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Cigarettes — Determination of water in smoke condensates —

Part 1:
Gas-chromatographic method

Cigarettes — Dosage de l'eau dans les condensats de fumée —

Partie 1: Méthode par chromatographie en phase gazeuse

Reference number
ISO 10362-1:1999(E)

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Foreword

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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 part of ISO 10362 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 10362-1 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

This second edition cancels and replaces the first edition (ISO 10362-1:1991), which has been editorially revised.

ISO 10362 consists of the following parts, under the general title *Cigarettes — Determination of water in smoke condensates:*

- *Part 1: Gas-chromatographic method*
- *Part 2: Karl Fischer method*

Annex A of this part of ISO 10362 is for information only.

Introduction

This part of ISO 10362 may be considered as part of a set produced by ISO/TC 126 which describes the determination of total and nicotine-free dry particulate matter (NFDPM) in cigarette smoke condensate. The set comprises:

ISO 3308, ISO 3402, ISO 4387, ISO 8243, ISO 10315 and ISO 10362-1.

Annex A provides information about the use of this method in conjunction or simultaneously with the gas-chromatographic method of nicotine determination described in ISO 10315.

A bibliography is provided.

Cigarettes — Determination of water in smoke condensates —

Part 1:
Gas-chromatographic method

1 Scope

This part of ISO 10362 specifies a method for the gas-chromatographic determination of water in cigarette smoke condensates. The smoking of cigarettes and collection of mainstream smoke are normally carried out in accordance with ISO 4387. However, the method of this part of ISO 10362 is applicable to the determination of water in smoke condensates obtained by non-standard smoking.

NOTE In countries not in a position to use the gas-chromatographic method, the determination of water in smoke condensates should be performed by a Karl Fischer method (ISO 10362-2). In such cases, values obtained for water in smoke condensate may be used with the addition of a note made in the expression of the results.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this part of ISO 10362. For dated references, subsequent amendments to, or revisions of, this publication do not apply. However, parties to agreements based on this part of ISO 10362 are encouraged to investigate the possibility of applying the most recent edition of the normative document 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 4387, *Cigarettes — Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine.*

3 Principle

The smoke condensate from the mainstream smoke is dissolved in a solvent containing an internal standard. The water content of an aliquot of the solution is determined by gas chromatography, and the water content of the whole of the smoke condensate is calculated.

4 Reagents

Use only reagents of recognized analytical reagent grade.

4.1 Carrier gas: helium or nitrogen (see note in 6.2).

4.2 Propan-2-ol, with maximum water content of 1,0 mg/ml.

4.3 Internal standard: ethanol, or methanol (of purity at least 99 %).

4.4 Extraction solvent: propan-2-ol (4.2) containing an appropriate concentration of internal standard (4.3), normally 5 ml/l.

Solvent not stored in a temperature-controlled laboratory shall be allowed to equilibrate to (22 ± 2) °C before use.

4.5 Reference substance: distilled water or deionized water.

4.6 Calibration solutions

Prepare a series of at least four calibration solutions whose concentrations of added water cover the range expected to be found in the test portion (usually up to 4 mg/ml) by adding weighed amounts of water (4.5) to the solvent (4.4). One of these calibration solutions shall be the solvent with no added water (solvent blank).

To prevent water being absorbed, the bulk solvent container shall be fitted with a water trap and all solutions shall be kept sealed. The solvent shall be stirred continuously to ensure the homogeneity of the water concentration in the solvent. The calibration solutions shall be made up using an extraction solvent from the same batch used in 6.1.

It is recommended that the calibration solutions be made up at least each week.

5 Apparatus

Usual laboratory apparatus and, in particular, the following items.

5.1 Gas-chromatograph, equipped with a thermal conductivity detector, recorder and integrator or other suitable data-handling equipment.

Glassware and septa for vials should be stored in a desiccator until use.

5.2 Column, of internal diameter between 2 mm and 4 mm and preferably of length 1,5 m to 2 m.

Stationary phase: Porapak Q[1] 150 µm (100 mesh) to 190 µm (80 mesh).

The column is preferably made of deactivated stainless steel but other materials such as glass or nickel may be used. Alternative stationary phases, such as Porapak QS[1] or Chromosorb 102[1], may be used.

5.3 Dispensing system, preferably automated, capable of delivering the required volume of solvent (4.4).

The dispensing system should be flushed prior to use by dispensing a volume of solvent of at least 50 ml which will then be rejected.

6 Procedure

6.1 Test portion

Prepare the test portion by dissolving the smoke condensate obtained by the machine smoking of a known number of cigarettes in a fixed volume of the solvent (4.4) of 20 ml for 44 mm discs or 50 ml for 92 mm discs, ensuring that the disc is fully covered. The volume may be adjusted to give a concentration of water appropriate for the calibration graph (see 6.3) provided that there is an adequate volume for effective extraction of the smoke condensate. For standard smoking, see ISO 4387.

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1) Porapak Q, Porapak QS and Chromosorb 102 are trade names of examples of suitable products available commercially. This information is given for the convenience of users of this part of ISO 10362 and does not constitute an endorsement by ISO of these products.

6.2 Setting up the apparatus

Set up the apparatus and operate the gas chromatograph (5.1) in accordance with the manufacturer's instructions. Ensure that the peaks for water, the internal standard and solvent are well resolved, the analysis time being about 4 min. Condition the system just prior to use by injecting a 2 µl aliquot of the extraction solvent as a primer.

Suitable operating conditions are as follows:

- column temperature: 170 °C (isothermal);
- injection temperature: 250 °C;
- detector temperature: 250 °C;
- carrier gas: helium at a flow rate of about 30 ml/min;
- injection volume: 2 µl.

NOTE Nitrogen may also be used as an alternative carrier gas if the detector sensitivity is sufficiently high.

6.3 Calibration of the gas chromatograph

Inject an aliquot (2 µl) of each of the calibration solutions (4.6) into the gas chromatograph. Record the peak areas (or heights) of the water and internal standard (4.3). Carry out the determination at least twice.

Calculate the ratio of the water peak to the internal standard peak from the peak area (or height) data for each of the calibration solutions including the solvent blank. Plot the graph of the concentrations of added water in accordance with the area ratios or calculate a linear regression equation from these data. Use the linear regression equation.

Perform this full calibration procedure daily. In addition, inject an aliquot of an intermediate concentration standard after every 20 sample determinations. If the calculated concentration for this solution differs by more than 5 % from the original value, repeat the full calibration procedure.

NOTE The regression line does not pass through zero due to water present in the extraction solvent.

If the water content of the solvent exceeds 1,0 mg/ml, the batch should be rejected.

6.4 Blank test

Due to the 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 including filters (at least 2 per 100 cigarettes smoked) in the same manner as that used for smoke collection. Place them near the smoking machine during smoking and extract and analyse them together with the smoke samples.

6.5 Determination

Inject aliquots (2 µl) of the test portion from the smoke traps (see 6.1) and blank traps (see 6.4). Calculate the ratio of the water peak/internal standard peak from the peak area (or height) data.

Carry out the determination at least twice under identical conditions. Calculate the mean value of the ratio from the replicate determinations.

NOTE Where results are obtained from a number of separate channels of smoking and where an autosampler is used, a single aliquot portion from the smoke traps is considered adequate.

7 Expression of results

Calculate the water concentration of the smoke trap and blank trap extracts using the graph or linear regression equation prepared in 6.3.

The water content of the smoke, m_W, in milligrams per cigarette, is given by the equation:

$$m_W = \frac{\rho_{WS} - \rho_{WB}}{q} \cdot V_{ES}$$

where

ρ_{WS} is the concentration of water in the sample smoke trap, in milligrams per millilitre;

ρ_{WB} is the concentration of water in the blank smoke trap, in milligrams per millilitre;

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

V_{ES} is the volume of extraction solvent in which the contents of the smoke trap were dissolved.

Express the test results in milligrams per cigarette for each channel to the nearest 0,01 mg, and the average per cigarette to the nearest 0,1 mg.

8 Repeatability and reproducibility

A major international collaborative study involving 30 laboratories and 6 samples, conducted in 1990, showed that when cigarettes are smoked in accordance with ISO 4387 and the resulting smoke solutions are analysed by this method, the following values for the repeatability limits (r) and the reproducibility limits (R) are obtained.

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

Mean value m_W mg per cigarette	**Repeatability limit** r mg per cigarette	**Reproducibility limit** R mg per cigarette
0,083	0,154	0,241
0,153	0,228	0,353
0,338	0,272	0,381
0,962	0,407	0,734
1,595	0,561	0,935
3,187	0,908	1,680

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.

9 Test report

The test report shall give the water content per cigarette smoked and the method used, and shall include all conditions which may affect the result (e.g. atmospheric test conditions during smoking). It shall also give all details necessary for the identification of the cigarettes smoked.

Annex A
(informative)

Use of this method in conjunction with the method of nicotine determination

This method can be used in conjunction with the method for the gas-chromatographic determination of nicotine in smoke condensates specified in ISO 10315. This may be carried out as follows:

- addition of an appropriate quantity of the internal standard specified for the nicotine determination in the solvent described in 4.4;
- injection of an aliquot of the smoke condensate solution onto a column for nicotine analysis, which is connected to a flame ionization detector, as well as onto the water column and detector described in this method.

A simultaneous automated analysis of nicotine and water may be achieved by using a splitting system or an auto-sampler with two injection positions. When determining nicotine and water from the same sample sequentially, the water determination is performed first to prevent absorption of water by the sample affecting the final result.

Bibliography

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

[2] ISO 3402, *Tobacco and tobacco products — Atmosphere for conditioning and testing.*

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

[4] ISO 10315, *Cigarettes — Determination of nicotine in smoke condensates — Gas-chromatographic method.*

[5] ISO 10362-2, *Cigarettes — Determination of water in smoke condensates — Part 2: Karl Fischer method.*

[6] CORESTA Report 91/1, *Internal Bulletin of Cooperation of the centre for scientific research relative to tobacco*, 1991-1, ISSN 0525-6240.

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