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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

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 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

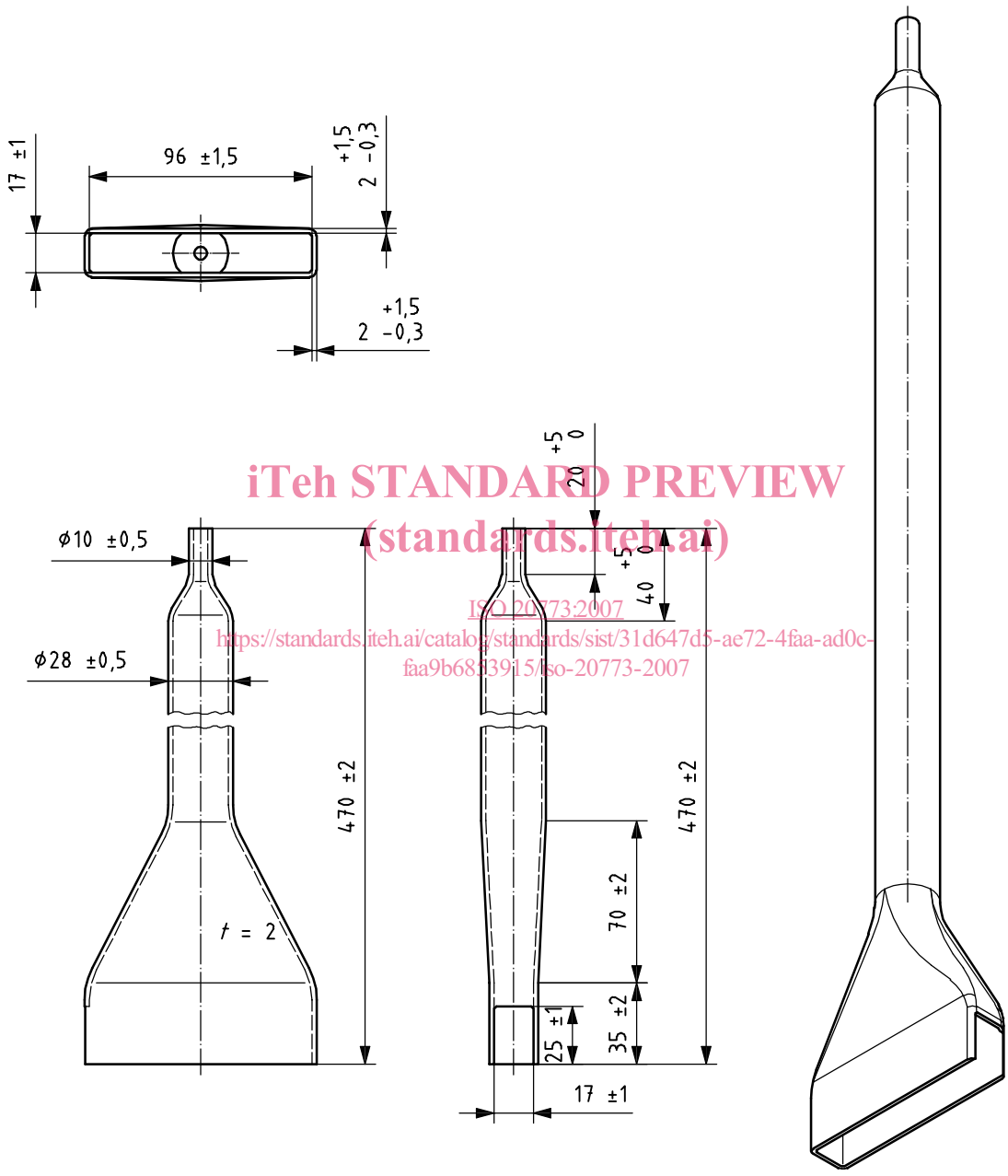
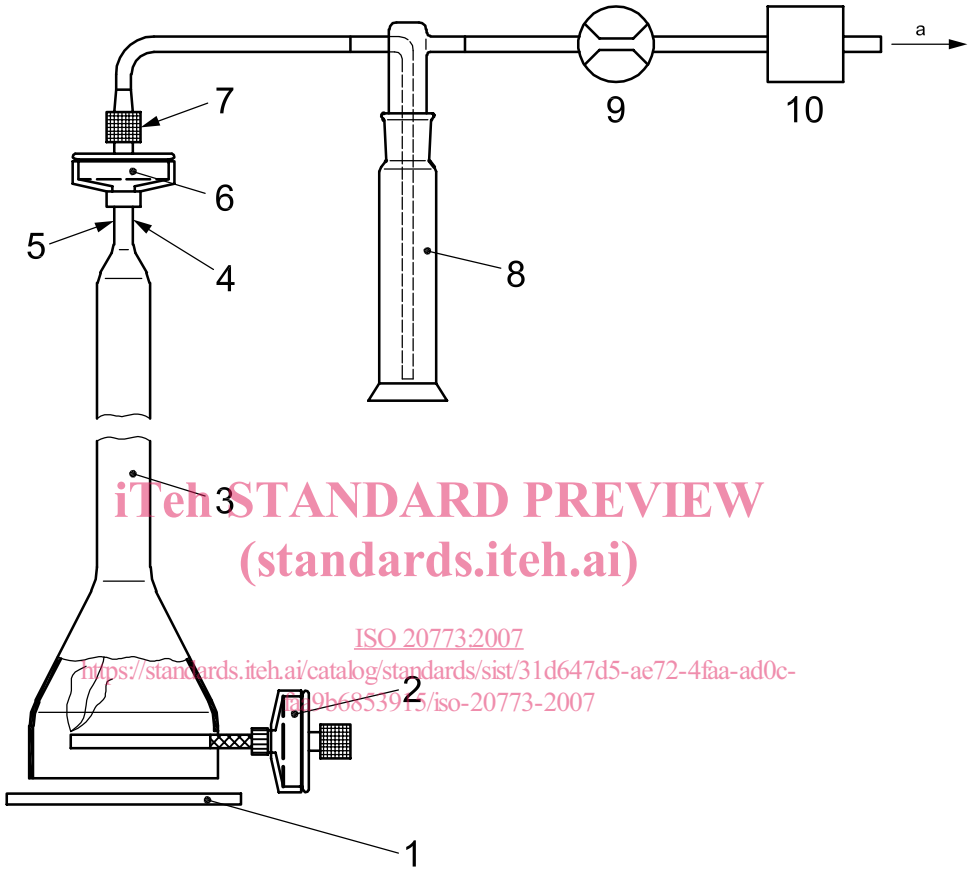


Figure 1 — Fishtail chimney dimensions

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 %).