INTERNATIONAL STANDARD

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Cigarettes — Determination of water in smoke condensates —

Part 2:
Karl Fischer method

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

Partie 2: Méthode de Karl Fischer

Reference number
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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

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The committee responsible for this document is ISO/TC 126, *Tobacco and tobacco products.*

This second edition cancels and replaces the first edition (ISO 10362-2:1994), which has been technically 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*

Introduction

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 International Standard;
- 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 smoke condensates —

Part 2:
Karl Fischer method

1 Scope

This part of ISO 10362 specifies the use of the Karl Fischer method for the 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 also applicable to the determination of water in smoke condensates obtained by non-standard smoking.

NOTE ISO 4387 specifies the use of gas chromatography for the determination of water in smoke condensate solutions (see also ISO 10362-1). In countries not in a position to use the gas-chromatographic method, the determination of water in smoke condensate can be performed by the method described in this part of ISO 10362 and an appropriate note made in the expression of the results.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3308, *Routine analytical cigarette-smoking machine — Definitions and standard conditions*

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

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

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 titration with standardized Karl Fischer reagent.

4 Reagents

Use only reagents of recognized analytical reagent grade and distilled water complying with grade 2 of ISO 3696 or better.

4.1 **Propan-2-ol**, $(CH_3)_2CHOH$, extraction solvent.

NOTE Propan-2-ol normally contains a small amount of water which is determined as a blank as outlined in the procedure. It is not recommended that specially dried solvent (e.g. using a molecular sieve) should be used since in this state it is extremely hygroscopic and further handling problems are introduced.

4.2 **Karl Fischer reagent** (KFR), which can be obtained commercially in two forms:

a) as a single reagent with an initial water equivalent of approximately 5 mg/ml; this value decreases on storage;

b) as two separate reagents (Karl Fischer solution A: methanol, pyridine, sulfur dioxide; Karl Fischer solution B: methanol, iodine) which, when mixed in equal volumes, give a reagent with a water equivalent of approximately 3 mg/ml.

Reagents which do not contain pyridine are preferred if these are available. If these are not available, take special care during handling. See also CAUTION concerning waste disposal in 6.4.1.

4.3 Methanol, CH_3OH, with a water content of less than 0,05 g per 100 g.

4.4 Silica gel, freshly activated.

5 Apparatus

Normal laboratory apparatus and in particular the following items:

5.1 Karl Fischer apparatus, preferably of the automatic or semi-automatic type set up according to the manufacturer's instructions for direct titration.

5.2 Solvent dispenser. A suitable container type is a 5 l aspirator bottle with a silica gel moisture trap in the neck and the bottom outlet connected to a 25 ml double-action automatic pipette or an electronically operated pipette. The silica gel has a drying effect on the surface of the solvent causing the water content to vary during use. To avoid this, stir the solvent continuously during use by mounting the dispenser on an automatic stirrer.

5.3 Syringe, of capacity 20 µl.

5.4 Syringe, of capacity 10 ml, fitted with a wide bore needle, or pipette of capacity 10 ml.

5.5 Hot-air oven, capable of maintaining a temperature of (105 ± 5) °C.

5.6 Flask shaker, horizontal-action type.

6 Procedure

6.1 General

Care shall be taken during all operations to avoid contamination from atmospheric moisture.

All glassware used in the preparation of the test portion and in the water determination shall be heated at (105 ± 5) °C for at least 1 h after visible water has evaporated, and cooled and stored in a desiccator over silica gel until used.

6.2 Test portion

The following procedure shall be used when cigarettes are smoked in accordance with ISO 4387. When non-standard smoking or collection of smoke condensate has been used, the procedure described below shall be modified accordingly to give a suitable smoke condensate solution. In this event, a note of the alternative procedure shall be included with the test results.

Collect the cigarette smoke condensate in accordance with ISO 4387 on a routine analytical cigarette smoking machine complying with the requirements of ISO 3308.

Wearing gloves, remove the sealing devices from the smoke trap, open it and remove the filter disc with forceps. Place the folded disc in a dry conical 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.

Run an appropriate quantity of extraction solvent (4.1) into the flask. The folded disc shall be covered with solvent. In the case of a 44 mm glass fibre filter trap, 25 ml is necessary. In the case of a 92 mm glass fibre filter trap, 50 ml is necessary.

Stopper the flask immediately and shake gently on an electric flask shaker (5.6) for at least 20 min, ensuring that the disc does not disintegrate. This provides the smoke condensate solution.

6.3 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.

6.4 Standardization of Karl Fischer reagent

6.4.1 Standardization procedure

Add sufficient methanol (4.3) to the Karl Fischer titration vessel (5.1) to immerse the tips of the electrodes. Titrate any residual solution (see note) in the titration vessel to its end-point by addition of Karl Fischer reagent (KFR).

Using the 20 µl syringe (5.3), add 20 µl (V_W) of water to the titration vessel. To ensure that the syringe does not contain air bubbles, fill it to above the 20 µl mark, invert it and tap the air bubbles to the top. Then depress the plunger to the 20 µl mark and remove excess water quickly from the needle tip with a tissue.

As an alternative, fill the syringe with 20 µl of water and weigh the syringe. After dosage, weigh the syringe again and note the exact mass of water.

Transfer the volume (V_W) of water to the titration vessel taking care to inject the water directly into the solution, not allowing any to fall on to the neck or walls of the vessel. Where the vessel can be fitted with a rubber membrane cap, this shall be used and the needle inserted through the cap. If a water droplet remains on the needle tip, remove it by touching the surface of the solution in the vessel.

Titrate with KFR (4.2) and record the titration value.

Repeat the process and again record the titration value. Repeat a third time. Calculate the mean titration volume (V_t)·

Standardize the KFR every working day.

For best results with the Karl Fischer technique, it is important to ensure that all stages of the analysis are carried out in a uniform manner, extract-to-extract, sample-to-sample, day-to-day.

NOTE It is common practice with the direct-titration technique to carry out a titration “on top of” residual solution in the titration vessel, i.e. without removing the residual solution. When the volume in the vessel reaches a certain level, the liquid is run to waste, retaining just sufficient for the electrode tips to be immersed. In time, however, the methanol concentration in the vessel falls to a level such that the reaction cannot proceed satisfactorily. A precipitate may form and false titration values be obtained. Only experience will tell when titrating “on top of” residual solution has reached this point and then the titration vessel should be completely emptied to waste, rinsed, and recharged with methanol.

CAUTION — Waste from the titration vessel should be run into a container which is kept stoppered until such time as safe disposal can be arranged, preferably through a waste-disposal agency, or in compliance with other national regulations.

6.4.2 Calculation of water equivalent

The water equivalent, E, of the Karl Fischer reagent, expressed in milligrams of water per millilitre, is given by the equation

$$E = \frac{m_W}{V_t} \tag{1}$$

where

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

V_t is the mean volume, in millilitres, of the Karl Fischer reagent used for the titration of the water.

6.5 Determination

Add to the titration vessel 10 ml of the propan-2-ol extract from the blank, preferably using a syringe with a wide-bore needle (5.4) to enable rapid transfer. Titrate with KFR and record the titration volume.

Repeat and again record the titration volume. Determine the mean titration volume for the blank, V_B. Repeat the duplicate determination for all sample blanks.

Add 10 ml of the smoke condensate solution to the titration vessel. Use a minimum volume of solution to wash the syringe or pipette since the residue of the extract may be required for the determination of nicotine. Titrate with KFR and record the titration volume of the sample, V_S.

Dependent on the use to be made of the remaining condensate solution, it may be possible to repeat this determination. However, when this method is used to determine the water content, the remaining solution is frequently used to determine nicotine by steam distillation. This does not allow sufficient solution to duplicate the water determination.

7 Expression of results

The water content, W, of the smoke condensate for each trap, expressed in milligrams per cigarette, is given by the equation

$$W = \frac{(V_S - V_B) \times E \times V_k}{q \times V_a} \tag{2}$$

where

V_S is the volume, in millilitres, of the Karl Fischer reagent used for the titration of the smoke condensate solution;

V_B is the mean volume, in millilitres, of the Karl Fischer reagent used for the blank titration;

E is the water equivalent, in milligrams of water per millilitre, of the Karl Fischer reagent;

V_k is the volume, in millilitres, of solvent used for dissolving the smoke condensate;

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

V_a is the volume, in millilitres, of the smoke condensate solution for the titration.

Express the test results as follows:

a) water content, expressed in milligrams per cigarette smoked, to the nearest 0,01 mg for each individual smoking run;

b) mean water content, expressed in milligrams per cigarette smoked, to the nearest 0,1 mg for the whole test sample.

8 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. 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 (e.g. smoking regime used), or regarded as optional, as well as any circumstances that may have influenced the results (e.g. atmospheric test conditions during smoking). 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

The following particulars shall be included:

- 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

The following particulars shall be included:

- 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 (total particulate matter) 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 (dry particulate matter) 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 (nicotine-free dry particulate matter) content (in milligrams per cigarette) for each channel to the nearest 0,1 mg, and the average per cigarette to the nearest 1 mg.

Bibliography

[1] ISO 10362-1, *Cigarettes — Determination of water in smoke condensates — Part 1: Gas-chromatographic method*

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