
**Plastics — Determination of water
content**

Plastiques — Dosage de l'eau

iTeh STANDARD PREVIEW
(standards.iteh.ai)

[ISO 15512:2008](https://standards.iteh.ai/catalog/standards/sist/fff7971d-7875-4d41-870c-b99ef7ba7183/iso-15512-2008)

<https://standards.iteh.ai/catalog/standards/sist/fff7971d-7875-4d41-870c-b99ef7ba7183/iso-15512-2008>



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)

ISO 15512:2008

<https://standards.iteh.ai/catalog/standards/sist/fbf7971d-7875-4d41-870c-b99ef7ba7183/iso-15512-2008>



COPYRIGHT PROTECTED DOCUMENT

© ISO 2008

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

Contents

Page

Foreword.....	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Method A — Extraction with anhydrous methanol	2
3.1 Principle	2
3.2 Reagents	2
3.3 Apparatus	2
3.4 Preparation of test sample	2
3.5 Procedure	3
3.6 Expression of results	3
3.7 Precision	4
4 Method B — Water vaporization	4
4.1 Principle	4
4.2 Reagents	4
4.3 Apparatus	5
4.4 Preparation of sample	6
4.5 Procedure	6
4.6 Expression of results	8
4.7 Precision	8
5 Method C — Manometric method	8
5.1 Principle	8
5.2 Reagent	8
5.3 Apparatus	9
5.4 Preparation of sample	9
5.5 Procedure	11
5.6 Expression of results	13
5.7 Precision	13
6 Test report	13
Annex A (informative) Selection of the optimum heating temperature for the water content determination	14
Bibliography	16

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 15512 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This second edition cancels and replaces the first edition (ISO 15512:1999), which has been technically revised.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 15512:2008

<https://standards.iteh.ai/catalog/standards/sist/fbf7971d-7875-4d41-870c-b99ef7ba7183/iso-15512-2008>

Introduction

The inter-laboratory comparability of water content determinations is often low. Major causes for this are the sample packaging, sample handling and differences between equipment and settings. In order to be able to compare data between two laboratories, special care needs to be taken with sample packaging and sample handling. Samples should e.g. be packed in special glass containers or water barrier sealed bags. Sample handling should preferably be carried out in a dry nitrogen or air environment. To improve the repeatability and reproducibility, the procedure specified in this International Standard should be followed strictly.

The temperature settings for the vaporization method described in this International Standard are not specified in the standard. For the manometric method, a temperature of 200 °C is often used. However, for some condensation materials this might be too high and could e.g. cause generation of water due to a condensation reaction.

The heating temperature should be optimized concerning the material to be tested, the equipment in use and the practical circumstances. If the temperature is too low, the total amount of water in the material to be tested will not be evaporated completely, whereas too high temperatures cause water generation due to effects like degradation and condensation reactions.

In this International Standard, a procedure is included for optimization of the heating temperature in order to choose the correct temperature for the water content determination and to improve the inter-laboratory comparability.

ITEH STANDARD PREVIEW
(standards.iteh.ai)

[ISO 15512:2008](https://standards.iteh.ai/catalog/standards/sist/fbf7971d-7875-4d41-870c-b99ef7ba7183/iso-15512-2008)

<https://standards.iteh.ai/catalog/standards/sist/fbf7971d-7875-4d41-870c-b99ef7ba7183/iso-15512-2008>

iTeh STANDARD PREVIEW
(standards.iteh.ai)

ISO 15512:2008

<https://standards.iteh.ai/catalog/standards/sist/fff7971d-7875-4d41-870c-b99ef7ba7183/iso-15512-2008>

Plastics — Determination of water content

1 Scope

1.1 This International Standard specifies methods for the determination of the water content of plastics in the form of granules and finished articles. These methods do not test for water absorption (kinetics and equilibrium) of plastics as measured by ISO 62. The methods are suitable for the determination of water content as low as the following levels:

- Method A 0,1 % or better;
- Method B 0,01 % or better;
- Method C 0,01 % or better.

Water content is an important parameter for processing materials, and should remain below the level specified in the appropriate material standard.

1.2 Three alternative methods are specified in this International Standard:

- a) **Method A** is an extraction method using anhydrous methanol followed by a Karl Fischer titration of the extracted water. It can be used for all plastics and is applicable to granules having a maximum size of 4 mm × 4 mm × 3 mm.
- b) **Method B** is a vaporization method using heated, dry air or nitrogen gas to evaporate the water, followed by a Karl Fischer titration of the collected water. It can be used for all plastics and is applicable to granules smaller than 4 mm × 4 mm × 3 mm.
- c) **Method C** is a manometric method. The water content is determined from the increase in pressure which results when the water is evaporated under a vacuum. This method is not applicable to plastic samples containing volatile compounds, other than water, in amounts contributing significantly to the vapour pressure at room temperature. Checks for the presence of large amounts of volatile compounds should be carried out periodically, for example by gas chromatography. Such checks are particularly required for new types or grades of material.

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 760, *Determination of water — Karl Fischer method (General method)*

3 Method A — Extraction with anhydrous methanol

3.1 Principle

A test portion is extracted with anhydrous methanol and the extracted water determined by titration using the Karl Fischer method.

3.2 Reagents

During the analysis, use only reagents of recognized analytical grade.

3.2.1 Methanol, anhydrous, having a water content less than 0,1 % by mass.

3.2.2 Karl Fischer reagent, with an equivalence factor of approximately 3 mg/ml to 5 mg/ml of water. If the reagent is prepared, check its equivalence factor as specified in ISO 760.

3.3 Apparatus

Ordinary laboratory apparatus and the following:

3.3.1 Glass flasks, 250 ml capacity, provided with ground-glass or rubber stoppers.

3.3.2 Conical titration flasks, 150 ml capacity, with standard ground necks and provided with ground-glass stoppers.

3.3.3 Reflux condensers, with ground neck capable of being fitted on to the flasks (3.3.2) and on to the tubes (3.3.4).

3.3.4 Water-absorption tubes with ground joints, containing calcium chloride or other drying agent.

3.3.5 Electrical or hot-air heaters, for the flasks (3.3.2).

3.3.6 Pipettes, 50 ml capacity. (Automatic filling pipettes are acceptable.)

3.3.7 Woulfe bottles, with two tubes.

3.3.8 Curved or U-shaped water-absorption tubes, filled with calcium chloride.

3.3.9 Rubber pipette filler.

3.3.10 Pipette, 10 ml capacity.

3.3.11 Desiccator, containing calcium chloride.

3.3.12 Analytical balance, accurate to 0,2 mg.

3.3.13 Karl Fischer apparatus, for determining water content in accordance with ISO 760.

3.4 Preparation of test sample

3.4.1 Granules

Take a representative sample of approximately 100 g. Put the sample into a pre-dried glass flask (3.3.1) and immediately close it with a stopper.

NOTE It is desirable to pre-dry the container in an oven, then cool it over a suitable water absorbent, for instance silica gel.

3.4.2 Finished articles

Cut or saw the sample into pieces of appropriate size, i.e. having a maximum size of 4 mm × 4 mm × 3 mm. Proceed quickly to minimize moisture absorption.

3.5 Procedure

3.5.1 Precautions

Due to the low quantities of water measured, maximum care shall be exercised at all times to avoid contaminating the sample with water from the sample container, the atmosphere or transfer equipment. Hygroscopic resin samples shall be protected from the atmosphere.

3.5.2 Preparation of test portions

Conduct the test on two test portions from the same sample. Use test portions containing 10 mg to 20 mg of water based on the estimated water content of the sample.

3.5.3 Determination

Carefully dry the apparatus.

Weigh each test portion to the nearest 1 mg into a conical titration flask (3.3.2) fitted with a ground-glass stopper. Pipette 50 ml (3.3.6) of anhydrous methanol (3.2.1) into the conical flask containing the test portion. At the same time, add 50 ml of anhydrous methanol to another conical flask for a blank test. Stopper the flasks. Keep the stoppered flasks in the desiccator (3.3.11) pending continuation of the test.

Unstopper the flasks and quickly attach them to reflux condensers (3.3.3) fitted with calcium chloride tubes (3.3.4). Reflux the contents of the conical flasks for 3 h, then leave them for 45 min to cool to room temperature. Separate the flasks from the condensers, quickly stopper them and place them in the desiccator.

Use the Karl Fischer apparatus (3.3.13) to titrate the contents of each flask with Karl Fischer reagent (3.2.2).

3.6 Expression of results

The water content w , expressed as a percentage by mass, for each of the two determinations is given by the following formula:

$$w = \frac{(V_1 - V_2) T}{m} \times 100$$

where

V_1 is the volume, expressed in millilitres, of Karl Fischer reagent used for the determination;

V_2 is the volume, expressed in millilitres, of Karl Fischer reagent used for the blank test;

T is the water equivalent, expressed in grams of water per millilitre of reagent, of Karl Fischer reagent;

m is the mass, in grams, of the test portion.

The two values for the water content shall not differ by more than 10 % relative or 0,02 % absolute, whichever is the greater. If the difference is greater, repeat the measurements until acceptable consecutive values are obtained and discard all unacceptable results.

The result is expressed as the average of these two determinations, rounded to the nearest 0,01 % by mass.

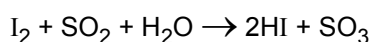
3.7 Precision

The precision of this test method is not known because interlaboratory data are not available. If and when interlaboratory data are obtained, a precision statement will be added at a subsequent revision.

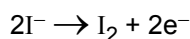
4 Method B — Water vaporization

4.1 Principle

A test portion is weighed then placed in an oven. The water contained in the test portion is vaporized and carried to the titration cell by a dry nitrogen carrier gas. The water is then titrated using the coulometric Karl Fischer method. This method is based on the reduction of iodine by sulfur dioxide in the presence of water to form sulfur trioxide and hydroiodic acid as follows:



Unlike the conventional Karl Fisher reagents that include iodine, the coulometric technique generates the iodine electrolytically from iodide



with 10,71 C of generating current corresponding to 1 mg of water in accordance with Faraday's law.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

4.2 Reagents

During the analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

ISO 15512:2008

<https://standards.iteh.ai/catalog/standards/sist/fbf7971d-7875-4d41-870c-09961799185bc-15512-2008>

4.2.1 Anode solution, containing iodide ions to generate iodine in the reaction mixture, in accordance with the equipment manufacturer's instructions (for use when a titration cell with a diaphragm is being used).

4.2.2 Cathode solution, containing a suitable salt in methanol (or another suitable organic solvent), prepared in accordance with the equipment manufacturer's instructions (for use when a titration cell with a diaphragm is being used).

4.2.3 Universal reagent, containing iodide ions to generate iodine in the reaction mixture, prepared in accordance with the equipment manufacturer's instructions (for use when a titration cell without a diaphragm is being used).

4.2.4 Neutralization solution, consisting of approximately 4 mg/ml of water in propylene carbonate, ethylene glycol monomethyl ether (2-methoxyethanol) or methyl cellosolve.

4.2.5 Silica gel, in the form of granules of approximately 2 mm diameter, for use as a desiccant.

4.2.6 SICAPENT[®] 1) molecular sieve or **phosphorus pentoxide**, for use as a carrier-gas desiccant.

4.2.7 Grease, containing little or no water and having low water absorptivity, for lubricating the ground-glass joints to maintain the airtightness of the system.

4.2.8 Nitrogen gas (N₂), containing less than 5 µg/g of water.

1) SICAPENT[®] is a trade name of MERCK. It is an example of a suitable product available commercially. This information is given for the convenience of users of this International Standard and does not constitute an endorsement by ISO of this product.

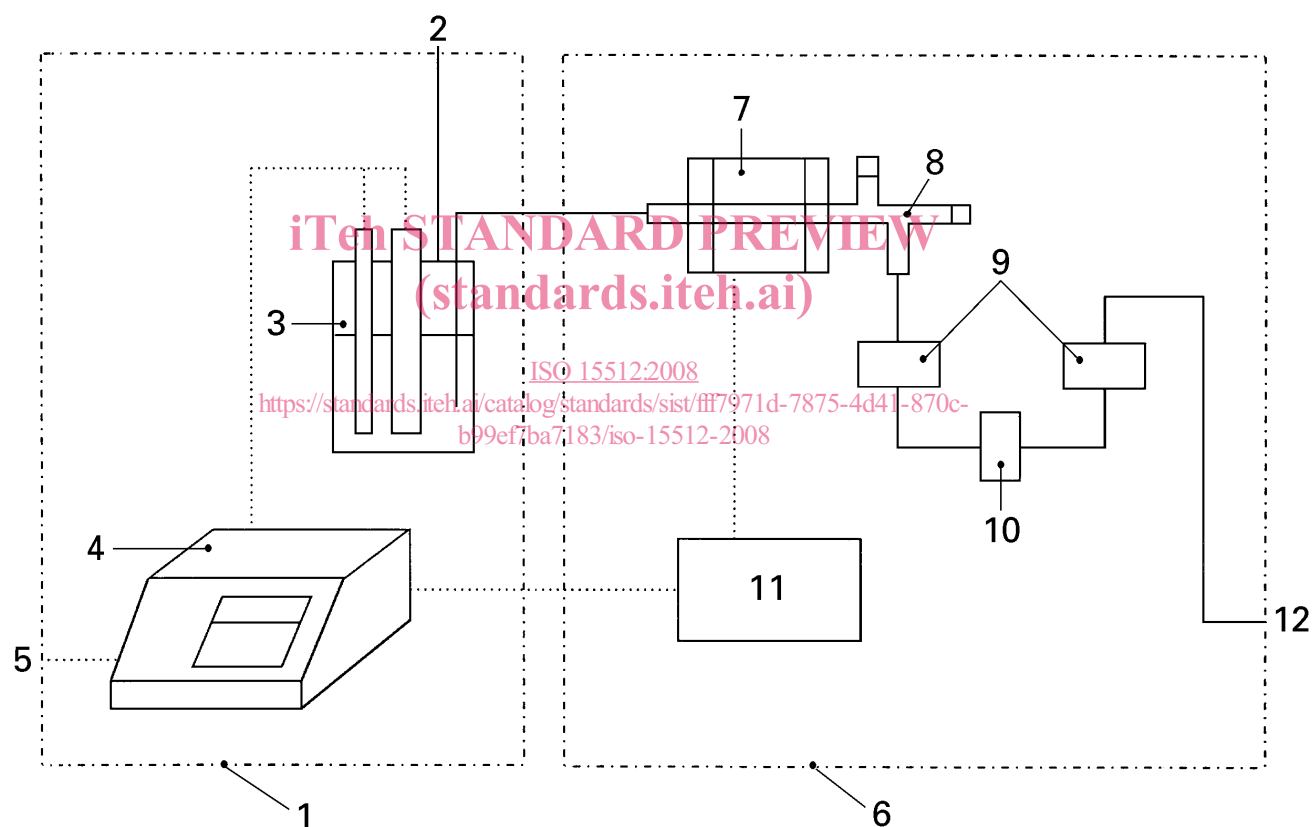
4.3 Apparatus

Ordinary laboratory apparatus and the following:

4.3.1 Coulometric Karl Fischer titrator, consisting of a control unit and a titration-cell assembly equipped with a generator cell with or without a diaphragm, dual platinum sensing electrodes and a magnetic stirrer (see Figure 1). The instrument is designed to coulometrically generate iodine that reacts stoichiometrically with the water present in the cell. The number of coulombs of electricity required to generate the reagent are converted to micrograms of water which is given as a direct digital readout.

A diaphragm-free cell is accurate enough for many applications. However, check with the equipment supplier to verify whether the particular applications for which this method is being used require a diaphragm. A cell with a diaphragm is recommended if the best possible accuracy is required.

4.3.2 Water vaporizer, consisting of an oven capable of heating the test portion to at least 300 °C, a heating tube, a temperature control unit, a carrier-gas flowmeter and carrier-gas drying tubes containing desiccant (see Figures 1 and 2).



Key

- | | | | |
|---|--------------------------------|----|--|
| 1 | coulometric Karl Fischer meter | 7 | oven |
| 2 | exhaust gas | 8 | heating tube |
| 3 | titration cell | 9 | water-absorption tubes (filled with desiccant, for example P ₂ O ₅) |
| 4 | titration controller | 10 | flowmeter |
| 5 | power supply | 11 | temperature controller |
| 6 | water vaporizer | 12 | N ₂ gas |

Figure 1 — Flow chart of system used for the determination of water content using method B