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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Cigarettes — Determination of total and dry particulate matter using a routine analytical cigarette-smoking machine — Glass fibre filter smoke trap method

Cigarettes — Détermination du condensat de fumée brut et anhydre au moyen d'une machine à fumer analytique de routine pour cigarettes — Méthode par piégeage sur disque en fibre de verre

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Foreword

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Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

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

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

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Cigarettes — Determination of total and dry particulate matter using a routine analytical cigarette-smoking machine — Glass fibre filter smoke trap method

0 Introduction

The procedure specified in this International Standard is applicable to all types of cigarettes; however, the results obtained from machine smoking of certain types of cigarettes may not be suitable for ranking or consumer information and may reveal the need for further and more adequate information to be made available.

ISO/TC 126 is studying this problem and will try to find a solution which will be taken into account in a future revision of this International Standard.

1 Scope and field of application

This International Standard specifies a method for determining the total and dry particulate matter present in the smoke from cigarettes generated and collected using a routine analytical cigarette-smoking machine equipped with glass fibre filter smoke traps.

2 References

ISO 760, *Determination of water — Karl Fischer method (General method).*

ISO 2971, *Tobacco and tobacco products — Cigarettes and filters — Determination of nominal diameter.*

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

ISO 3400, *Tobacco and tobacco products — Determination of alkaloids in cigarette smoke condensates — Spectrophotometric method.*

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

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

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

ISO 8243, *Cigarettes — Sampling.* [1]

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 alkaloids content, expressed as milligrams per cigarette (mg/cig.).

3.4 clearing puff : Any puff taken after a cigarette has been extinguished.

3.5 smoking process : The use of a smoking machine to smoke one cigarette per port from lighting to final puff.

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

3.7 laboratory sample : The cigarettes sent to the laboratory for testing.

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

3.9 test portion : The conditioned cigarettes smoked for particulate matter yield tests.

1) At present at the stage of draft.

4 Principle

Smoking of the test cigarettes in an automatic smoking machine and collection of the mainstream smoke by a glass fibre filter smoke trap. Gravimetric determination of the mass of total particulate matter so collected. Extraction of the total particulate matter from the trap and determination of the water content by Karl Fischer or gas chromatographic methods.

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, range 0 to 50 ml, graduated in 0,1 ml divisions.

5.3 Apparatus for the determination of puff duration and frequency.

5.4 Analytical balance.

5.5 Pressure drop testing equipment.

5.6 Conditioning enclosure, maintained accurately in accordance with the conditions specified in ISO 3402.

5.7 Equipment for the determination of water content.

This can be either Karl Fischer equipment in accordance with ISO 760 or a suitable gas chromatograph (see 8.1 or 8.2).

5.8 Glass fibre filter smoke trap, comprising :

— **Filter holders** made of an airtight non-hygroscopic and chemically inert material, preferably transparent, able to contain a filter disc of glass fibre material 1 to 2 mm thick and with a diameter of at least 44 mm. The rough filter surface shall face the oncoming smoke.

— **Filter material** which shall retain at least 99,9 % of all particles having a diameter equal to or greater than 0,3 µm of a dioctyl phthalate aerosol at a linear air velocity of 140 mm/s. The pressure drop of the filter assembly shall not exceed 900 Pa (9 mbar) at this air velocity. The content of polyacrylate binder shall not exceed 5 % (m/m).

The filter assembly shall be capable of quantitatively retaining all the mainstream smoke produced by the cigarette without loss of particulate matter. In addition, the filter assembly shall be chosen so that the increase in pressure drop of the assembly does not exceed 250 Pa (2,5 mbar), at a flow rate of 17,5 ml/s when measured after the smoking run.

5.9 Apparatus for measuring the angular position of the cigarettes.

This instrument shall rest on a horizontal plane of the smoking machine at the level of the smoking ports. It shall consist of a set of calipers, one leg of which is in the horizontal plane while the other is adjustable so as to be parallel to the cigarettes inserted in the cigarette holders on the smoking machine. By using a protractor, it will be possible to ensure that the cigarette position with respect to the horizontal plane is in agreement with the standard conditions.

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

5.11 Apparatus for the determination of diameter.

6 Sampling

A laboratory sample, representative of the cigarette type to be sampled, shall be provided by a sampling scheme such as one of those in ISO 8243. This sample will normally contain cigarettes taken from different parts of the population.

For any particular test, for example determination of total particulate matter, it should then be possible to 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

In 7.1 and 7.6 below, the following symbols are used :

N is the number of cigarettes of a given type to be smoked;

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 (test sample).

7.1 Preparation of the cigarettes for smoking

If N cigarettes of a given type are to be smoked, $C \times N$ cigarettes should be prepared from Q for conditioning and butt marking. 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 (experience suggests 2,0 to 4,0), depending on the selection process.

The precision normally required generally demands that $80 \leqslant N \leqslant 100$. This number may be substantially augmented if the variability of the sample is high; on the contrary, in certain comparisons made of homogeneous samples, this number may be reduced. As a precaution it is recommended that N should never be less than 40.

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 should be selected at random so that every cigarette has an equal probability of being chosen.

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

7.1.3 Duplicate test portions

Provided that the test sample is sufficiently large ($\geqslant 2\,C \times N$), it would be prudent to reserve a duplicate set of n test portions. 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 $Q/P \geqslant 2\,C \times q$ and $Q/P < 2\,C \times q$.

7.2 Butt marking

The butt length should be marked on the cigarettes in accordance with 4.9 of ISO 3308 before conditioning.

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.

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 is not to be considered as a method of reducing the number of cigarettes to be smoked.

7.4 Conditioning

Condition all the test portions in conditioning atmosphere A of ISO 3402 for a minimum of 48 h and for a maximum of 1 week.

It is recommended that the testing atmosphere in the laboratory where the smoking is to be carried out is as close as possible to the conditioning atmosphere. If the testing atmosphere is different, place the conditioned test sample in an airtight container (just large enough to contain the portion) and do not remove it from the container until just before the smoking.

7.5 Preliminary tests for physical parameters

The following data which may be required in the test report should be determined :

7.5.1 Total length of the cigarette.

7.5.2 Nominal diameter determined according to ISO 2971.

7.5.3 Length of filter and tipping paper.

7.5.4 Draw resistance of the cigarette determined according to ISO 6565.

7.6 Smoking and collection of particulate matter

7.6.1 Smoking plan

A smoking plan shall be chosen; examples are given in the annex to this International Standard.

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 glass fibre filter discs

For all operations the operator shall prevent contamination from the fingers by wearing gloves of a suitable material.

Insert the filter discs into their holders and assemble the holders. If the filter disc material has a rough and a smooth side, place the rough side facing the cigarettes. After assembly, examine the filter holders to ensure that the discs have been properly fitted and then expose them to the air in the laboratory for at least 12 h. Weigh the assembled filter holders to the nearest 0,1 mg.

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.

If draught screens are needed to achieve the standard ambient conditions (see 5.4 of ISO 3308) set them in place.

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 — 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. This will determine the puff frequency. The timer used shall be suitable for measuring to the nearest 0,1 s. It should be coupled directly to the triggering circuits.

7.6.3.3 Measurement of puff volume

Fit the prepared smoking trap or traps and cigarette holders onto the machine.

For each channel attach a resistance equal to the mean draw resistance of the test cigarettes plus 200 Pa. Check the puff volume of 35 ± 0,1 ml and readjust if necessary.

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

A suitable indicator shall be accurate over a range 0 to 50 ml and shall be graduated in 0,1 ml divisions. It shall be connected through a standard resistance (equal to the mean resistance of the cigarettes to be tested plus 200 Pa) to the cigarette holder of the smoking machine channel under test. Before use on a series of measurements, the instrument shall be wetted twice with detergent solution and then allowed to drain for a period of between 30 s and 45 s.

The bubble flow meter shall contain an aqueous solution of a surface active agent of adequate concentration.

Replicate determinations shall be made until the necessary precision of measurement is obtained.

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 to the standard depth avoiding 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. Using the apparatus described in 5.9, ensure that the cigarettes are positioned correctly; 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 cotton threads (30 to 40 denier) is used to terminate smoking at the butt mark, the cotton shall just touch the cigarettes at the butt mark, without modifying the cigarette positioning.

Zero the puff counters and light each cigarette at the beginning of its first puff. When each butt mark has been reached, remove the burning coal from the cigarette, and note the final reading of the puff counters. Take at least one clearing puff immediately the smoking process is complete and remove the cigarette butt.

NOTE — Avoid disturbance of the smoking by artificial removal of ash.

New cigarettes shall be inserted immediately and the smoking process repeated until the predetermined number of cigarettes, according to the smoking plan, has been smoked into the smoke trap. Begin the determination of total particulate matter as described in 7.7 immediately.

7.7 Determination of total particulate matter

Remove the smoke traps from the smoking machine. Where necessary, remove the cigarette holder from the smoke trap. It is recommended, particularly when plain cigarettes have been smoked, that this latter operation be conducted with the smoke trap held with its cigarette-facing side downwards to avoid any possible contaminants from the cigarette holder reaching the filter.

Check the back of each filter disc to ensure that there are no brown stains indicating overloading. Discard any disc showing such stains.

Immediately weigh the smoke traps to the nearest 0,1 mg.

7.8 Calculation of total particulate matter

The mean mass per cigarette of total particulate matter T for each channel, expressed in milligrams per cigarette, is given by the equation

$$T = \frac{m_1 - m_0}{q}$$

where

m_0 is the mass, in milligrams, of the smoke trap before smoking;

m_1 is the mass, in milligrams, of the smoke trap after smoking;

q is the number of cigarettes smoked into the trap.

7.9 Treatment of total particulate matter

The total particulate matter may be used for

— determination of water content and calculation of dry particulate matter;

— determination of nicotine alkaloids content according to ISO 3400 and calculation of nicotine-free dry particulate matter.

8 Determination of water content of the total particulate matter

This may be determined either by the Karl Fischer method or by gas chromatography. Care shall be taken during all operations to avoid contamination from atmospheric moisture.

All glassware used in the water determination shall be heated at 105 °C for at least 1 h after visible water has evaporated, and cooled and stored in a desiccator over silica gel until used.

8.1 Karl Fischer method

8.1.1 Apparatus

NOTE — The use of automatic apparatus is recommended for the pipetting and titration of the solvents and solutions.

8.1.1.1 Karl Fischer apparatus for automatic titration, with automatic burettes of 25 ml capacity each, graduated in 0,05 ml, provided with connecting pieces and tubes, and one additional automatic burette of 50 ml capacity.

8.1.1.2 Sleeve caps with conical 29/32 ground glass joints and taps.

8.1.1.3 Conical stopper 29/32, made of PTFE, provided with a ventilation piece.

8.1.1.4 Micro-syringe, capacity 50 µl.

8.1.1.5 Conical flasks with a conical 29/32 ground glass joint, capacity 150 ml.

8.1.2 Reagents

All reagents used shall be of analytical reagent quality.

8.1.2.1 Karl Fischer reagent (KFR) with a water equivalent between 3,5 and 4,5 mg/ml.

8.1.2.2 Methanol with a water content of less than 0,05 g/100 g.

8.1.2.3 Silica gel, freshly activated.

8.1.3 Standardization of Karl Fischer reagent

Transfer, by means of an automatic burette, about 25 ml of methanol into the titration vessel and titrate automatically with Karl Fischer reagent. Add, by means of a micro-syringe, 50 µl of water (V_w) and titrate again with Karl Fischer reagent (V_t). Carry out this procedure three times and calculate the mean value ($\overline{V}_t$). Repeat the standardization of Karl Fischer reagent on every working day.

8.1.4 Calculation of the water equivalent

The water equivalent E of the Karl Fischer reagent, expressed in milligrams H_2O per millilitre, is given by the equation

$$E = \frac{m_w}{\overline{V}_t}$$

where

m_w is the mass, in milligrams, of the volume of water (V_w) used for the standardization of the Karl Fischer reagent;

$\overline{V}_t$ is the mean volume, in millilitres, of the Karl Fischer reagent used for the titration of the water.

8.1.5 Procedure

Dismantle the holder and remove any sealing ring with forceps.

Remove the disc with forceps and fold twice.

Place the folded disc in a dry 150 ml conical flask.

Wipe the inner surface of the holder front with two quarters of the blank filter disc held with a pincette and put these into the flask.

Run a maximum of two portions of 20 ml of methanol into the flask. The folded disc shall be covered with solvent. In the case of a 92 mm glass fibre filter trap, 80 ml of methanol is necessary.

Close the flask as quickly as possible and shake on an electrical shaker for 20 min or leave for 16 h, ensuring that the disc does not disintegrate.

Measure the water content of this solution by injecting a suitably sized aliquot (e.g. 10 ml) into the titration vessel.

Prepare a number of blank holders, at least one before and one after the solution of the smoke particulate matter samples, and treat in exactly the same manner as above, taking the same precautions.

8.1.6 Calculation of the water content

The water content W of the total particulate matter for each trap, expressed in milligrams per cigarette, is given by the equation

$$W = \frac{(V - V_b) \times E \times V_k}{q \times V_a}$$

where

V is the volume, in millilitres, of the Karl Fischer reagent used for the titration of the total particulate matter solution;

V_b is the volume, in millilitres, of the Karl Fischer reagent used for the blank titration;

E is the water equivalent of the Karl Fischer reagent, in milligrams H_2O per millilitre;

— Water content (% by mass) of conditioned cigarettes (see ISO 6488).

— Average draw resistance of conditioned cigarettes.

— Diameter of cigarettes.

— Average number of puffs per cigarette for each channel to the nearest 0,1 puff.

— Total particulate matter (mg/cig.) for each channel to the nearest 0,1 mg.

— Dry particulate matter (mg/cig.) for each channel to the nearest 0,1 mg.

— Nicotine-free dry condensate (mg/cig.) for each channel to the nearest 0,1 mg.

Annex

Smoking plans

(This annex does not form an integral part of the International Standard.)

In the majority of cases the results of mechanical smoking permit a comparison of types of cigarettes (treatments). This comparison must be made according to a smoking plan established in advance; the smoking plan must take account of :

— the capacity and the variability of the smoking machine : number of channels;

— the capacity of the smoke traps : it determines the number of cigarettes to be smoked in each channel;

— the nature of the cigarettes : for those of high condensate yield it is prudent to reduce the number to be smoked in each channel; on the contrary for cigarettes which give very low levels of condensate the number must be raised;

— required precision : the results of smoking always give a certain variability; the distribution of the treatments in each smoking run and of the smoking runs in time must reduce the effects of uncontrolled or badly controlled factors (mechanical or personal); in general, the larger the test portion, the greater the precision.

The order of magnitude of the number N of cigarettes in a test portion is fixed for each type as a function of various factors, in particular :

— the precision sought;

— the time necessary for the smoking processes, itself related to the capacity of the machine.

The exact value to be selected for N, chosen in the ranges above (see 7.1) taking into account the preceding factors, is determined by calculation for each experiment taking into account the parameters which characterize it.

Also if

t denotes the number of types to be compared (treatments);

s denotes the number of smoking runs to be carried out;

c denotes the number of channels on the machine;

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

then the different parameters are related by the equation

$$t \times N = s \times c \times q$$

The examples of smoking plans proposed below illustrate the preceding remarks. They could correspond to the following objectives :

Example I Comparison of two types of cigarettes on one single channel smoking machine. The smoke trap can collect the condensate of five cigarettes.

Example II Comparison of three types of cigarettes on one single channel smoking machine. The smoke trap can collect the condensate of twenty cigarettes.

Example III Comparison of two types of cigarettes on one four channel smoking machine. The smoke trap can collect the condensate of five normal cigarettes. Test cigarettes having high condensate yield (e.g. above 30 mg per cigarette).

Example IV Comparison of seven types of cigarettes on one four channel smoking machine. The smoke trap can collect the condensate of five normal cigarettes. Test cigarettes having low condensate yield (e.g. in the neighbourhood of 5 mg per cigarette).

Example V Comparison of twenty types of cigarettes on one twenty channel smoking machine. The smoke trap can collect the condensate of five normal cigarettes. Higher precision required.

Example VI Comparison of five types of cigarettes on one twenty channel smoking machine. The smoke trap can collect the condensate of five normal cigarettes. Higher precision required.

Example I

Comparison of two types of cigarettes on one single channel smoking machine

Number of treatments	$t = 2$ (A, B)
Number of cigarettes in the test sample	$N = 40$
Number of cigarettes per channel	$q = 5$
Number of channels	$c = 1$
Number of smoking runs	$s = 16$ (1, 2, ... 16)

$$2 \times 40 = 16 \times 1 \times 5$$

The number N of cigarettes to be smoked is limited to 40 of each type, so that the duration of the smoking process is not too long. Each smoking run carries only one treatment. Distribute the runs in time while repeating the following sequence four times (k represents successive values 0, 4, 8 and 12) :

Runs	Treatments
$1 + k$	A
$2 + k$	B
$3 + k$	B
$4 + k$	A

Example II

Comparison of three types of cigarettes on one single channel smoking machine

Number of treatments	$t = 3$ (A, B, C)
Number of cigarettes in the test sample	$N = 60$
Number of cigarettes per channel	$q = 20$
Number of channels	$c = 1$
Number of smoking runs	$s = 9$ (1, 2, ... 9)

$$3 \times 60 = 9 \times 1 \times 20$$

Each smoking run carries only one treatment. The runs are distributed in time in an ordered fashion, e.g. by means of a matrix of the following type :

B A C
C B A
A B C

Run	1	2	3	4	5	6	7	8	9
Treatments	B	A	C	C	B	A	A	C	B

Example III

Comparison of two types of cigarettes on one four channel smoking machine

Number of treatments	$t = 2$ (A, B)
Number of cigarettes in the test sample	$N = 48$
Number of cigarettes per channel	$q = 3$
Number of channels	$c = 4$ (a, b, c, d)
Number of smoking runs	$s = 8$ (1, 2, ... 8)

$$2 \times 48 = 8 \times 4 \times 3$$