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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Cigarettes — Determination of total and dry particulate matter using a routine analytical cigarette-smoking machine — Electrostatic 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 électrostatique

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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 8453 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.

Cigarettes — Determination of total and dry particulate matter using a routine analytical cigarette-smoking machine — Electrostatic 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 an electrostatic smoke trap.

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.

4 Principle

Smoking of the test cigarettes in an automatic smoking machine and collection of the mainstream smoke by an electrostatic smoke trap. Gravimetric determination of the mass of

1) At present at the stage of draft.

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 Electrostatic smoke trap (see figure), made of an air-tight and chemically inert material, preferably transparent, and consisting of

- 1) precipitation tube;
- 2) holding device for precipitation tube;
- 3) cigarette holder;
- 4) central electrode (positive);
- 5) screw cap;
- 6) seal (e.g. O-ring seal);
- 7) insulating socket;
- 8) outlet for gas phase to suction source;
- 9) earthing coil;
- 10) high voltage inlet (up to 25 kV);
- 11) earthing connection.

The precipitation tube (1) shall be carefully brought to equilibrium with the ambient conditions before inserting into the smoking machine. After it has been inserted into the seal (6) and connected with the earthing coil (9), the high voltage is switched on.

The smoke trap shall be capable of quantitatively retaining at least 99,9 % of the mainstream smoke produced by the cigarette without loss of particulate matter.

To check the efficiency of smoke precipitation by the electrostatic trap, a glass fibre filter shall be attached between the electrostatic smoke trap and the piston. This additional trap shall not retain more than 0,05 % of the total particulate matter.

The smoke trap 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.

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 shall 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 which is 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 $N > 100$. 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 100.

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 $Q/P \geq C \times q$, 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

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

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 ($\geq 2C \times N$), 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 $Q/P \geq 2C \times q$ and $Q/P < 2C \times q$.

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 be 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 electrostatic smoke trap

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

The precipitation tube shall be brought to equilibrium with the ambient conditions before use. Usually 10 min is sufficient for equilibration to the conditioning atmosphere as defined in ISO 3402. Before connecting the glass tube to the smoking machine, weigh it to the nearest 0,5 mg. When the tube has been inserted and earthed, set the high voltage to 20 kV.

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 $35 \pm 0,1$ 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, but no more than three, 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 smoke trap.

Immediately weigh the smoke traps to the nearest 0,5 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 (\bar{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 H₂O per millilitre, is given by the equation

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

where

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

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

8.1.5 Procedure

Fit the precipitation tube of the electrostatic smoke trap charged with smoke particulate matter onto a dry conical flask of capacity 150 ml and make an airtight closure by means of a sleeve cap with tap.

Introduce through the aperture of the tap, by means of an automatic burette, a maximum of two portions of 20 ml of methanol in such a way that the precipitation tube is rinsed. Close the tap and shake until the smoke particulate matter is completely dissolved and the solution is mixed. Disconnect the precipitation tube from the conical flask, excluding atmospheric moisture, and close the flask immediately by means of the PTFE stopper.

For rinsing, transfer by means of an automatic burette three portions of about 4 ml of the smoke particulate matter solution from the conical flask into the titration vessel and titrate. Transfer in the same manner 10 ml of the smoke particulate matter solution (V_a) into the titration vessel. Determine the volume of Karl Fischer reagent used for the titration (V).

Treat a blank trap stored under the same conditions as those used for smoking in exactly the same manner as described above to determine the volume of Karl Fischer reagent corresponding to the blank titration (V_b).

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 H₂O per millilitre;

q is the number of cigarettes smoked into each smoke trap;

V_k is the volume, in millilitres, of methanol used for dissolving the total particulate matter;

V_a is the volume, in millilitres, of the total particulate matter solution used for the titration.

8.2 Gas chromatographic method

8.2.1 Apparatus

Gas chromatography equipment, comprising for example a dual-column gas chromatograph equipped with

- a) a **Katharometer detector**;
- b) **column packing material** : a porous polymer composed of chains of ethylvinylbenzene cross-linked by divinylbenzene¹⁾, in the form of beads sized between 149 and 177 μm ;
- c) **helium** of high purity as carrier gas;
- d) **conical flasks** with 29/32 ground glass joint, capacity 150 ml.

8.2.2 Solvent

200 : 1 (V/V) mixture of **propan-2-ol** and **ethanol** as internal standard (analytical grade).

8.2.3 Calibration of gas chromatograph

Weigh four dry stoppered 150 ml conical flasks [8.2.1 d)]. Inject 10, 20, 30 and 40 μl of water into the flasks and reweigh. Pipette 20 ml portions of solvent (8.2.2) into each flask and a similar empty flask. Shake all the flasks thoroughly to disperse the water. Carry out duplicate determinations using appropriate injections of each solution and calculate the ratio of water peak area to ethanol peak area. Plot a graph of the peak area ratio against weighed water content and construct the best linear fit for all the points.

NOTE — If an integrator is not available, the water peak height : ethanol peak height ratio may be used instead.

8.2.4 Calculation of calibration factor

The calibration factor F is given by the equation

$$F = \frac{S}{A_t - A_0}$$

where

S is the mass, in milligrams, of the volume of water used for the top standard (40 μl);

A_t is the peak area or peak height ratio of the intersect between the regression line and the top standard;

A_0 is the peak area or peak height ratio of the intersect between the regression line and the blank.

8.2.5 Procedure

Put the precipitation tube of the electrostatic smoke trap charged with smoke particulate matter onto a dry conical flask of capacity 150 ml and make an airtight closure by means of a sleeve cap with tap.

Introduce through the aperture of the tap, by means of an automatic burette, two portions of 10 ml of solvent (8.2.2) in such a way that the precipitation tube is rinsed. Close the tap and shake until the smoke particulate matter is completely dissolved and the solution is mixed. Disconnect the precipitation tube from the conical flask, excluding atmospheric moisture, and close the flask immediately by means of a PTFE stopper.

Measure the water content of this solution by injecting suitably sized aliquots into the gas chromatograph.

Prepare a blank trap and treat in exactly the same manner as above, using the same precautions.

For each sample, calculate the ratio of water peak : ethanol peak by area or height, as in 8.2.3.

8.2.6 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

$$W = \frac{(A_1 - A_2) \times F}{q}$$

where

A_1 is the peak area or peak height ratio of the sample;

A_2 is the peak area or peak height ratio of the blank;

F is the calibration factor;

q is the number of cigarettes smoked into each smoke trap.

8.3 Calculation of dry particulate matter

This is calculated for each trap from the equation

$$D = T - W$$

1) For example Porapak Q or Chromosorb 102 or similar material.

where

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

T is the total particulate matter, in milligrams per cigarette;

W is the water content of the total particulate matter, in milligrams per cigarette.

9 Determination of nicotine-free dry particulate matter

9.1 Procedure

Measure the nicotine alkaloids content in accordance with ISO 3400.

9.2 Calculation

This is calculated for each trap from the equation

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

where

G is the nicotine-free dry particulate matter, in milligrams per cigarette;

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

H_{nic} is the nicotine alkaloids in the total particulate matter, in milligrams per cigarette, measured in accordance with ISO 3400.

10 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. Where appropriate, record the information in 10.1 to 10.4.

10.1 Characteristic data about the cigarette

— Cigarette identification. In the case of a commercial cigarette this should include :

- a) product name;
- b) date of sampling;
- c) place of purchase or sampling;
- d) kind of sampling point;
- e) sampling point (e.g. address of retail outlet or machine number);
- f) packet number (of that product sampled that day);
- g) laboratory;
- h) marks on any tax stamp.

- Name of manufacturer, country of manufacture.
- Length of cigarette.
- Length of filter.
- Length of overwrap.

10.2 Sampling

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

10.3 Description of test

- 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 (°C) during smoking operation.
- Relative humidity (%) during smoking operation.
- Atmospheric pressure (N/m²) during smoking operation.
- Type of test procedures used.

10.4 Test results

- Average mass (mg/cig.) of conditioned cigarettes selected for smoking operation.
- Average mass of tobacco (mg/cig.) in conditioned cigarettes.
- 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.