SLOVENSKI STANDARD
SIST ISO 10362-2:1995

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Cigarete - Določanje vode v dimnem kondenzatu - 2. del: Karl Fischerjeva metoda

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

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INTERNATIONAL STANDARD

ISO 10362-2

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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
ISO 10362-2:1994(E)

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 10362-2 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

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*

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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 1 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 should 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 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 3308:1991, *Routine analytical cigarette-smoking machine — Definitions and standard conditions.*

ISO 4387:1991, *Cigarettes — Determination of total and nicotine-free dry particulate matter using a routine analytical smoking machine.*

ISO 10362-1:1991, *Cigarettes — Determination of water in smoke condensates — Part 1: Gas-chromatographic method.*

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 or water of at least equivalent purity.

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

NOTE 2 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)

Karl Fischer reagent 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.

The latter reagent is strongly recommended since in the unmixed form it is quite stable, especially if stored in a refrigerator. Its water equivalent is also more appropriate to this analysis. There is a limit, however, to the time the mixed reagent can be stored, even in a refrigerator, and regular replacements are necessary.

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 6.3.1.

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 litre 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

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.

6.1 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.

6.2 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.

6.3 Standardization of Karl Fischer reagent

6.3.1 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 3) 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 3 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 must 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.

6.3.2 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

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

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.

6.4 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

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

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;

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

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

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.