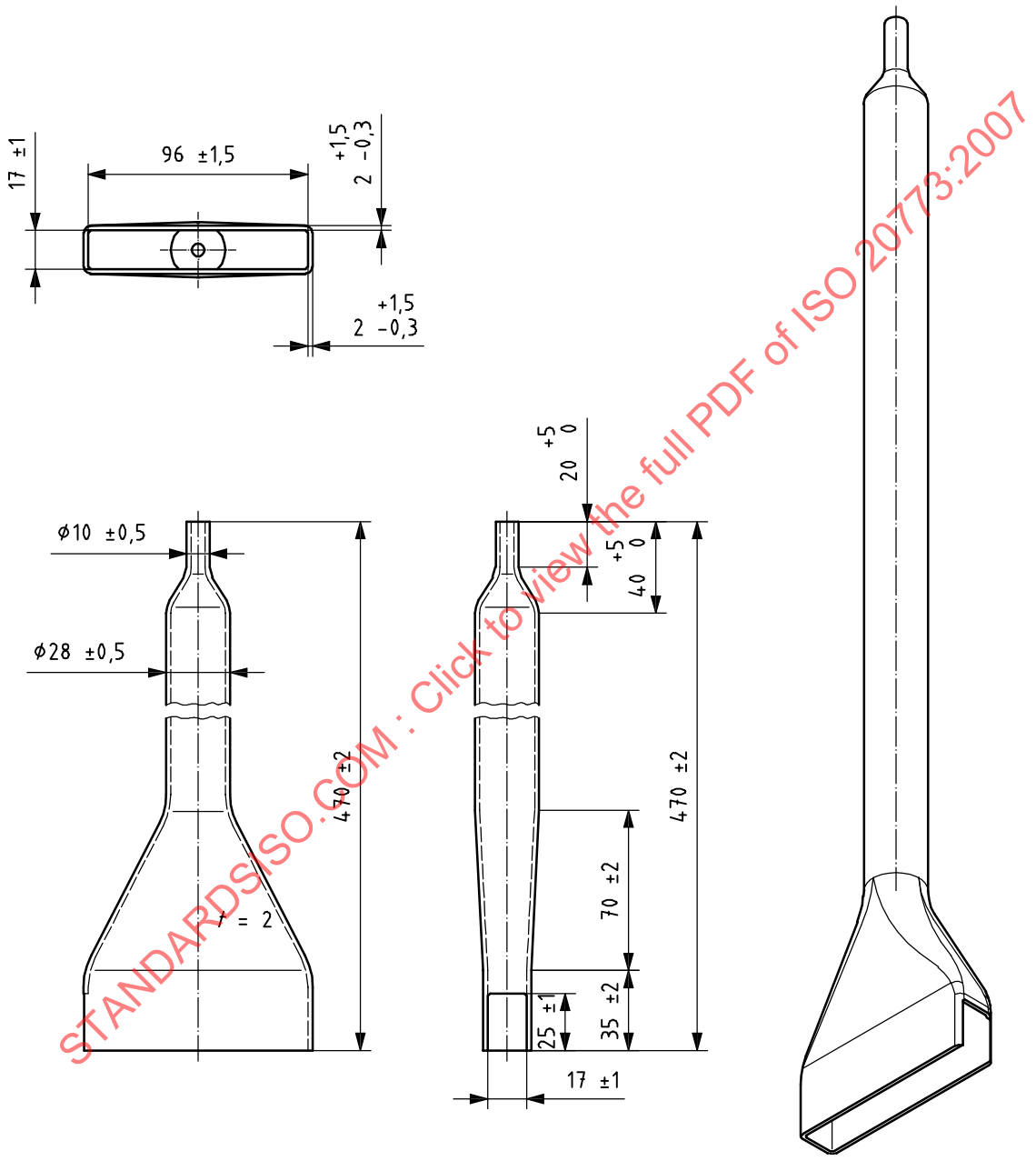
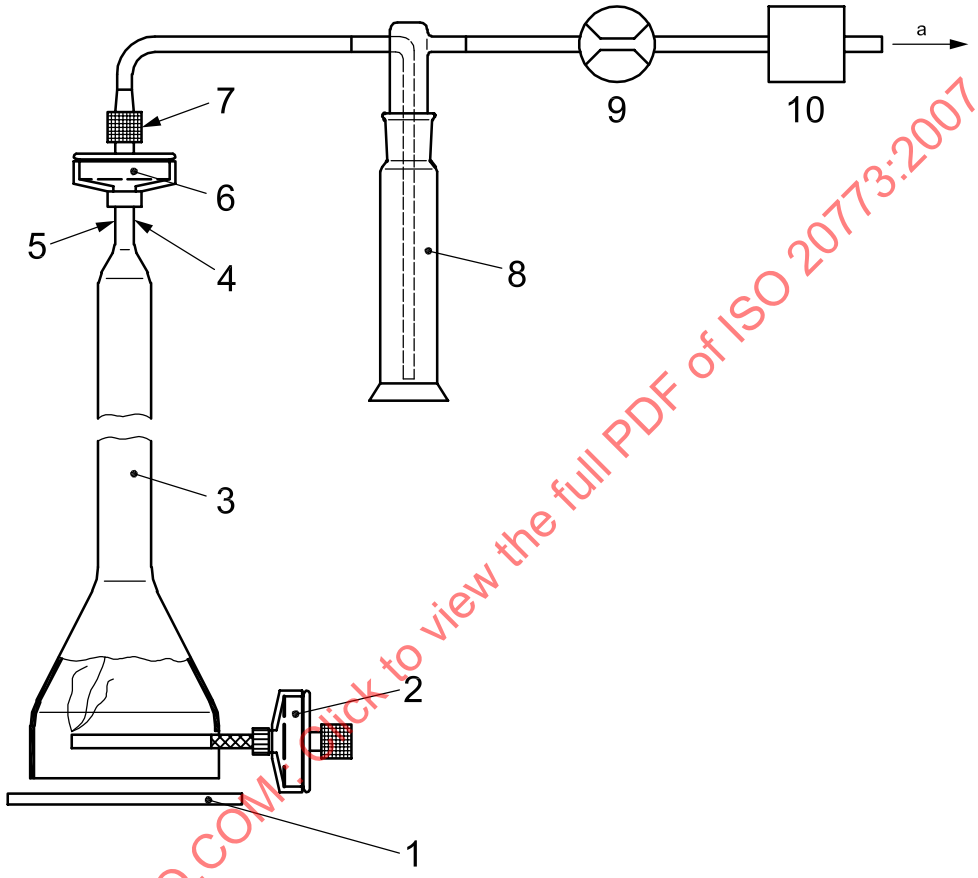


Figure 1 — Fishtail chimney dimensions

1) These fishtail chimneys are commercially available and may be obtained from Borgwaldt Technik GmbH and Cerulean. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of the product named. Equivalent products may be used if they can be shown to lead to the same results.

5.2 Routine analytical cigarette-smoking machine, modified to accept fishtail chimneys and complying with the requirements of ISO 3308 with the exception of the specifications on air velocity control. A plate shall be fixed underneath each channel, with a minimum length of 120 mm and a minimum width of 50 mm. This plate is positioned so as to cover the totality of the opening at the fishtail chimney bottom, as shown in Figure 2.

5.3 Impinger traps, Drechsel gas-washing bottle, 100 ml or 125 ml, with open-ended stem. An example is shown in Figure 2.



Key

1 horizontal plate
2 mainstream smoke trap and cigarette holder
3 fishtail chimney
4 location of calibration flow measurement
5 pressure and vacuum tubing
6 sidestream smoke trap
7 quick connect
8 Drechsel gas washing bottle
9 flow meter
10 flow regulator

[a] To pump.

Figure 2 — Sidestream smoke collection system with impringer in place

5.4 Vacuum pump or **pumps and flow control devices**, capable of maintaining an air flow of 3 l/min through each fishtail chimney and collection train.

5.5 PVC tubing, of approximately 8 mm inside diameter, 11 mm outside diameter, to connect the sidestream trap, impinger, in-line flow meter, flow regulator and vacuum pump.

5.6 Flow monitoring and regulating system on each channel, comprising an in-line continuous-reading flow meter, capable of monitoring the flow with a resolution of 0,2 l/min, followed by a precision flow-regulating device.

5.7 Primary flow meter, capable of accurately measuring a flow-rate of 3 l/min with an accuracy of 0,1 l/min, to be used in setting the air flow in each fishtail chimney before a smoke run. As this is a primary measurement, the flow meter should measure the time needed to flush a known volume.

5.8 Soap bubble flow meter or **alternative displacement flow meter**, capable of measuring a displaced volume of at least the desired puff volume, with an accuracy of ± 0,2 cm^3 and a resolution of 0,1 cm^3.

5.9 Apparatus for the determination of puff duration and frequency.

5.10 Analytical balance, with a resolution of 0,1 mg.

5.11 Draw resistance testing equipment, as specified in ISO 6565.

5.12 Conditioning enclosure, carefully maintained in accordance with the conditions specified in ISO 3402.

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

5.14 Apparatus for the determination of diameter, in accordance with ISO 2971.

5.15 Filter holder sealing device, with end caps made from the same non-hygroscopic and chemically inert material as the filter holder.

5.16 Gloves, cotton or non-talc surgical.

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

5.18 Laboratory shaker, capable of shaking at about 3,3 s^{-1} (200 rpm).

5.19 UV spectrophotometer, preferably equipped with a batch sampler.

6 Reagents

Use only reagents of recognised analytical reagent grade.

6.1 Propan-2-ol, with a maximum water content of 1,0 mg/ml.

6.2 Internal standard for nicotine analysis, *n*-heptadecane or quinaldine (of purity of at least 99 %).

NOTE Carvone, *n*-octadecane or other appropriate internal standards can be used after assessment of their purity and determination that the internal standard does not co-elute with other components in the smoke extract, as described in ISO 10315.

6.3 Internal standard for water analysis, dried ethanol or methanol (purity of at least 99 %).

6.4 Extraction solution, propan-2-ol (6.1) containing an appropriate concentration of internal standards; this is normally in the range of approximately 0,2 mg/ml to 0,5 mg/ml for nicotine and 3,75 ml/l to 5 ml/l for water.

Solvent not stored in a temperature-controlled laboratory shall be allowed to equilibrate to (22 ± 2) °C before use. To prevent water being absorbed, the bulk solvent container shall be fitted with a water trap and all solutions shall be kept sealed. The solvent shall be stirred continuously to ensure the homogeneity of the water concentration in the solvent.

6.5 Reference substance for nicotine, nicotine of known purity and verified in accordance with ISO 13276. Store this at between 0 °C and 4 °C and exclude light. Nicotine salicylate of known purity and verified in accordance with ISO 13276 may also be used.

6.6 Reference substance for water, distilled or deionized water.

6.7 Calibration solutions for nicotine and water in the sidestream glass-fibre filter pads.

6.7.1 Preparation of calibration solution for nicotine.

Dissolve the nicotine (6.5) in the extraction solution (6.4) to prepare a series of at least four calibration standards for nicotine, whose concentrations cover the range expected to be found in the test portion (usually 0,2 mg/ml to 0,8 mg/ml).

6.7.2 Preparation of calibration solution for water.

Prepare a series of at least five calibration standards whose concentrations of added water cover the range expected to be found in the test portion (usually 0,2 mg/ml to 0,8 mg/ml) by adding weighed amounts of water (6.6) to the extraction solution (6.4). One of these calibration solutions shall be extraction solution with no added water (solvent blank).

The calibration solutions shall be made up using an extraction solution from the same batch used for the test portion extraction. It is recommended that water calibration solutions be made up at least each week.

6.8 Calibration solutions for chimney and impinger nicotine.

Prepare fresh with each batch of extraction solution (6.4).

Prepare a series of at least five calibration standards for nicotine whose concentrations cover the range expected to be found in the chimney and impinger test portions (0,01 mg/ml to 0,2 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.

7 Sampling and preparation of cigarettes

7.1 General

Provide a laboratory sample (see 3.7), by using a suitable sampling scheme. Guidance may be found in ISO 8243. The sample will normally contain cigarettes taken from different parts of the population. Make up the test sample required for the test by randomly selecting cigarettes from the different parts of the population represented in the laboratory sample.

7.2 Symbols

In 7.3, 7.4 and 8.1, the symbols listed in Table 1 are used.

Table 1 — Symbols used

Symbol	Variable
N	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	Multiplying factor, value greater than 1, to allow for loss due to damage or selection procedures between initial sampling and smoking.
n	Number of replicate determinations of total sidestream particulate matter.
q	Number of cigarettes smoked into the same sidestream trap.
P	Total number of packets of cigarettes available.
Q	Total number of cigarettes available (laboratory sample, see 3.7).

7.3 Preparation of the cigarettes for smoking

If N cigarettes of a given type are to be smoked, $C \times N$ cigarettes should be prepared from Q for conditioning and butt marking. The multiplier C is usually at least 1,2 to provide extra cigarettes in case some are damaged. If selection by mass or draw resistance (or any other parameter) is necessary, C will have to be much larger (experience suggests 2,0 to 4,0) depending on the selection process.

NOTE The precision data given in this method are based on eight replicates of three cigarettes. Any reduction in the number of replicates will affect the precision. It is not recommended to smoke less than five replicates.

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.

7.4 Selection of test portions of cigarettes

7.4.1 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 should be selected at random so that every cigarette has an equal probability of being chosen.

7.4.2 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

$$Q \times k/P \geqslant C \times q$$

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.

7.4.3 Duplicate test portions

Provided that the test sample is sufficiently large ($\geqslant 2\ C \times N$), it would be prudent to reserve a duplicate set of n test portions. In this event the parallel selection of a test portion and its duplicate would seem sensible. In this case the two selection conditions of 7.4.2 would need to be changed to:

$$Q/P \geqslant 2\ C \times q \text{ and } Q/P < 2\ C \times q$$

7.5 Marking the butt length

7.5.1 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;

— 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 The butt length is defined in ISO 3308 as the length of unburnt cigarette remaining at the moment when smoking is stopped.

7.5.2 Measurement of length of filter

The length of filter as defined in 7.5.1 shall be the mean value of 10 cigarettes taken from the laboratory sample measured to an accuracy of 0,5 mm. The mean shall be expressed 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.

7.5.3 Measurement of length of overwrap

The length of overwrap as defined in 7.5.1 shall be the mean value of 10 overwraps taken from the laboratory sample measured to an accuracy of 0,5 mm. The mean shall be expressed 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.

7.5.4 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 7.5.1 can be pre-set, it is not necessary to mark the butt lengths on the cigarettes themselves.

7.6 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 is not to be considered as a method of reducing the number of cigarettes to be smoked.

7.7 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 d.

If for any reason test samples are to be kept longer than 10 d 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.

7.8 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 in percent, of the conditioned cigarettes in accordance with ISO 6488.

8 Preparation for the smoking run

8.1 Smoking plan

Choose a smoking plan; examples are given in Annex A.

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

8.2 Preparation of mainstream and sidestream smoke traps and cigarette holders

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

Prepare the mainstream smoke traps and cigarette holders in accordance with ISO 3308.

Insert into the sidestream smoke trap filter discs that have been conditioned in the test atmosphere for at least 12 h, and assemble, placing the rough side of the filter disc so that it will face the oncoming smoke. Assemble the filter holder making sure that the conditioned filter disc is fitted correctly.

Weigh the assembled sidestream smoke traps to the nearest 0,1 mg. Due to absorption of water by smoke traps and solvent, determine a value for the sample blank. Prepare sample blanks by treating additional smoke traps in the same manner as that used for smoke collection.

8.3 Setting up the smoking machine

Set up the smoking machine in accordance with ISO 4387.

8.4 Assembly of fishtail chimney, sidestream trap and impinger trap

Each fishtail chimney shall be attached to an adjustable-height mounting block in such a way that it is securely held. Depending on the type of smoking machine and the degree of automation available, the mounting block may be manually or automatically raised and lowered. In its lowered position, the bottom of the fishtail chimney shall be at a distance of 6 mm from the horizontal plate of the smoking machine. The raised position shall be at a height sufficient for convenient access for loading cigarettes and removing extinguished butts.

The sidestream glass-fibre pad holder is attached to the top of the fishtail chimney by means of a suitable connector or a short piece of vacuum tubing.

The sidestream impinger trap inlet is connected to the glass-fibre pad holder by flexible tubing. This should be as short as practically possible and should be of polyvinyl chloride or, preferably, polyethylene polymer. Rubber or silicone rubber tubing shall not be used, as it may absorb vapour-phase nicotine. The sidestream impinger trap shall be held in a suitable clamp or clip. Conveniently, the impinger trap may be attached to the fishtail chimney mounting block. Its outlet is connected to the vacuum system by flexible tubing.

9 Procedure for smoking run and collection of sidestream smoke

9.1 Preparation of fishtail chimney

Secure each fishtail chimney in its lower position, measuring the distance from the horizontal plate with a suitable 6 mm spacer. Raise the chimney to its upper position.

9.2 Preparation of impinger trap

Add 40 ml of propan-2-ol containing the internal standard to each impinger trap, and connect the inlet to a sidestream glass-fibre pad holder by a short length of tubing. Connect each impinger trap outlet to the corresponding vacuum system.

The immersion depth of the impinger stem in the solvent shall be at least 36 mm.

9.3 Setting the fishtail chimney flow rate

Switch on the vacuum pumps. By means of the associated rotameters and needle valves, adjust the flow through each sidestream filter pad holder and associated impinger trap to (3,0 ± 0,1) l/min, using a suitable primary flow meter attached to the inlet of the sidestream filter pad holder. Switch off the vacuum pumps.

If a soap bubble flow meter is used, the sidestream pad may absorb water during the flow adjustment procedure, and should therefore be reweighed before the smoke run begins.

This procedure should be done as quickly as possible to minimize evaporation of the solvent from the impinger traps.

9.4 Connection of sidestream glass-fibre pad holders

Attach each sidestream trap securely to its fishtail chimney by means of a short piece of vacuum tubing or suitable connector.

9.5 Record the atmospheric conditions

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

9.6 Loading the cigarettes

Insert the conditioned cigarettes into the cigarette holders to the insertion depth recommended in ISO 3308 (9 mm). Avoid any leaks or deformation. Any cigarettes found to have obvious defects, or which have been damaged during insertion, shall be discarded and replaced with spare, conditioned cigarettes.

Ensure that the cigarettes are positioned correctly so that the angle formed by the longitudinal axis of the cigarette and the horizontal plane shall be as small as possible. It shall not exceed 10° if the centre of the butt end is lower than the centre of the other end and 5° if the centre of the butt end is higher than the centre of the other end.

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

Ensure that the cigarette position is centred with respect to the fishtail, and that the fishtail covers a maximum length of the cigarette while ensuring that the distance between the end of the cigarette and the front wall of the chimney is never less than 5 mm. In the case of long cigarettes this requirement may mean that the chimney may need to be moved along the axis of the cigarette as smoking progresses, in order to ensure that the fishtail covers the butt mark of the cigarette well before this is reached. The central axis of the cigarette will be positioned at a minimum of 15 mm above the bottom edge of the fishtail chimney.

Return the fishtail chimneys to their lowest position compatible with the lighting system (a distance of about 60 mm above the fixed plate has been found suitable).

9.7 Smoking the cigarettes

Switch on the vacuum pumps. Zero the puff counters and light each cigarette at the beginning of its first puff as specified by ISO 4387. As each butt mark is reached, immediately raise the fishtail chimney and remove the burning coal from the cigarette. Record the final reading of the puff counters. After the smoking process is complete, allow the vacuum pump to run for a minimum of 30 s, in order to clear any sidestream smoke from the chimney.

If required, new cigarettes shall be inserted immediately and the smoking process repeated until the predetermined number of cigarettes (normally three) has been smoked on each channel. Begin the determination of total particulate matter as described in Clause 10.

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

10 Determination of total particulate matter

10.1 Total sidestream particulate matter retained on glass-fibre filter pads

Wearing gloves, remove sidestream smoke traps from the smoking machine. Where necessary, remove the cigarette holder from the smoke trap.

Cover the front and back apertures of the traps with the sealing devices (5.15). 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. 44 mm glass-fibre filter pads are capable of

retaining up to 150 mg of total particulate matter. 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. Since sidestream TPM yields of 30 mg are not unusual, smoking three cigarettes normally provides an adequate margin to prevent overloading the glass-fibre filter pads.

10.2 Calculation of total sidestream particulate matter retained on glass-fibre filter pads

The mean mass per cigarette of total sidestream particulate matter retained on the glass-fibre pad for each channel, T_p, expressed in milligrams per cigarette, is given by Equation (1):

$$T_p = (m_1 - m_0)/q \quad (1)$$

where

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

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

q is the number of cigarettes smoked into the trap.

NOTE The total mainstream particulate matter can also be recorded, as it might serve as a check on the smoking procedure, although not directly the subject of this method.

10.3 Treatment of total sidestream particulate matter retained on glass-fibre filter pads

10.3.1 Extraction procedure

Wearing gloves, remove the sealing devices from the sidestream smoke trap, 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 a dry conical flask (maximum flask volume 150 cm^3 for 44 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 20 cm^3 extraction solution (propan-2-ol containing the internal standards for both nicotine and water determinations) into each flask.

Stopper the flask immediately and shake gently on a shaker for at least 20 min, ensuring that the disc does not disintegrate.

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

10.3.2 Determination of dry sidestream particulate matter in the glass-fibre filter pads

Water in the supernatant solution in each flask is determined according to ISO 10362-1.

The dry sidestream particulate matter retained in the glass-fibre filter pad, D_p, is calculated for each trap using Equation (2):

$$D_p = T_p - W_p \quad (2)$$

where

T_p is the total sidestream particulate matter, in milligrams per cigarette;

W_p is the water content in the total sidestream particulate matter, in milligrams per cigarette.

10.4 Extraction of sidestream particulate matter in the fishtail chimney

The fishtail chimney is rinsed with extraction solution and the rinsings are collected in a 50 ml volumetric flask, which is then brought to volume, stoppered and reserved for UV absorption measurement and nicotine determination (see 10.5).

The rinsing should be done as soon as possible after completion of smoking. Where condensed matter cannot readily be solubilised, this may be aided by scraping carefully with a glass rod.

10.5 Estimation of sidestream particulate matter in the fishtail chimney

10.5.1 General

Chimney and sidestream pad extracts shall be analysed within 36 h of smoking.

10.5.2 Principle of the method

In order to obtain an estimate of the amount of sidestream nicotine-free dry particulate matter condensed on the fishtail chimney walls, the UV absorbance at 310 nm is measured for the fishtail chimney extract and the sidestream glass-fibre filter pad extract after appropriate dilution. At this wavelength, nicotine and water are not significant absorbers, and the ratio of absorbances, together with the initial volumes and dilution factors, enables the estimation of nicotine-free dry particulate matter condensed on the fishtail chimney walls. Annex B gives informative examples of typical dilutions that may be applied for cigarettes of varying deliveries.

10.5.3 Spectrophotometric analysis of the sidestream filter and fishtail chimney extracts

Turn on the spectrophotometer at least 60 min prior to use. Set the wavelength to 310 nm. Zero the spectrophotometer with propan-2-ol.

Dilute the sidestream pad and chimney extracts as necessary. The absorbance measurement made for the determinations should not exceed 0,75 AU. Dilution of the sidestream filter extracts with propan-2-ol will normally be necessary, (except for the very lowest delivery cigarettes), to an extent dependent largely on the yields of the cigarettes (see Annex B). The dilution factor applied to each extract should be carefully recorded. Obtain duplicate readings of the absorbance of each extract. Record readings for calculation.

The concentration of nicotine-free dry particulate matter (NFDPM) in the fishtail chimney extract, x_c, expressed in milligrams per millilitre, is given by Equation (3):

$$x_c = \frac{(x_p \times a_c \times d_c)}{a_p \times d_p} \quad (3)$$

where

x_p is the concentration of NFDPM in sidestream pad extract, in milligrams per millilitre;

a_c is the absorbance of diluted chimney NFDPM extract;

a_p is the absorbance of diluted sidestream pad extract;

d_c is the dilution factor applied to chimney NFDPM extract;

d_p is the dilution factor applied to sidestream pad extract.

The amount of nicotine-free dry particulate matter in the chimney extract is then obtained by multiplying the concentration, x_c, by the volume of chimney extract (50 ml).

The estimated sidestream particulate matter condensed on the walls of the fishtail chimney, G_c, expressed in milligrams per cigarette, is calculated by Equation (4):

$$G_c = x_c \times 50/q \quad (4)$$

11 Determination of sidestream nicotine

NOTE It is recommended that capillary or megabore GC columns be used for the GC analyses listed in 11.1 to 11.3.

11.1 Determination of nicotine in the sidestream glass-fibre filter pad, N_p

Nicotine in the supernatant solution in each flask obtained as described in 10.3.1 is determined in accordance with ISO 10315.

11.2 Determination of nicotine in the fishtail chimney extract, N_c

The solution obtained by washing the fishtail chimney (see 10.4) is analysed for its nicotine content as described in ISO 10315.

11.3 Determination of nicotine in the impinger trap, N_i

The content of the impinger trap is transferred to a 50 ml flask. The trap and the tubing connecting it to the glass-fibre filter are rinsed with a small volume of pure propan-2-ol, and the rinsings are added to the flask. The solution is then analysed for its nicotine content in accordance with ISO 10315.

NOTE It is not necessary to make the solution up to a known final volume with pure propan-2-ol, although this may be done if desired. The calculation of the amount of nicotine present may be made using the initial volume of 40 ml. It has been shown that although solvent is evaporated from the impinger trap during the smoking run, the internal standard is not lost to any significant extent.

11.4 Calculation of total sidestream nicotine, N

The total sidestream nicotine is calculated as the sum of the nicotine found in the fishtail chimney, the glass-fibre filter pad and the impinger trap, in milligrams per cigarette, using Equation (5):

$$N = N_c + N_p + N_i \quad (5)$$

12 Determination of nicotine-free dry sidestream particulate matter

12.1 Determination of nicotine-free dry sidestream particulate matter on the glass-fibre filter pad

The nicotine-free dry sidestream particulate matter retained on the glass-fibre filter pad, G_p, expressed in milligrams per cigarette, is calculated for each glass-fibre filter pad using Equation (6):

$$G_p = D_p - N_p \quad (6)$$

where D_p is the dry sidestream particulate matter, in milligrams per cigarette.

12.2 Determination of total nicotine-free dry sidestream particulate matter

The total nicotine-free dry sidestream particulate matter, G, expressed in milligrams per cigarette, is calculated using Equation (7):

$$G = G_p + G_c \quad (7)$$

13 Test report

13.1 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. Where appropriate, record the information in 13.2 to 13.5.

13.2 Characteristic data about the cigarette

All details necessary for the identification of the cigarette smoked shall be given. In the case of a commercial cigarette this may include:

— name of manufacturer, country of manufacture;

— product name;

— date of sampling;

— place of purchase or sampling;

— kind of sampling point;

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

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

— marks on any tax stamp;

— printed mainstream smoke yields (if any);

— length of cigarette;

— length of filter;

— length of overwrap.

13.3 Data about sampling

— type of sampling procedure;

— number of cigarettes in laboratory sample;

— date and location of purchase.

13.4 Description of test

— date of test;

— type of smoking machine used;

— type of smoke trap used;

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

— number of cigarettes smoked into each smoke trap;

— butt length;

— room temperature (in degrees centigrade) during smoking operation;

— relative humidity (in percent) during smoking operation;

— atmospheric pressure (in kilopascals) during smoking operation.

13.5 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, to the nearest 0,1 mm;

— average length of the filter, to the nearest 0,1 mm;

— average length of the overwrap, to the nearest 0,1 mm;

— butt length to which cigarettes were smoked, to the nearest 0,1 mm;

— average lengths of tobacco portion smoked, 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;

— water content, as a mass fraction in percent, of the conditioned cigarettes (see ISO 6488);

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

— total sidestream particulate matter, in milligrams per cigarette, for each channel, to the nearest 0,1 mg and the average per cigarette, to the nearest 1 mg;

— dry sidestream particulate matter, in milligrams per cigarette, for each channel, to the nearest 0,1 mg, and the average per cigarette, to the nearest 1 mg;

— nicotine-free dry sidestream particulate matter, in milligrams per cigarette, for each channel, to the nearest 0,1 mg, and the average per cigarette, to the nearest 1 mg;

— total sidestream nicotine, in milligrams per cigarette, for each channel, to the nearest 0,01 mg, and the average per cigarette, to the nearest 0,1 mg.

14 Repeatability and reproducibility

A major international collaborative study involving fifteen laboratories and seven cigarette samples including the CM3 test piece and spanning a wide range of blends and construction was conducted in 2001 and gave the following values for repeatability, r, and reproducibility, 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 value, r, on average not more than once in twenty 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 twenty cases in the normal and correct operation of the method.

Data analysis for the seven cigarettes gave the estimates as summarized in Table 2.

Table 2 — Estimates given by data analysis

Values in milligrams per cigarette

Cigarette sample	Total sidestream NFDPM			Total sidestream nicotine		
	Mean value	r	R	Mean value	r	R
A	13,97	2,66	4,64	4,107	0,578	1,066
B	27,14	3,74	4,89	5,509	0,588	1,072
C	20,40	3,15	5,27	2,879	0,408	0,673
D	22,04	2,96	4,39	5,786	0,745	0,826
E	27,25	3,88	6,15	5,061	0,735	0,768
F	21,58	3,84	5,11	4,996	0,666	1,229
CM3	27,04	3,84	5,44	5,022	0,681	1,042

For the purposes of calculating r and R, one test result was defined as the mean yield obtained from smoking three cigarettes in a single run. Eight test results were obtained for each cigarette type by each of the participating laboratories.

Annex A
(informative)

Smoking plans

In the majority of cases the results of mechanical smoking permit a comparison of types of cigarette (treatment). This comparison should be made according to a smoking plan established in advance. The smoking plan should take account of:

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

b) the capacity of the sidestream smoke collection system: it determines the number of cigarettes to be smoked in each channel;

c) required precision: the results of smoking always give a certain variability; the distributions 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, itself related to the capacity of the machine.

The exact value to be selected for N, chosen in the ranges above (see 7.3) taking into account the preceding factors, is determined by calculation for each experiment taking into account the parameters which characterize that value.

Also if

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

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

c denotes the number of channels on the machine;

q denotes the number of cigarettes smoked into the same sidestream smoke collection system;

then the different parameters are related by Equation (A.1):

$$t \times N = s \times c \times q \tag{A.1}$$

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

— Example 1: comparison of two types of cigarette on one single-channel smoking machine. The sidestream smoke collection system can collect the sidestream condensate of three cigarettes.

— Example 2: comparison of three types of cigarette on one single-channel smoking machine. The sidestream smoke collection system can collect the sidestream condensate of three cigarettes.

— Example 3: comparison of two types of cigarette on one four-channel smoking machine. The sidestream smoke collection system can collect the sidestream condensate of three cigarettes.

— Example 4: comparison of five types of cigarette on one twenty-channel smoking machine. The sidestream smoke collection system can collect the sidestream condensate of three cigarettes.