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

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

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Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

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

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

Annex A of this International Standard is for information only.

Introduction

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

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

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

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

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

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

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

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

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

1 Scope

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

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

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

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

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

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

ISO 8243, *Cigarettes — Sampling.*

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

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

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

3 Terms, definitions and abbreviated terms

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

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

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

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

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

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

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

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

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

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

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

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

4   Principle

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

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

5   Apparatus

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

5.1   **Routine analytical cigarette-smoking machine**, complying with the requirements of ISO 3308.

5.2   **Soap bubble flow meter**, graduated at 35 ml to an accuracy of $\pm$ 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 (3.6) shall be taken by a sampling scheme such as one of those given in ISO 8243.

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

7   Determination of total particulate matter

7.1   Preparation of the cigarettes for smoking

7.1.1   General

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

The symbols used in this clause are as follows:

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

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

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

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

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

P is the total number of packets of cigarettes available.

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

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

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

The N cigarettes to be smoked will be tested in $n = N/q$ determinations if q cigarettes are smoked into one trap. As far as possible these n determinations should correspond to different test portions of the test sample. Selection of each test portion will depend upon the form of the test sample.

7.1.2 Selection of test portions from a bulk of Q cigarettes

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

7.1.3 Selection of test portions from P packets

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

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

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

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

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

7.1.4 Duplicate test portions

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

7.2   Marking the butt length

7.2.1   Standard butt length

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

⎯   23 mm,

⎯   length of filter + 8 mm, or

⎯   length of overwrap + 3 mm,

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

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

7.2.2   Measurement of length of filter

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

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

7.2.3   Measurement of length of overwrap

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

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

7.2.4   Butt length to be marked on the cigarettes before conditioning

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

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

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

7.3   Selection of cigarettes

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

7.4   Conditioning

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