



SLOVENSKI STANDARD
SIST ISO 4387:1995

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

iTeh STANDARD PREVIEW

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

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Ta slovenski standard je istoveten z: **ISO 4387:1991**

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ICS:

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and related equipment

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INTERNATIONAL STANDARD

ISO
4387

Second edition
1991-10-15

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

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*Cigarettes — Détermination de la matière particulaire totale et de la
matière anhydre et exempte de nicotine au moyen d'une machine à
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Reference number
ISO 4387:1991(E)

ISO 4387:1991(E)

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.

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.

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

This second edition cancels and replaces the first edition (ISO 4387:1987), which has been technically revised as a result of extensive collaborative testing by members of CORESTA.

Subclauses 3.7 to 3.11 have been changed to harmonize with other definitions in other International Standards developed by ISO/TC 126; notably ISO 8243:1991, *Cigarettes — Sampling*.

Clause 4 has been modified to comprise the preparation of samples for the determination of water and nicotine by gas chromatography.

Subclauses 7.2.2 and 7.2.3 have been added to specify how measurements shall be made for use in 7.2.1.

Subclause 7.6.2 has been changed to explain how the perforated disc (now specified in ISO 3308:1991, 4.8) shall be assembled in the holder.

Subclause 7.9.1 describes the extraction procedures to provide samples for the estimation of nicotine and water by gas chromatography.

Clause 9 gives details of repeatability (r) and reproducibility (R).

Annexes A and B of this International Standard are for information only.

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International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

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 to complicate its composition further.

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, 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 upon 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.

ISO 10315:1991, *Cigarettes — Determination of nicotine in smoke condensates — Gas-chromatographic method.*

ISO 10362-1:1991, *Cigarettes — Determination of water in smoke condensates — Part 1: Gas-chromatographic method.*

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 2971:1987, *Cigarettes and filters — Determination of nominal diameter — Pneumatic method.*

ISO 3308:1991¹⁾, *Routine analytical cigarette-smoking machine — Definitions and standard conditions.*

ISO 3402:1991, *Tobacco and tobacco products — Atmosphere for conditioning and testing.*

ISO 6488:1981, *Tobacco — Determination of water content (Reference method).*

ISO 6565:1983, *Tobacco and tobacco products — Draw resistance of cigarettes and filter rods — Definitions, standard conditions and general aspects.*

ISO 8243:1991, *Cigarettes — Sampling.*

3 Definitions

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

3.1 total particulate matter; crude smoke condensate: That portion of the mainstream smoke which is trapped in the smoke trap, expressed as milligrams per cigarette (mg/cig.).

3.2 dry particulate matter; dry smoke condensate: The total particulate matter after deduction of its water content, expressed as milligrams per cigarette (mg/cig.).

3.3 nicotine-free dry particulate matter; nicotine-free dry smoke condensate: The dry particulate matter after deduction of its nicotine content, expressed as milligrams per cigarette (mg/cig.).

3.4 clearing puff: Any puff taken after the cigarette has been extinguished or removed from the cigarette holder.

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

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

3.7 laboratory sample: The sample intended for laboratory inspection or testing and which is rep-

1) To be published.

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representative of the gross sample or the sub-period sample.

3.8 conditioning sample: The cigarettes selected from the test sample for conditioning prior to tests for particulate matter 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: A 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 particulate matter yield tests.

4 Principle

Sampling of the test cigarettes. Conditioning of the test cigarettes. Smoking of the test cigarettes on an automatic smoking machine with simultaneous collection of total particulate matter in a glass fibre filter trap. Gravimetric determination of the mass of total particulate matter so collected. Extraction of the total particulate matter from the trap for the determination of the water and nicotine contents by gas chromatography.

NOTE 1 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 nicotine alkaloids, and the determination of water in smoke condensate should be performed by a Karl Fischer method²⁾. 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 with a resolution of 0,1 ml.

5.3 Apparatus for the determination of puff duration and frequency.

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

5.5 Draw resistance testing equipment.

5.6 Conditioning enclosure, carefully maintained under the conditions specified in ISO 3402.

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

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

5.9 Sealing device, end caps made from the same non-hygroscopic and chemically inert material as the filter holder.

5.10 Gloves, cotton or non-talc surgical.

6 Sampling

Provide a laboratory sample (see 3.7) by a sampling scheme such as one of those given in ISO 8243.

NOTE 2 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 particulate matter

The following symbols are used in 7.1 and 7.6:

- 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, value greater than 1, to allow for loss due to damage or selection procedures between initial sampling and smoking;
- 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;
- Q** is the total number of cigarettes available (laboratory sample, see 3.7).

7.1 Preparation of the cigarettes for smoking

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

NOTE 3 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 (ex-

2) ISO/TC 126 is at present examining a working draft for the Karl Fischer method of water determination.

perience 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.

NOTE 4 40 cigarettes are required 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.

7.1.1 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 shall be selected at random so that every cigarette has an equal probability of being selected.

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

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

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

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.

7.1.3 Duplicate test portions

Provided that the test sample is sufficiently large ($\geq 2 C \times N$), 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 7.1.2 would need to be changed to $Q/P \geq 2 C \times q$ and $Q/P < 2 C \times q$.

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
- 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 5 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 20 cigarettes taken from the laboratory sample measured to an accuracy of 0,5 mm. Express the mean of the 20 measurements to the nearest 0,1 mm.

7.2.3 Measurement of length of overwrap

The length of overwrap shall be the mean value of 20 overwraps taken from the laboratory sample measured to an accuracy of 0,5 mm. Express the mean to the nearest 0,1 mm.

7.2.4 The butt length to be marked on the cigarettes before conditioning

It is recommended that two thin lines be drawn using a fine, soft-tipped marker. Draw the first line at 9 mm to an accuracy of 0,5 mm from the mouth end of the cigarettes (corresponding to the standard depth of insertion: ISO 3308:1991, 4.8) and the second 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.

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

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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 d.

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

7.5 Preliminary tests before smoking

Determine the following data which may be required in the test report.

7.5.1 Total length of the cigarette.

7.5.2 Nominal diameter, determined in accordance with ISO 2971.

7.5.3 Draw resistance of the cigarette, determined in accordance with ISO 6565.

7.5.4 Average mass of the conditioned cigarettes selected for the smoking operation (milligrams per cigarette).

7.5.5 Water content (m/m) of the conditioned cigarettes, determined in accordance with ISO 6488.

7.6 Smoking and collection of particulate matter

7.6.1 Smoking plan

Choose a smoking plan; examples of plans are given as an indication in annex A.

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

7.6.2 Preparation of smoke traps and cigarette holders

For all operations the operator shall prevent con-

tamination from the fingers by wearing gloves of a suitable material.

Insert filter discs which have been conditioned in the test atmosphere for at least 12 h into their holders, and assemble, placing the rough side of the filter disc so that it will face the oncoming smoke. After assembly, examine the filter holders to ensure that the discs have been properly fitted. If the smoke trap is designed to contain the perforated disc (washer), insert it and fit the sealing devices (end caps). If the cigarette holder is designed to contain a perforated disc, insert it into the cigarette holder before attaching the labyrinth seals (see ISO 3308:1991, 4.8). Weigh the assembled 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 (at least 2 per 100 cigarettes) in the same manner as that used for smoke collection.

7.6.3 Setting up the smoking machine

If necessary replace any protective filters on the machine. Switch on and allow to warm up on automatic cycling for at least 20 min.

With the machine warmed up, check that the puff duration and puff frequency on each channel are in accordance with the standard conditions.

7.6.3.1 Measurement of puff duration

A timer, working with reference to a crystal-controlled oscillator, shall be used to measure the period of time which elapses between the triggering operations which begin and end a puffing action of the smoking machine. The accuracy of the timing device shall be such as to ensure that a 1 % error in the puff duration can be detected. The timer should be coupled directly to the triggering circuits.

NOTE 6 It is not possible to specify the method of measurement beyond a statement of principle because of the variety of types of suitable timers and smoking machines available.

7.6.3.2 Checking of puff frequency

Measure the period of time which elapses between the triggering operations which begin successive puffing actions of the smoking machine, thus determining the puff frequency. The timer used shall be suitable for measuring to the nearest 0,1 s and should, preferably, be coupled directly to the triggering circuits.

7.6.3.3 Measurement of puff volume

The displacement of the bubble in a soap bubble flow meter (5.2) gives a direct measurement of puff volume and also provides a check for leaks in the

system. A suitable indicator graduated at 35 ml shall have a resolution of 0,1 ml. It shall be connected through a standard pressure drop device of 100 mm water gauge (approximately 1 kPa) to the cigarette holder of the smoking machine channel under test. Before use on a series of measurements wet the instrument twice with detergent solution and then allow to drain for a period of between 30 s and 45 s.

The bubble flow meter shall contain a 15 % aqueous solution of a surface active agent. Teepol L[®] 3) has been found to be satisfactory.

Fit the prepared smoking trap or traps and cigarette holders on to the machine. Use a plastic insert as shown in figure 1 to measure puff volume. Attach the correct size insert for the labyrinth seals in the cigarette holder to the resistance in the soap bubble flow meter indicator. Prepare the soap bubble flow meter by wetting the inside of the tube with the detergent solution to above the top graduation mark. Connect the indicator to the cigarette holder in port 1 and determine the puff volume; adjust if necessary to $35 \text{ ml} \pm 0,3 \text{ ml}$. Repeat for all remaining ports in turn.

Repeat the determinations until the necessary precision of measurement is obtained. If the number of replicates exceeds three, continue until the correct precision is obtained but replace the pad before smoking, reweigh the smoke trap and recheck the

puff volume with the new pad in place. Measure the temperature and relative humidity of the air surrounding the smoking machine and note the atmospheric pressure. Check the ambient conditions if it is suspected that the air current may be too high.

7.6.4 Procedure for smoking run

Insert the conditioned cigarettes into the cigarette holders so that the butt end impinges upon the perforated disc fitted within the filter trap; this is usually at the standard depth of 9 mm. Avoid any leaks or deformations. 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; the axis of cigarettes shall coincide with the axis of the ports. Adjust the position of each cigarette so that when the burning coal reaches the butt mark, the puff termination device is activated. If the burning through of 100 % cotton thread (40 denier) is used to terminate smoking at the butt mark, the cotton shall just touch the cigarette at the butt mark, without modifying the cigarette positioning.

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Dimensions in millimetres

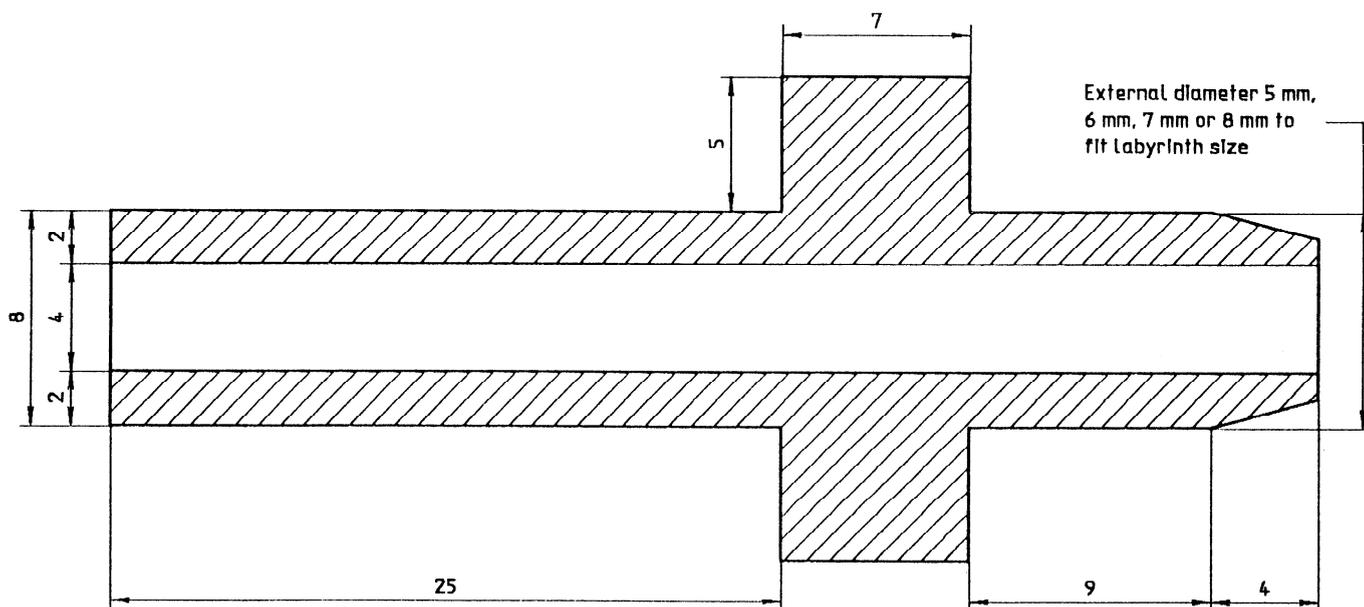


Figure 1 — Example of plastic insert for use during puff volume adjustment

3) Teepol L is the trade name of a product supplied by Shell. 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.