INTERNATIONAL STANDARD

**ISO 10362-1**

Third edition
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# Cigarettes — Determination of water in total particulate matter from the mainstream smoke —

## Part 1: Gas-chromatographic method

*Cigarettes — Dosage de l'eau dans la matière particulaire totale du courant principal de fumée —*

*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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products.*

This third edition cancels and replaces the second edition (ISO 10362-1:1999), which has been technically revised. It also incorporates the Amendment ISO 10362-1:1999/AMD 1:2011. The main changes compared to the previous edition are as follows:

— capillary column procedure as an alternative gas chromatographic procedure has been added;

— the estimates for the repeatability limits ($r$) and the reproducibility limits ($R$) has been updated.

A list of all parts in the ISO 10362 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.



# Introduction

This document may be considered as part of a set of standards which describes the determination of total and nicotine-free dry particulate matter (NFDPM) in total particulate matter from the mainstream smoke. The set comprises ISO 3308, ISO 3402, ISO 4387, ISO 8243, ISO 10315 and this document.

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.

No machine smoking regime can represent all human smoking behaviour:

— it is recommended that cigarettes also be tested under conditions of a different intensity of machine smoking than those specified in this document;

— machine smoking testing is useful to characterize cigarette emissions for design and regulatory purposes, but communication of machine measurements to smokers can result in misunderstandings about differences in exposure and risk across brands;

— smoke emission data from machine measurements may be used as inputs for product hazard assessment, but they are not intended to be nor are they valid as measures of human exposure or risks. Communicating differences between products in machine measurements as differences in exposure or risk is a misuse of testing using ISO standards.

# Cigarettes — Determination of water in total particulate matter from the mainstream smoke —

# Part 1:
# Gas-chromatographic method

## 1 Scope

This document specifies a method for the gas-chromatographic determination of water in total particulate matter from the mainstream smoke. The smoking of cigarettes and collection of mainstream smoke are normally carried out in accordance with ISO 4387. However, the method of this document is also applicable to the determination of water in total particulate matter from the mainstream smoke obtained by non-standard smoking.

NOTE In countries not in a position to use the gas-chromatographic method, the determination of water in total particulate matter from the mainstream smoke is performed using the Karl Fischer method (see ISO 10362-2). In such cases, values obtained for water in total particulate matter from the mainstream smoke are used with the addition of a note made in the expression of the results.

## 2 Normative reference

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 4387, *Cigarettes — Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine*

## 3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

## 4 Principle

The total particulate matter 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 total particulate matter from the mainstream smoke is calculated.

## 5 Reagents

Use only reagents of recognized analytical reagent grade.

**5.1 Carrier gas,** helium or nitrogen (see note in 7.2) or hydrogen (see note in 8.4).



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

**5.3 Internal standard,** ethanol or methanol (of purity at least 99 %).

**5.4 Extraction solution,** propan-2-ol (5.2) containing an appropriate concentration of internal standard (5.3), normally in the range of 1 ml/l to 5 ml/l.

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

To prevent water being absorbed, the bulk extraction solution container shall be fitted with a water trap and shall be kept sealed. The extraction solution shall be stirred continuously to ensure the homogeneity of the water concentration.

**5.5 Reference substance,** complying with grade 2 of ISO 3696:1987, or better.

**5.6 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 (5.5) to the extraction solution (5.4). One of these calibration solutions shall be the extraction solution with no added water (extraction solution blank).

To prevent water being absorbed, all solutions shall be kept sealed. The calibration solutions shall be made up using an extraction solution from the same batch used in 7.1.

Calibration solutions are known to be stable for at least one week when properly stored, but the laboratory should verify calibration solution stability.

## 6 Apparatus

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

**6.1 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.

**6.2 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 may be used (see Clause 8).

**6.3 Dispensing system,** preferably automated, capable of delivering the required volume of extraction solution (5.4).

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

1) Porapak Q is a trade names of an example of a suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

# 7 Procedure

## 7.1 Test portion

Prepare the test portion by dissolving the total particulate matter obtained by the machine smoking of a known number of cigarettes in a fixed volume of the extraction solution (5.4) 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 7.3) provided that there is an adequate volume for effective extraction of the total particulate matter. Analysis should be performed as soon as possible, but if storage is inevitable then store the sample at between 0 °C and 4 °C. The laboratory should verify test portion stability. For standard smoking, see ISO 4387.

## 7.2 Setting up the apparatus

Set up the apparatus and operate the gas chromatograph (6.1) 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 solution 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 is also used as an alternative carrier gas if the detector sensitivity is sufficiently high.

## 7.3 Calibration of the gas chromatograph

Inject an aliquot (2 µl) of each of the calibration solutions (5.6) into the gas chromatograph. Record the peak areas of the water and internal standard (5.3). Carry out the determination at least twice.

Calculate the ratio of the water peak to the internal standard peak from the peak area data for each of the calibration solutions including the extraction solution blank. Plot the graph of the water concentrations in accordance with the area ratios, and calculate a linear regression equation of the peak area ratios as a function of the concentration of water from these data.

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.

## 7.4 Blank test

Due to the absorption of water by smoke traps and extraction solution, it is necessary to determine a value for the sample blank. Prepare sample blanks by treating additional smoke traps including filters (at least 2 smoke traps including filters per 100 cigarettes smoked) in the same manner as that used for smoke collection. Place smoke traps, including filters, near the smoking machine during smoking. Extract and analyse the blank test portions together with the test portions.

### 7.5 Determination

Inject aliquots (2 µl) of the test portion (see 7.1) and blank test portions (see 7.4) into the gas chromatograph. Calculate the ratio of the water peak/internal standard peak from the peak area data.

Carry out two determinations on the same test portion (see 7.1).

Calculate the mean value of the ratio from the two determinations.

Where an autosampler is used, a single aliquot portion from each smoke trap is considered adequate.

## 8 Alternative gas chromatographic procedures

### 8.1 General

Alternative gas-chromatographic columns, both packed and capillary, have been found suitable for the determination of water in total particulate matter from the mainstream smoke. If these are used, it is necessary to ensure that the peaks due to water and the internal standard are well resolved from peaks due to other smoke components and the solvent.

### 8.2 Packed columns

The following may be used as alternative stationary phases in the column described in 6.2:

— Porapak QS[2)], or

— Chromosorb 102[2)].

### 8.3 Capillary columns

PoraPLOT Q[2)], length between 15 m and 30 m, internal diameter between 320 µm and 530 µm, film thickness between 20 µm and 40 µm.

### 8.4 Gas chromatographic conditions

The alternative column described in 8.3 requires the use of purpose-made injection systems. Suitable operating conditions may vary depending on the type of column used and they may need to be optimized following the manufacturer's instructions. Isothermal oven temperature or oven temperature programming, hold times, carrier gas and linear velocity, split ratio and injection volume shall be set for the type of capillary column used. For example, for a 30 m, 0,53 mm internal diameter, 40 µm film thickness capillary column, typical conditions might be as described in 7.2 with the following changes:

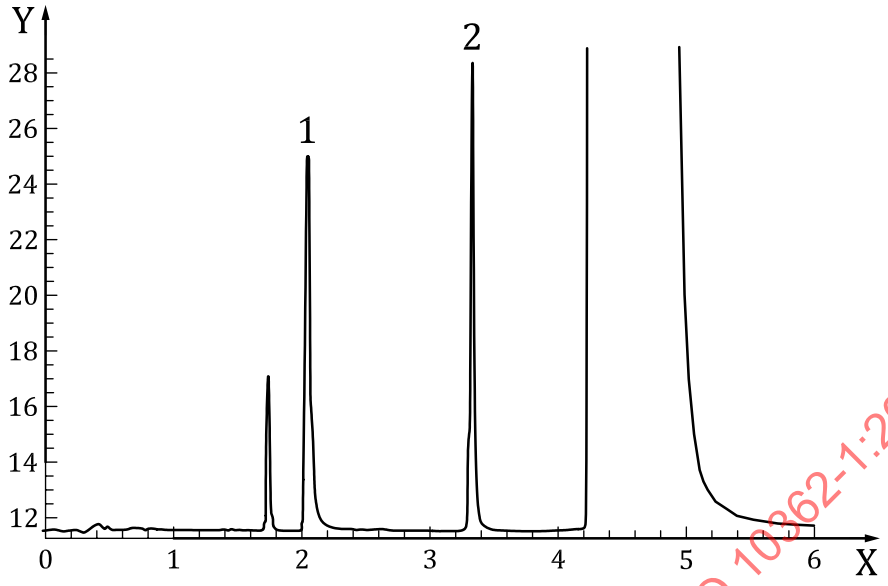
— carrier gas helium at a linear flow rate of about 3 ml/min;

— split ratio 5:1.

Using the above conditions, the analysis time is approximately 6 min (see Figure 1).



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




**Key**

**Peak identification**

1 water

2 internal standard (ethanol)

X time in minutes

Y response

**Figure 1 — Example of a chromatogram of a test portion using capillary column**

NOTE Hydrogen is also used as an alternative carrier gas when using a capillary column.

## 9 Expression of results

Calculate the concentration of water in the test portion and blank test portion using the graph and linear regression equation prepared in 7.3.

From the concentration of water in the test portion subtract the concentration of water in the blank test portions, calculate the amount of water in the total particulate matter by multiplying by the volume of extraction solution. Deduce the amount in the cigarettes smoked. Express the test results in milligrams per cigarette, $m_W$, for each channel to the nearest 0,01 mg and the average per cigarette to the nearest 0,1 mg.

The test result for water content in smoke, $m_W$, in milligrams per cigarette, is given by Formula (1):

$$m_W = \frac{\rho_{WS} - \rho_{WB}}{q} \cdot V_{ES} \quad (1)$$

where

$\rho_{WS}$ is the concentration of water in the test portion, in milligrams per millilitre;

$\rho_{WB}$ is the average concentration of water in the blank test portions, in milligrams per millilitre;

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

$V_{ES}$ is the volume of extraction solution in which the contents of the smoke trap were dissolved, in millilitres.

## 10 Repeatability and reproducibility

A major interlaboratory study involving 64 laboratories and 5 samples, conducted in 2016, 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 2 and Table 3.

**Table 1 — Estimates given by data analysis (all data)**

| Mean value<br>$m_W$<br>mg per cigarette | Repeatability limit<br>$r$<br>mg per cigarette | Reproducibility limit<br>$R$<br>mg per cigarette |
|---|---|---|
| 0,10 | 0,13 | 0,18 |
| 0,29 | 0,23 | 0,31 |
| 0,77 | 0,27 | 0,53 |
| 1,53 | 0,37 | 0,82 |
| 1,82 | 0,42 | 1,00 |

**Table 2 — Estimates given by data analysis (data by packed column)**

| Mean value<br>$m_W$<br>mg per cigarette | Repeatability limit<br>$r$<br>mg per cigarette | Reproducibility limit<br>$R$<br>mg per cigarette |
|---|---|---|
| 0,10 | 0,13 | 0,19 |
| 0,29 | 0,24 | 0,30 |
| 0,75 | 0,25 | 0,57 |
| 1,53 | 0,38 | 0,88 |
| 1,79 | 0,41 | 1,12 |

**Table 3 — Estimates given by data analysis (data by capillary column)**

| Mean value<br>$m_W$<br>mg per cigarette | Repeatability limit<br>$r$<br>mg per cigarette | Reproducibility limit<br>$R$<br>mg per cigarette |
|---|---|---|
| 0,10 | 0,13 | 0,17 |
| 0,28 | 0,21 | 0,31 |
| 0,79 | 0,28 | 0,51 |
| 1,52 | 0,35 | 0,77 |
| 1,84 | 0,43 | 0,90 |

NOTE No statistically relevant differences were observed between the results obtained with capillary or packed columns.