



SLOVENSKI STANDARD SIST ISO 6488:2004

01-oktober-2004

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

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

Tabac et produits du tabac -- Détermination de la teneur en eau -- Méthode de Karl Fischer

ITEH STANDARD PREVIEW
(standards.iteh.ai)

Ta slovenski standard je istoveten z: **ISO 6488:2004**

<https://standards.iteh.ai/catalog/standards/sist/3d3300d9-f68f-4b89-8101-c1d73f6f4954/sist-iso-6488-2004>

ICS:

65.160

V[àæ Ë[àæ } àæ à^ \ àæ
[] !^ { æ

Tobacco, tobacco products
and related equipment

SIST ISO 6488:2004

en

iTeh STANDARD PREVIEW
(standards.iteh.ai)

SIST ISO 6488:2004

<https://standards.iteh.ai/catalog/standards/sist/3d3300d9-f68f-4b89-8101-c1d73f6f4954/sist-iso-6488-2004>

INTERNATIONAL STANDARD

**ISO
6488**

Second edition
2004-02-15

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

*Tabac et produits du tabac — Détermination de la teneur en eau —
Méthode de Karl Fischer*

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[SIST ISO 6488:2004](https://standards.iteh.ai/catalog/standards/sist/3d3300d9-f68f-4b89-8101-c1d73f6f4954/sist-iso-6488-2004)

[https://standards.iteh.ai/catalog/standards/sist/3d3300d9-f68f-4b89-8101-
c1d73f6f4954/sist-iso-6488-2004](https://standards.iteh.ai/catalog/standards/sist/3d3300d9-f68f-4b89-8101-c1d73f6f4954/sist-iso-6488-2004)



Reference number
ISO 6488:2004(E)

© ISO 2004

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

iTeh STANDARD PREVIEW (standards.iteh.ai)

SIST ISO 6488:2004

<https://standards.iteh.ai/catalog/standards/sist/3d3300d9-f68f-4b89-8101-c1d73f6f4954/sist-iso-6488-2004>

© ISO 2004

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

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

Published in Switzerland

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

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.

ISO 6488 was prepared by Technical Committee ISO/TC 126, *Tobacco and tobacco products*.

This second edition of ISO 6488 cancels and replaces ISO 6488-1:1997, which has been technically revised.

ITC STANDARD PREVIEW
(standards.iteh.ai)
SIST ISO 6488:2004
<https://standards.iteh.ai/catalog/standards/sist/3d3300d9-f68f-4b89-8101-c1d73f6f4954/sist-iso-6488-2004>

Introduction

During the development of International Standards, inter-laboratory tests were carried out using two different principles for the determination of water content of raw tobacco and tobacco taken from finished products. These were

- this Karl Fischer procedure, and
- the gas chromatographic procedure.

These studies show that no differences occur between the results obtained by the two different methods. The gas chromatographic method is described in ISO 16632.

iTeh STANDARD PREVIEW (standards.iteh.ai)

[SIST ISO 6488:2004](https://standards.iteh.ai/catalog/standards/sist/3d3300d9-f68f-4b89-8101-c1d73f6f4954/sist-iso-6488-2004)

<https://standards.iteh.ai/catalog/standards/sist/3d3300d9-f68f-4b89-8101-c1d73f6f4954/sist-iso-6488-2004>

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

1 Scope

This International Standard 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 referenced documents are indispensable for the application 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 — One-mark pipettes*

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

ISO 4874, *Tobacco — Sampling of batches of raw material — General principles*

ISO 8243, *Cigarettes — Sampling*

ISO 10362-2, *Cigarettes — Determination of water in smoke condensates — Part 2: Karl Fischer method*

ISO 15592-1, *Fine-cut tobacco and smoking articles made from it — Methods of sampling, conditioning and analysis — Part 1: Sampling*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1

high-moisture tobacco

any tobacco sample containing volatile matter over 20 % as determined by drying at between 100 °C and 105 °C

4 Principle

The water content of a sample of tobacco or a tobacco product is determined 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 method is applicable to any type of tobacco sample provided that the particle size reduction is less than 4 mm.

NOTE 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.

ISO 6488:2004(E)

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.

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

WARNING — Methanol is hazardous to human subjects and to the environment. Handle it with care.

5.3 Desiccant, silica gel, freshly activated.

5.4 Water, complying 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 $(105 \pm 5) ^\circ\text{C}$ 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 endpoint, according to the biamperometrical 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.

6.2 Mechanical shaker, rotary platform, adjustable to a shaking frequency of 155 min^{-1} .

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

6.4 One-mark pipettes, of capacities 10 ml and 20 ml, complying with Class A of 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 $(105 \pm 5) ^\circ\text{C}$.

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 endpoint 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.

Standardize the Karl Fischer reagent 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 enough 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 may be obtained. Only experience will tell when titration “on top of” residual solution has reached this point and then the titration vessel has to be completely emptied to waste, rinsed, and recharged with methanol.

Calculate the water equivalent of the Karl Fischer reagent according to the following equation:

$$E = \frac{m_w}{V_w} \quad (1)$$

SIST ISO 6488:2004
<https://standards.iteh.ai/catalog/standards/sist/3d3300d9-f68f-4b89-8101-c1d73f6f4954/sist-iso-6488-2004>

where

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

m_w is the mass, in milligrams, of the water used for the standardization of the Karl Fischer reagent;

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

Repeat the determination of the water equivalent daily and on each new batch of Karl Fischer reagent.

8 Sampling

Sample raw tobacco in accordance with ISO 4874 and cigarettes in accordance with ISO 8243. Sample fine-cut tobacco in accordance with ISO 15592-1.

Each time a sample is collected and stored, it should be placed in an airtight container having a size just sufficient to contain the sample.

If samples are stored at 4 °C, allow the closed container to equilibrate at room temperature before opening.