
**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*

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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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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 (see 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 (see 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.

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*.

This fourth edition cancels and replaces the third edition (ISO 4387:2000), which has been technically revised. It also incorporates the Amendments ISO 4387:2000/Amd.1:2008 and ISO 4387:2000/Amd.2:2017.

The main change compared to the previous edition is as follows:

- in 7.6.4, smoking and collection of particular matter has been modified by moving wording from ISO 8454 into ISO 4387.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Cigarettes are manufactured to close tolerances using strict quality control procedures. However, all the constituents involved in the manufacture (such as tobacco, cigarette paper, tipping, etc.) are derived from natural products and this results in a final product which is intrinsically variable. The complexity does not end 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 complicates its composition.

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

From the time that scientists have attempted to determine a value for NFDPM, a number of 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 document, 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 document 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.

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 document.
- 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.

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

1 Scope

This document 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 documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 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 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 16055, *Tobacco and tobacco products — Monitor test piece — Requirements and use*

3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at <https://www.iso.org/obp>
- IEC Electropedia: available at <http://www.electropedia.org/>

3.1

**total particulate matter
crude smoke condensate
TPM**

portion of the mainstream smoke which is trapped in the smoke trap

Note 1 to entry: It is expressed as milligrams per cigarette.

3.2
dry particulate matter
dry smoke condensate
DPM

total particulate matter (3.1) after deduction of its water content

Note 1 to entry: It is expressed as milligrams per cigarette.

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

dry particulate matter (3.2) after deduction of its nicotine content

Note 1 to entry: It is 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* (3.4) to produce such smoke from a sample of cigarettes as is necessary for the determination of the smoke components

3.6
clearing puff

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

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* (3.6) and which are representative of each of the increments making up the laboratory sample

3.9
conditioning sample

cigarettes selected from the *test sample* (3.7) for conditioning prior to tests

3.10
test portion

group of cigarettes prepared for a single determination and which is a random sample from the *test sample* (3.7) or conditioned sample, as appropriate

3.11
monitor test piece

cigarette taken from a batch specially fabricated under controlled manufacturing conditions

Note 1 to entry: 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.

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 $\pm 0,2$ 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 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 required for the test by randomly selecting cigarettes from the different parts of the population represented in the laboratory sample.

7 Determination of total particular 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.

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

The precision normally required generally demands that $80 \leq N \leq 100$. 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.

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

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