SLOVENSKI STANDARD
SIST ISO 6488:2021

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Nadomešča:
SIST ISO 6488:2004

Tobak in tobačni proizvodi - Določevanje vode – Metoda po Karl Fischerju

Tobacco and tobacco products - Determination of water content - Karl Fischer method

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Foreword

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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).

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

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This third edition cancels and replaces the second edition (ISO 6488:2004), which has been technically revised. It also incorporates the Technical Corrigendum ISO 6488:2004/Cor 1:2008.

The main changes compared to the previous edition are as follows:

— the term high-moisture tobacco has been deleted;

— further information on interlaboratory testing including additional sample types and corresponding statistical data have been added.

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.

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Introduction

This document specifies a Karl Fischer method for the determination of the water content of tobacco and tobacco products. Independent collaborative studies were conducted in 2002, 2009, and 2018. This method is applicable to ground tobacco, a range of smokeless tobacco products, cigarette filler and ground cigars.

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Tobacco and tobacco products — Determination of water content — Karl Fischer method

1 Scope

This document specifies a method for the determination of water content by the Karl Fischer method. It is applicable to raw tobacco as well as tobacco taken from finished products. The method is suitable for water contents ranging from a mass fraction of at least 2 % to 55 %.

2 Normative references

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 648, *Laboratory glassware — Single-volume pipettes*

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

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3 Terms and definitions

No terms and definitions are listed in this document.

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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 https://www.electropedia.org/

4 Principle

The water content of a sample of tobacco or a tobacco product is determined, using the volumetric procedure, by extraction of water by shaking the sample with dry methanol, followed by injection of an aliquot portion into the titration vessel, titration with pyridine-free Karl Fischer reagent and calculation of the water content. The results are reported as mass percent (%).

If a size reduction (grinding or cutting) is applied, it can create a decrease in the original water content. Cryogenic techniques may be used to prevent such moisture losses.

5 Reagents

Use only reagents of recognized analytical grade.

5.1 Karl Fischer reagent, free from pyridine, having a water equivalent per millilitre of reagent of approximately 2 mg to 5 mg.

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

Methanol is hygroscopic so it is recommended to cap the bottle with an automatic delivery pipette equipped with drying tube.

5.3 Desiccant, silica gel, freshly activated.

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5.4 Water, in accordance with grade 2 of ISO 3696, or better.

6 Apparatus

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

All glassware used in the preparation and in the water determination shall be heated at (105 ± 5) °C for at least 1 h after visible water has evaporated. The glassware shall then be cooled and stored in a desiccator over desiccant (5.3) until used.

6.1 Karl Fischer apparatus for automatic titration, consisting of items 6.1.1 to 6.1.5.

6.1.1 Apparatus for the detection of the titration end point, according to the biamperiometrical method.

6.1.2 Double electrode, made of platinum.

6.1.3 Magnetic stirrer.

6.1.4 Titration vessel.

6.1.5 Automatic burettes, for methanol and Karl Fischer reagent.

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6.2 Mechanical shaker, rotary platform, adjustable to a shaking frequency suitable for vigorous mixing.

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6.3 Microsyringe, for the determination of the water equivalent, of capacity 50 µl.

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6.4 Glass volumetric pipettes, class A, of capacities 10 ml and 20 ml, in accordance with ISO 648.

6.5 Conical flasks, of capacities 250 ml and 500 ml, with a conical ground glass joint.

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

7 Standardization of the Karl Fischer reagent

Add sufficient methanol (5.2) to the titration vessel (6.1.4) 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 (5.1).

Add 50 µl of water to the titration vessel using the microsyringe (6.3). To ensure that the syringe does not contain air bubbles, fill it to above the 50 µl mark, invert it and tap the air bubbles to the top. Then depress the plunger to the 50 µl mark and remove excess water quickly from the needle tip with a tissue. As an alternative, fill the syringe with 50 µl of water and weigh the syringe. After dosage, weigh the syringe again and note the exact mass of water.

Transfer the 50 µl of water to the titration vessel, taking care to inject the water directly into the solution, not allowing any to fall onto 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 Karl Fischer reagent (5.1) and record the titration value. Repeat the process. If the difference between the values of two determinations is not greater than 0,05 ml, take the mean of two determinations. Otherwise, repeat the whole determination.

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