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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 anhydre et exempte de nicotine au moyen d'une machine à
fumer analytique de routine*



Reference number
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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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.

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*.

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*.

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.

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

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.

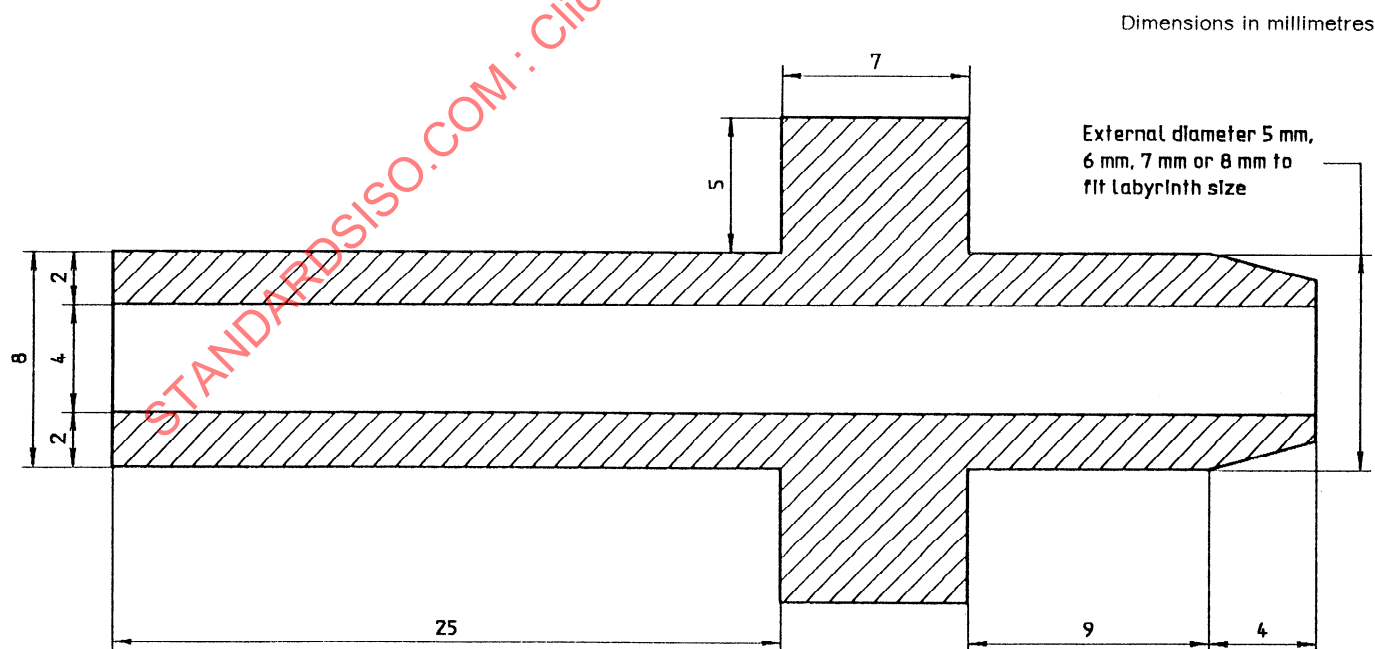


Figure 1 — Example of plastic insert for use during puff volume adjustment

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.

Zero the puff counters and light each cigarette at the beginning of its first puff. Should it be necessary to relight a cigarette, a hand-held electrical lighter may be used. When each butt mark has been reached, remove the burning coal from the cigarette and note the final reading of the puff counters. After the smoking process is complete, leave the cigarette butt in place for at least 30 s to enable deposition of any residual smoke in the trap.

NOTE 7 Avoid disturbance of the smoking by artificial removal of ash. Allow ash to fall naturally into the ashtray.

If required, new cigarettes shall be inserted immediately and the smoking process repeated until the predetermined number of cigarettes, in accordance with 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 (gloves shall be worn). Where necessary, remove the cigarette holder from the smoke trap.

Cover the front and back apertures of the trap with the sealing devices (5.9).

NOTE 8 It is recommended, particularly when plain cigarettes have been smoked, that the removal of the holder 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 disc.

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

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

Glass fibre filter pads of 44 mm diameter are capable of retaining up to 150 mg of total particulate matter (TPM) and pads of 92 mm diameter are capable of retaining 600 mg of TPM. If, during smoking, this mass is exceeded, the number of cigarettes shall be reduced and a calculation made to allow for the reduced number of cigarettes smoked.

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 of the smoke trap before smoking, in milligrams;
- m_1 is the mass of the smoke trap after smoking, in milligrams;
- q is the number of cigarettes smoked into the trap.

7.9 Treatment of total particulate matter

7.9.1 Extraction procedure

Remove the sealing devices from the smoke trap (gloves shall be worn), open it and remove the filter disc with forceps. Fold it twice, total particulate matter inwards, being careful to handle only the edge with forceps and gloved fingers. Place the folded disc in a dry conical flask (maximum 150 ml for 44 mm discs, maximum 250 ml for 92 mm discs). Pipette solvent (propan-2-ol containing the internal standards for both nicotine and water determinations) into the flask (20 ml for 44 mm discs or 50 ml for 92 mm discs) (see ISO 10315 and ISO 10362).

Wipe the inner surface of the filter holder front with two separate quarters of an unused conditioned filter disc and add these to the flask. Stopper the flask immediately and shake gently on an electric shaker for at least 20 min, ensuring that the disc does not disintegrate.

Follow the same procedure with each of the blank smoke traps used for the determination of water.

7.9.2 Water determination

Carry out the water determination in the supernatant solution in each flask in accordance with ISO 10362-1.

The dry particulate matter, D , expressed in milligrams per cigarette, for each trap is given by the equation

$$D = T - W$$

where

- T is the total particulate matter, in milligrams per cigarette;
- W is the water content in the total particulate matter, in milligrams per cigarette.

7.9.3 Nicotine determination

Carry out the determination of nicotine in the supernatant solution in each flask in accordance with ISO 10315.

The nicotine-free dry particulate matter, G , expressed in milligrams per cigarette, for each trap is given by the equation

$$G = D - H_{\text{nic}}$$

where

D is the dry particulate matter, in milligrams per cigarette;

H_{nic} is the nicotine content in the total particulate matter, in milligrams per cigarette.

8 Test report

The test report shall show the method used and the results obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the results. The test report shall include all details required for complete identification of the sample. If appropriate, the information given in 8.1 and 8.4 shall be recorded.

8.1 Characteristic data about the cigarette

- Cigarette identification. In the case of a commercial cigarette this should include
 - a) name of manufacturer, country of manufacture;
 - b) product name;
 - c) date of sampling;
 - d) place of purchase or sampling;
 - e) kind of sampling point;
 - f) sampling point (e.g. address of retail outlet or machine number);
 - g) packet number (of that product sampled that day);
 - h) marks on any tax stamp;
 - i) printed smoke yields (if any);
 - j) length of cigarette;
 - k) length of filter;
 - l) length of overwrap.

8.2 Sampling

- Type of sampling procedure.

- Number of cigarettes in laboratory sample.
- Date and location of purchase.

8.3 Description of test

- Reference to this International Standard.
- Date of test.
- Type of smoking machine used.
- Type of smoke trap used.
- Total number of cigarettes smoked in the entire determination on that cigarette type.
- Number of cigarettes smoked into each smoke trap.
- Butt length.
- Room temperature (in degrees Celsius) during smoking operation.
- Relative humidity (in percent) during smoking operation.
- Atmospheric pressure (in kilopascals) during smoking operation.

8.4 Test results

The expression of the laboratory data depends on the purpose for which the data are required, and the level of laboratory precision. Confidence limits shall be calculated and expressed on the basis of the laboratory data before any rounding has taken place.

- Average length of the cigarettes to the nearest 0,1 mm.
- Average length of the filter to the nearest 0,1 mm.
- Average length of the overwrap to the nearest 0,1 mm.
- Butt length to which cigarettes were smoked to the nearest 0,1 mm.
- Average length of tobacco portion smoked to the nearest 0,1 mm.
- Average diameter of the cigarettes (in millimetres).
- Average draw resistance of the conditioned cigarettes.

- Average mass (milligrams per cigarette) of the conditioned cigarettes selected for the smoking operation.
- Water content (m/m) of the conditioned cigarettes (see ISO 6488).
- Average number of puffs per cigarette for each channel to the nearest 0,1 puff.
- Total particulate matter (milligrams per cigarette) for each channel to the nearest 0,1 mg and the average per cigarette to the nearest 1 mg.
- Dry particulate matter (milligrams per cigarette) for each channel to the nearest 0,1 mg and the average per cigarette to the nearest 1 mg.
- Nicotine-free dry particulate matter (milligrams per cigarette) for each channel to the nearest 0,1 mg and the average per cigarette to the nearest 1 mg.

9 Repeatability and reproducibility

A major international collaborative study involving 30 laboratories and 6 samples conducted in 1990 showed the following values for repeatability (r) and reproducibility (R) of this method.

The difference between two single results found on matched cigarette samples by one operator using the same apparatus within the shortest feasible time interval will exceed the repeatability value (r) on average not more than once in 20 cases in the normal and correct operation of the method.

Single results on matched cigarette samples reported by two laboratories will differ by more than the reproducibility value (R) on average not more than once in 20 cases in the normal and correct operation of the method.

Data analysis gave the estimates as summarized in table 1.

Table 1 — Estimates given by data analysis

Mean yield of NFDPM	Repeatability conditions	Reproducibility conditions
mg	r	R
0,82	0,40	0,60
1,61	0,52	0,74
3,31	0,52	0,90
7,70	0,88	1,51
12,61	1,06	1,70
17,40	1,19	1,84

For the purpose of calculating r and R , one test result was defined as the mean yield obtained from smoking 20 cigarettes in a single run.

For further details of the interaction of r and R with other factors, see CORESTA Report 91/1.

The subject of tolerances due to sampling is dealt with in ISO 8243.

Annex A (informative)

Smoking plans

In the majority of cases the results of mechanical smoking permit a comparison of types of cigarettes (treatments). This comparison should be made according to a smoking plan established in advance; the smoking plan should 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;
- 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 should 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 1

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

EXAMPLE 2

Comparison of three types of cigarettes on one single-channel smoking machine. The smoke trap can collect the condensate of 20 cigarettes.

EXAMPLE 3

Comparison of two types of cigarettes on one four-channel smoking machine. The smoke trap can collect the condensate of five normal cigarettes. As the test cigarettes have high condensate yield (e.g. above 30 mg per cigarette) the number smoked should be reduced to three.

EXAMPLE 4

Comparison of 20 types of cigarettes on one 20-channel smoking machine. The smoke trap can collect the condensate of five normal cigarettes. Higher precision required.

EXAMPLE 5

Comparison of five types of cigarettes on one 20-channel smoking machine. The smoke trap can collect the condensate of five normal cigarettes. Higher precision required.

EXAMPLE 1

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	$s = 16(1, 2, \dots, 16)$
	$2 \times 40 = 16 \times 1 \times 5$

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 2

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	$s = 9(1, 2, \dots, 9)$
	$3 \times 60 = 9 \times 1 \times 20$

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

EXAMPLE 3

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 $s = 8(1, 2, \dots, 8)$

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

Allocate the smoking channels to the two treatments utilizing the matrix below, which is constructed for four treatments but which is easily adapted to the case of two treatments by identifying A with C on the one hand and B with D on the other. (In general, all matrices of dimensions g can be utilized for a number of treatments which are sub-multiples of g .)

		A	B	C	D				
		D	C	A	B				
		B	A	D	C				
		C	D	B	A				
	Channel		a	b	c	d			
Run									
1			A	B	A	B			
2			B	A	A	B			
3			B	A	B	A			
4			A	B	B	A			
5			A	B	A	B			
6			B	A	A	B			
7			B	A	B	A			
8			A	B	B	A			

In each smoking run, two channels are allocated to each treatment. For example, in run 6:

cigarette A is smoked in channels b and c,

cigarette B is smoked in channels a and d.

Each type is smoked four times in each of the four channels.

EXAMPLE 4

Comparison of 20 types of cigarettes on one 20-channel smoking machine

Number of treatments $t = 20(A, B, \dots T)$

Number of cigarettes in the test sample $N = 100$

Number of cigarettes per channel $q = 5$

Number of channels $c = 20(a, b, \dots t)$

Number of smoking runs $s = 20(1, 2, \dots 20)$

$$20 \times 100 = 20 \times 20 \times 5$$

Allocate the smoking channels to the 20 treatments utilizing the matrix below:

Channel Run	a	b	c	d	e	f	g	h	i	j	k	l	m	n	o	p	q	r	s	t
1	A	B	C	D	E	F	G	H	I	J	K	L	M	N	O	P	Q	R	S	T
2	D	N	B	J	A	R	H	L	C	O	Q	F	S	K	T	I	E	P	M	G
3	I	A	M	E	K	Q	O	F	H	B	R	J	G	P	C	N	L	S	T	D
4	K	C	I	N	Q	H	M	A	J	F	S	R	B	O	G	L	D	T	P	E
5	B	J	H	S	F	M	P	K	N	A	T	C	R	Q	E	O	G	L	D	I
6	H	D	Q	M	C	S	F	P	T	G	O	E	K	A	I	J	B	N	R	L
7	E	L	G	Q	D	P	K	T	M	S	A	I	N	F	R	C	O	H	J	B
8	M	H	D	P	L	G	S	C	K	T	F	O	J	R	B	Q	I	E	N	A
9	L	Q	F	B	J	O	N	G	R	C	P	K	H	S	D	T	A	I	E	M
10	G	R	L	T	N	D	A	J	Q	H	E	B	O	M	K	F	S	C	I	P
11	N	E	T	I	O	B	J	R	F	K	C	G	L	D	H	M	P	Q	A	S
12	C	O	K	F	B	J	Q	N	A	S	P	M	S	I	E	L	H	T	D	G
13	F	P	A	O	G	C	B	M	S	D	L	N	T	I	J	E	R	K	H	Q
14	P	T	R	H	S	N	D	E	G	I	J	M	F	L	Q	B	K	A	O	C
15	R	K	P	G	T	E	I	O	L	N	H	D	Q	C	S	A	J	M	B	F
16	T	G	E	C	I	K	L	S	O	M	D	Q	P	H	A	R	N	B	F	J
17	S	F	N	R	H	L	T	B	E	Q	I	A	C	J	P	D	M	G	K	O
18	Q	M	O	L	P	T	E	I	D	R	G	H	A	B	N	S	F	J	C	K
19	O	S	J	A	R	I	C	Q	P	E	B	T	D	G	M	K	H	F	L	N
20	J	I	S	K	M	A	R	D	B	L	N	P	E	T	F	G	C	O	Q	H

All the treatments are represented in each smoking run. Overall, each treatment is smoked once in each of the 20 channels.

EXAMPLE 5

Comparison of five types of cigarettes on one 20-channel smoking machine

Number of treatments $t = 5(A, B, C, D, E)$

Number of cigarettes in the test sample $N = 200$

Number of cigarettes per channel $q = 5$