



BSI Standards Publication

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

National foreword

This British Standard is the UK implementation of ISO 20773:2013. It supersedes BS ISO 20773:2007 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee AW/40, Tobacco and tobacco products.

A list of organizations represented on this committee can be obtained on request to its secretary.

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ISBN 978 0 580 74580 5

ICS 65.160

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This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 September 2013.

Amendments issued since publication

Date	Text affected
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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 d'une cheminée individuelle en forme de queue de poisson





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Published in Switzerland

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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

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received. www.iso.org/patents

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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

This second edition cancels and replaces the first edition (ISO 20773:2007), which has been editorially revised.

Introduction

Cigarettes are manufactured to close tolerances using strict quality control procedures.

However, the main constituents involved in the manufacture are derived from natural products (such as tobacco and paper) 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 (which is called mainstream smoke).

NOTE Sidestream smoke 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 (www.coresta.org) 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 ISO standards for the determination of mainstream smoke 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 smoke 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 monitor test piece in the smoking plans, as is done in mainstream smoke determinations. It is possible to use the CORESTA monitor or any other internally designed monitor test piece for this purpose. The use of an internationally recognized one 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

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 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 adaptable to other sidestream smoke analytes.

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 2971, *Cigarettes and filter rods — Determination of nominal diameter — Method using a non-contact optical 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 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*

3 Terms and definitions

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

3.1
total sidestream smoke particulate matter
crude sidestream smoke condensate

portion of the sidestream smoke which is trapped on the sidestream smoke filter pad, together with that portion of the sidestream smoke which condenses on the wall of the fishtail chimney

Note 1 to entry: Expressed as milligrams per cigarette.

3.2
dry sidestream smoke particulate matter
dry sidestream smoke condensate

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

Note 1 to entry: Expressed as milligrams per cigarette.

3.3
nicotine-free dry sidestream smoke particulate matter
nicotine-free dry sidestream smoke condensate

dry sidestream smoke particulate matter, after deduction of its nicotine content

Note 1 to entry: Expressed as milligrams per cigarette.

3.4
sidestream smoke nicotine

sum of the nicotine condensed on the walls of the fishtail chimney, the nicotine collected on the sidestream smoke filter pad and the nicotine collected in the impinger trap

Note 1 to entry: 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
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

3.9
conditioning sample

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

3.10
test portion

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

3.11

monitor test piece

sample produced for a specific test purpose, validated to fulfil requirements within specified tolerances and intended to be used for laboratory purposes only and labelled to clearly indicate that it is not for human use

Note 1 to entry: A monitor test piece is a sample taken from a batch of cigarettes that show the greatest homogeneity with regard to their physical, chemical and smoke yield characteristics.

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 smoke particulate matter on the walls of the fishtail chimney and in a filter pad, and collection of vapour phase sidestream smoke nicotine in an impinger trap.
- Gravimetric determination of the mass of total sidestream smoke particulate matter collected on the filter pad.
- Extraction of the total sidestream smoke particulate matter from the filter pad for the determination of water and nicotine contents by gas chromatography.
- Estimation of the nicotine-free dry sidestream smoke 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 filter pad.
- Analytical determination by gas chromatography of the nicotine collected in the fishtail chimney, the filter pad and the impinger trap.

NOTE In 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 chimney¹⁾, manufactured in glass, of design and dimensions shown in Figure 1.

1) Details of where to obtain fishtail chimneys are available from ISO/TC 126.