

# INTERNATIONAL STANDARD

**ISO**  
**10362-1**

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## **Cigarettes — Determination of water in smoke condensates —**

### **Part 1:**

Gas-chromatographic method

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

ISO 10362-1:1991

*Partie 1. Méthode par chromatographie en phase gazeuse*

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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.

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 10362-1 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*, in collaboration with the Cooperation Centre for Scientific Research *Relating to Tobacco* (CORESTA).

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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.

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## 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 3308:1991<sup>1)</sup>, *Routine cigarette-smoking machine — Definitions and standard conditions.*

ISO 3402:1991, *Tobacco and tobacco products — Atmosphere for conditioning and testing.*

ISO 4387:1991<sup>1)</sup>, *Cigarettes — Determination of total and nicotine free dry particulate matter using a routine analytical smoking machine.*

ISO 8243:1991<sup>1)</sup>, *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.*

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.

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1) To be published

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# 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 is applicable to the determination of water in smoke condensate obtained by non-standard smoking.

NOTE 1 In countries not in a position to use the gas-chromatographic method, the determination of water in smoke condensate should be performed by a Karl Fischer method. 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 result.

### 2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this part of ISO 10362. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 10362 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 4387:1991<sup>2)</sup>, *Cigarettes — Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine.*

2) To be published.

ISO 8243:1991<sup>2)</sup>, *Cigarettes — Sampling.*

### 3 Principle

Dissolution of the smoke condensate from the mainstream smoke in a solvent. Determination of the water content of an aliquot of the solution by gas chromatography, and calculation of the water content of the whole of the smoke condensate.

### 4 Reagents

Use only reagents of recognized analytical reagent grade.

#### 4.1 Carrier gas.

Helium or nitrogen (see 6.2, note 6).

#### 4.2 Propan-2-ol.

Maximum water content: 1,0 mg per cm<sup>3</sup>.

#### 4.3 Internal standard.

Ethanol, or methanol (of purity at least 99 %).

#### 4.4 Extraction solvent.

Propan-2-ol (4.2) containing 5 cm<sup>3</sup> per litre of internal standard (4.3).

#### 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 per cm<sup>3</sup>) 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 water concentration in the solvent. The calibration solutions shall be made up using an extraction solvent from the same batch used in 6.1.

NOTE 2 It is recommended that the calibration solutions be made up weekly.

### 5 Apparatus

Normal laboratory apparatus and in particular the following items:

**5.1 Gas-chromatograph**, equipped with a thermal conductivity detector and recorder or integrator.

**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<sup>3)</sup>, 80 mesh to 100 mesh.

**5.3 Automated dispensing system**, capable of delivering the required volume of solvent.

#### NOTES

3 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<sup>4)</sup> or Chromosorb 102<sup>4)</sup> may be used.

4 Glassware and septa for vials should be stored under desiccation before use.

5 The dispensing system should be flushed prior to use by dispensing a volume of solvent of at least 50 cm<sup>3</sup> 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 20 cm<sup>3</sup> for 44 mm disc or 50 cm<sup>3</sup> for 92 mm disc of the solvent (4.4) to obtain the concentration of water adjusted to the calibration graph (6.3). For standard smoking, see ISO 4387.

#### 6.2 Setting up the apparatus

Set up the apparatus and operate the gas chromatograph, recorder or integrator and autosampler (if one is used) in accordance with the manufacturer's instructions. Ensure that the peaks for water, 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

Column temperature: 170 °C (isothermal)

Injection temperature: 250 °C

Detector temperature: 250 °C

Carrier gas: Helium at a flow rate of about 30 cm<sup>3</sup> per min

Injection volume: 2 µl

NOTE 6.991 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. 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 (concentration of added water in accordance with the area ratios) from these data. Use the slope of 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 3 % from the original value, repeat the full calibration procedure.

3) Trade name. Porapak Q is an example of a suitable product 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 this product.

4) Trade names. Porapak QS and Chromosorb 102 are 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.

NOTE 7 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/cm<sup>3</sup> this batch should be rejected.

#### 6.4 Blank test

Due to the 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 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 (6.1) and blank traps (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.

### 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, in milligrams per cigarette, is given by

$$\frac{C_S - C_B}{N} \times V$$

where

$C_S$  is the concentration of water in the sample smoke trap, in milligrams per cubic centimetre;

$C_B$  is the concentration of water in the blank smoke trap, in milligrams per cubic centimetre;

$N$  is the number of cigarettes smoked through each smoke trap;

$V$  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 were smoked in accordance with ISO 4387 and the resulting smoke solutions were analysed by this method, the following values for repeatability ( $r$ ) and reproducibility ( $R$ ) were 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 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

Mean yield of water mg	Repeatability conditions $r$	Reproducibility conditions $R$
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 from each cigarette smoked and the method used, and shall include all conditions which may affect the result (e.g. atmospheric pressure 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 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 done by

- a) addition of an appropriate quantity of the internal standard prescribed for the nicotine determination in the solvent described in 4.4;
- b) injection of an aliquot of the smoke condensate solution on to a column for nicotine analysis,

which is connected to a flame ionization detector, as well as on to 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.

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