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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

Reference number
ISO 4387:2000(E)

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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.

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.

If for any reason test samples are to be kept for longer than 10 days before conditioning, 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

The following data may be required in the test report:

a) total length of the cigarette;

b) nominal diameter, determined in accordance with ISO 2971;

c) draw resistance of the cigarette, determined in accordance with ISO 6565;

d) average mass of the conditioned cigarettes selected for the smoking operation (in milligrams per cigarette);

e) water content (as a mass fraction) of the conditioned cigarettes, determined in accordance with ISO 6488-1.

7.6 Smoking and collection of particulate matter

7.6.1 Smoking plan

Choose a smoking plan; examples of plans are given in informative annex A.

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

The plan should include the use of a test portion of monitor test pieces. The test pieces are included in the plan as if they were a type of cigarette and prepared and smoked as in 7.6.4, 7.7, 7.8 and 7.9.

7.6.2 Preparation of smoke traps and cigarette holders

For all operations, the operator shall prevent contamination from the fingers by wearing gloves of a suitable material (5.9).

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 3308:2000, 4.8). Weigh the assembled smoke traps to the nearest 0,1 mg.

Because of absorption of water by smoke traps and solvent, it is necessary to 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

7.6.3.1 General

If necessary, replace any protective filters on the machine. Switch on the machine and allow it 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.

The puff volume should be checked if it is suspected that the smoking machine is subject to a large change in temperature during use.

7.6.3.2 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.

7.6.3.3 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.

7.6.3.4 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 1 kPa $\pm$ 5 % to the cigarette holder of the smoking machine channel under test. Before use for a series of measurements, wet the instrument twice with detergent solution and then allow it to drain for a period of between 30 s and 45 s.

The bubble flow meter shall contain a mass fraction of 15 % aqueous solution of a surface active agent. Teepol L ® [1)] has been found to be satisfactory. The concentration of Teepol as purchased must be known before carrying out further dilution.

Fit the prepared smoking trap or traps and cigarette holders onto the machine. Attach a plastic insert of an appropriate size for the labyrinth seals in the cigarette holder to the resistance in the tube from 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 (35,0 $\pm$ 0,3) 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.

7.6.4 Procedure for smoking run

Insert the conditioned cigarettes from the test portion into the cigarette holders so that the butt end impinges upon the perforated disc (washer) fitted within the filter trap. 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.

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1) 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 this product. Equivalent products may be used if they can be shown to lead to the same results.

Ensure that the cigarettes are positioned correctly so that the axes of the cigarettes coincide with the axes 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 of (48 ± 4) tex 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.

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.

Avoid disturbance of the smoking by artificial removal of ash. Allow the 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. Immediately begin the determination of total particulate matter as described in 7.7.

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 (5.8).

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 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 TPM content, m_{TPM}, for each channel, expressed in milligrams per cigarette, is given by the equation:

$$m_{\mathrm{TPM}} = \frac{m_1 - m_0}{q}$$

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 an appropriately shaped dry flask (maximum 150 ml for 44 mm discs, maximum 250 ml for 92 mm discs). 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. 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 10362-1).

Stopper the flask immediately and shake gently on an electric shaker for at least 20 min, ensuring that the disc does not disintegrate. The shaking time should be adjusted to ensure full extraction of the nicotine and water in the particulate matter.

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

7.9.2 Determination of water

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

The DPM content, m_{DPM}, for each trap, expressed in milligrams per cigarette, is given by the equation:

$$m_{DPM} = m_{TPM} - m_W$$

where

m_{TPM} is the TPM content, in milligrams per cigarette;

m_W is the water content in the TPM, in milligrams per cigarette.

7.9.3 Determination of nicotine

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

The NFDPM content, m_{NFDPM}, for each trap, expressed in milligrams per cigarette, is given by the equation:

$$m_{NFDPM} = m_{DPM} - m_N$$

where

m_{DPM} is the DPM content, in milligrams per cigarette;

m_N is the nicotine content in the TPM, 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 below in a) to d) shall be recorded.

a) Characteristic data about the cigarette

All details necessary for the identification of the cigarettes smoked shall be given. In the case of commercial cigarettes this should include:

— name of manufacturer and country of manufacture,

— product name,

— packet number (of the product sampled that day),

— marks on any tax stamp,

— printed smoke yields (if any),

— length of cigarette,

— length of filter,

— length of overwrap.

b) Data about sampling

— type of sampling procedure,

— date of sampling,

— place of purchase or sampling,

— kind of sampling point,

— sampling point (e.g. address of retail outlet or machine number),

— number of cigarettes in laboratory sample.

c) 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,

— 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.

d) 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. Details should include the following:

— average length of the cigarettes to the nearest 0,1 mm,

— average length of the filter to the nearest 0,5 mm,

— average length of the overwrap to the nearest 0,5 mm,

— butt length to which cigarettes were smoked,

— average diameter of the cigarettes to the nearest 0,01 mm,

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

— TPM content (in milligrams per cigarette) for each channel to the nearest 0,1 mg, and the average per cigarette to the nearest 1 mg,

— DPM content (in milligrams per cigarette) for each channel to the nearest 0,1 mg, and the average per cigarette to the nearest 1 mg,

— NFDPM content (in 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 the repeatability limits (r) and the reproducibility limits (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 limit (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 limit (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

Values in milligrams per cigarette

Mean value m_{NFDPM}	Repeatability limit r	Reproducibility limit 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 following:

a) the capacity and the variability of the smoking machine: number of channels;

b) the capacity of the smoke traps: this determines the number of cigarettes to be smoked in each channel;

c) the nature of the cigarettes: for those of high condensate yield it is prudent to reduce the number to be smoked in each channel;

d) 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, which is 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.

The different parameters are related by the equation

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

where

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

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

c is the number of channels on the machine;

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

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

a) EXAMPLE 1: Comparison of two types of cigarettes on one single-channel smoking machine. The smoke trap can collect the condensate of five cigarettes.

b) EXAMPLE 2: Comparison of three types of cigarettes on one single-channel smoking machine. The smoke trap can collect the condensate of 20 cigarettes.

c) 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.

d) 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.

e) 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, ... 16)

Thus testing 80 cigarettes $2 \times 40 = 16 \times 1 \times 5$

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 four times the sequence shown in Table A.1 (k represents successive values 0, 4, 8 and 12):

Table A.1

Run	Treatment
$1 + k$	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, ... 9)

Thus testing 180 cigarettes $3 \times 60 = 9 \times 1 \times 20$

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
Treatment	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, ... 8)

Thus testing 96 cigarettes $2 \times 48 = 8 \times 4 \times 3$

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 Run	a	b	c	d
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, ... T)

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

Number of cigarettes per channel $q = 5$

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

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

Thus testing 200 cigarettes $20 \times 100 = 20 \times 20 \times 5$

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	P	M	S	I	E	L	H	T	D	G	R
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 $q = 5$

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

Number of smoking runs $s = 10$ (1, 2, ... 10)

Thus testing 1 000 cigarettes $5 \times 200 = 10 \times 20 \times 5$

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

D	B	E	A	C
A	D	B	C	E
B	A	C	E	D
C	E	D	B	A
E	C	A	D	B

Channel	a	b	c	d	e	f	g	h	i	j	k	l	m	n	o	p	q	r	s	t
Run																				
1	D	B	E	A	C	C	E	D	A	B	E	C	B	A	D	B	D	A	C	E
2	A	D	B	C	E	A	C	E	B	D	C	E	A	D	B	A	B	D	E	C
3	B	A	C	E	D	E	B	C	D	A	A	D	C	B	E	D	E	C	A	B
4	C	E	D	B	A	B	D	A	E	C	D	B	E	C	A	E	C	B	D	A
5	E	C	A	D	B	D	A	B	C	E	B	A	D	E	C	C	A	E	B	D
6	C	A	E	B	D	B	A	D	E	C	D	A	B	C	E	E	C	A	D	B
7	E	C	B	D	A	D	B	E	C	A	B	D	A	E	C	C	E	D	B	A
8	D	E	C	A	B	A	D	C	B	E	E	B	C	D	A	B	A	C	E	D
9	A	B	D	E	C	C	E	A	D	B	A	C	E	B	D	A	D	B	C	E
10	B	D	A	C	E	E	C	B	A	D	C	E	D	A	B	D	B	E	A	C

In each smoking run, each treatment is smoked in four channels. For example, in run 7:

— cigarette A is smoked in channels e, j, m, t,

— cigarette B is smoked in channels c, g, k, s,

— cigarette C is smoked in channels b, i, o, p,

— cigarette D is smoked in channels d, f, l, r,

— cigarette E is smoked in channels a, h, n, q.

Overall, each treatment is smoked twice in each of the 20 channels.

NOTE It is not always possible to smoke each treatment equally in each of the channels. In the present case, if the number of cigarettes in the test sample were 160 it would be necessary to smoke 8 runs. One could distribute the cigarettes as seen above in runs 1 to 8. Then each type would be smoked once or twice in each of the 20 channels.

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