
**Plastics — Determination of water
content**

Plastiques — Dosage de l'eau

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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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

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

This fifth edition cancels and replaces the fourth edition (ISO 15512:2016), which has been technically revised. The main change compared to the previous edition is as follows:

- addition of two alternative methods for water determination (Methods D and E).

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.

Introduction

The interlaboratory comparability of the water content determination of plastics is often low. Major causes for this are the sample packaging, sample handling, and differences between equipment and settings. Samples should, e.g. be packed in special glass containers or water barrier sealed bags. Sample handling is preferably to be carried out in a dry nitrogen or air environment. For improving the repeatability and reproducibility, the procedure prescribed in this document is intended to be followed strictly.

The temperature settings for the vaporization method are not specified in this document. 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 needs to be optimized depending on 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 document, 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 interlaboratory comparability.

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Plastics — Determination of water content

1 Scope

This document specifies methods for the determination of the water content of plastics in the form of powder, granules, and finished articles. These methods do not test for water absorption (kinetics and equilibrium) of plastics as measured by ISO 62.

Method A is suitable for the determination of water content as low as 0,1 % with an accuracy of 0,1 %. Method B and Method C are suitable for the determination of water content as low as 0,01 % with an accuracy of 0,01 %. Method D is suitable for the determination of water content as low as 0,01 % with an accuracy of 0,01 %. Method E is suitable for the determination of water content as low as 0,001 % with an accuracy of 0,001 %. The stated accuracies are detection limits which depend also on the maximal possible sample mass. The water content is expressed as a percentage mass fraction of water.

Method D is suitable for polyamide (PA), polycarbonate (PC), polypropylene (PP), polyethylene (PE), epoxy resin, polyethylene terephthalate (PET), polyester, polytetrafluoroethylene (PTFE), polyvinyl chloride (PVC), polylactide (PLA), polyamidimid (PAI), it is especially not recommended for samples which can release NH_3 . Methods A, B, C and E are generally suitable for all types of plastic and moisture level.

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

Six alternative methods are specified in this document.

- **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 smaller than $4 \text{ mm} \times 4 \text{ mm} \times 3 \text{ mm}$. The method can also be used for, e.g. prepolymer materials in the form of a powder that are insoluble in methanol.
- **Method B1** is a vaporization method using a tube oven. The water contained in the test portion is vaporized and carried to the titration cell by a dry air or nitrogen carrier gas, followed by a Karl Fischer titration or a coulometric determination by means of a moisture sensor of the collected water. It can be used for all plastics and is applicable to granules smaller than $4 \text{ mm} \times 4 \text{ mm} \times 3 \text{ mm}$.
- **Method B2** is a vaporization method using a heated sample vial. The water contained in the test portion is vaporized and carried to the titration cell by a dry air or nitrogen carrier gas, followed by a Karl Fischer titration of the collected water. It can be used for all plastics and is applicable to granules smaller than $4 \text{ mm} \times 4 \text{ mm} \times 3 \text{ mm}$.
- **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 are to be carried out periodically, for example by gas chromatography. Such checks are particularly required for new types or grades of material.
- **Method D** is a thermocoulometric method using a diphosphorus pentoxide (P_2O_5) cell for the detection of the vaporized water. The water contained in the test portion is vaporized and carried to the sensor cell by a dry air or nitrogen carrier gas, followed by a coulometric determination of the collected water. 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. This is specially related to volatile components which can react with the acidic coating of the diphosphorus pentoxide sensor, e.g. ammonia or any kind of amines. Checks for the presence of large amounts of volatile compounds are to be carried out periodically. Such checks are particularly required for new types or grades of material.

- **Method E** is a calcium hydride based method. The water content of a sample evaporates due to a combination of vacuum and heating. The evaporated water reacts with calcium hydride to molecular hydrogen and calcium hydroxide. The hydrogen causes an increase of pressure in the vacuum that is proportional to the evaporated water. Volatile components, that do not react with calcium hydride condensate in a cooling trap and do not affect the measurement.

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

3 Terms and definitions

No terms and definitions are listed in this document.

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 <http://www.electropedia.org/>

4 Method A — Extraction with anhydrous methanol

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4.1 Principle

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

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4.2 Reagents

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

4.2.1 Titration medium, methanol anhydrous, having a water content less than 0,1 % mass fraction. Other solvents can be used if shown to be comparable.

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

4.3 Apparatus

Ordinary laboratory apparatus and the following.

4.3.1 Glass flasks, approximately 250 ml capacity, provided with a suitable cap preventing moisture uptake or release.

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

4.3.3 Reflux condensers, with ground neck capable of being fitted on to the flasks (4.3.2) and on to the tubes (4.3.4).

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

4.3.5 Heaters, suitable for the conical titration flasks ([4.3.2](#)).

4.3.6 Desiccator, containing a suitable desiccant.

4.3.7 Analytical balance, accurate to 0,2 mg.

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

4.4 Preparation of test sample

4.4.1 Granules or powder

Take a representative sample of approximately 100 g. Put the sample into a pre-dried glass flask ([4.3.1](#)) and immediately close it with a stopper.

It is desirable to pre-dry the container in an oven and then cool it over a suitable desiccant.

4.4.2 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.

4.5 Procedure

4.5.1 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.

4.5.2 Preparation of test portions

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

Using modern coulometric Karl Fischer titration instruments, test portions containing less than 10 mg can be applied if the same level of accuracy of results can be achieved.

4.5.3 Determination

Carefully dry the apparatus.

Weigh each test portion to the nearest 1 mg into a conical titration flask ([4.3.2](#)) fitted with a ground-glass stopper. Pipette 50 ml of anhydrous methanol ([4.2.1](#)) into the conical flask containing the test portion. At the same time, pipette 50 ml of anhydrous methanol to another conical flask for a blank test. Stopper the flasks. Keep the stoppered flasks in the desiccator ([4.3.6](#)) pending continuation of the test.

Unstopper the flasks and quickly attach them to reflux condensers ([4.3.3](#)) fitted with calcium chloride tubes ([4.3.4](#)). 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 (4.3.8) to titrate the contents of each flask with Karl Fischer reagent (4.2.2).

NOTE Alternative sample preparation methods and titration methods are given in Annex A.

4.6 Expression of results

The water content, w , expressed as a percentage mass fraction, for each of the two determinations is given by Formula (1):

$$w = \frac{(V_1 - V_2) \cdot T}{m} \cdot 100 \quad (1)$$

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.

NOTE For some equipment, V_2 might not be available separately but only used for internal calculation of $V_1 - V_2$. In this case, the numerator in Formula (1) simplifies to $V \times T$.

4.7 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 to a subsequent revision.

For comparison of data between two laboratories, special care needs to be taken on sample packing and sample handling.

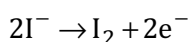
5 Method B1 — Water vaporization using a heating tube oven

5.1 Principle

The sample 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 Fischer 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.

5.2 Reagents

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

5.2.1 Anode solution, containing iodide ions to generate iodine in the reaction mixture, in accordance with the equipment manufacturer's instructions.

5.2.2 Cathode solution, containing a suitable salt in methanol (or another suitable organic solvent), prepared in accordance with the equipment manufacturer's instructions.

5.2.3 Universal single anode reagent, containing iodide ions to generate iodine in the reaction mixture, in accordance with the equipment manufacturer's instructions, for use in a diaphragm-free cell.

NOTE A universal single anode coulometric reagent is applied in diaphragm-free titration cells. Titration cells with diaphragm require the use of a separate anode and cathode solution.

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

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

5.3 Apparatus

Ordinary laboratory apparatus and the following.

5.3.1 Coulometric Karl Fischer titrator, consisting of a control unit and a titration-cell assembly equipped with a generator cell, with or without 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 coulombs of electricity required to generate the reagent are converted to micrograms of water and is given as a direct digital readout.

NOTE A diaphragm-free cell is accurate enough for many applications. However, some applications can require a diaphragm for the best possible accuracy. This can be checked with the equipment supplier.

5.3.2 Water vaporizer, consisting of an oven capable of heating the sample to at least 300 °C, a heating tube (see [Figure 2](#)), a temperature control unit, a carrier-gas flow meter, and carrier-gas drying tubes containing desiccant.

5.3.3 Microsyringe, with capacity of 10 µl, calibrated.

5.3.4 Sample boat.

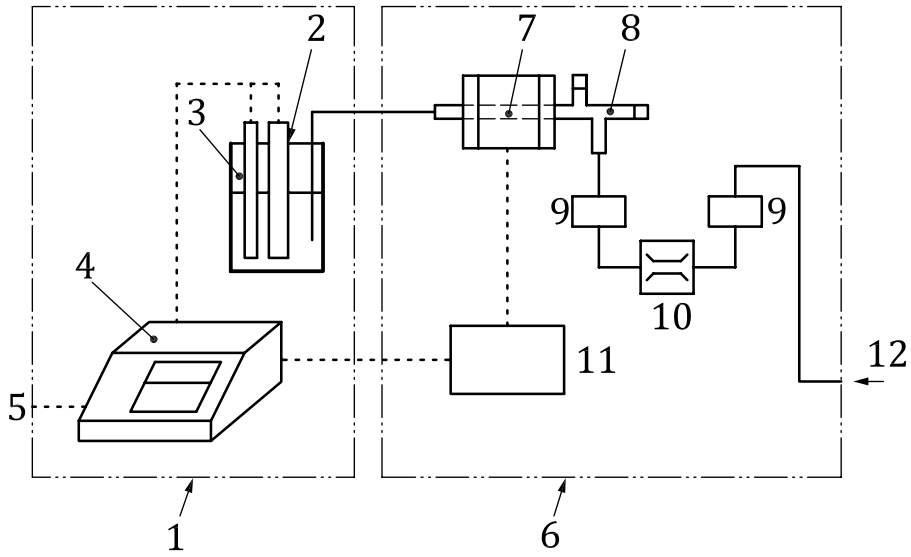
Aluminium foil can be used to hand-fashion a disposable boat large enough to contain the sample and small enough to fit into the heating tube.

5.3.5 Suitable desiccant, for the carrier-gas drying tubes ([5.3.2](#)).

5.3.6 Molecular sieve, pore size 0,3 nm, desiccant for carrier gas.

5.3.7 PTFE sleeves, to be used to maintain the air-tightness of the ground glass joints of the system.

Alternatively, grease, containing little or no water and having low water absorptivity, can be used.



Key

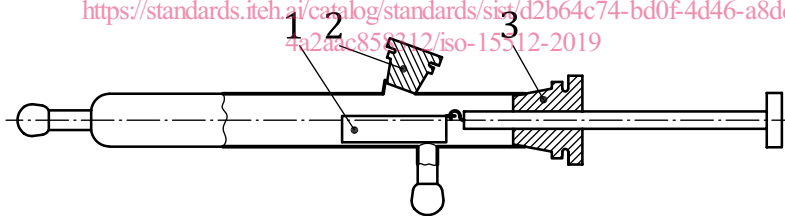
- | | |
|----------------------------------|--|
| 1 coulometric Karl Fischer meter | 7 oven |
| 2 exhaust gas | 8 heating tube |
| 3 titration cell | 9 water-absorption tubes (filled with desiccant, for example P ₂ O ₅) |
| 4 titration controller | 10 flow meter |
| 5 power supply | 11 temperature controller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

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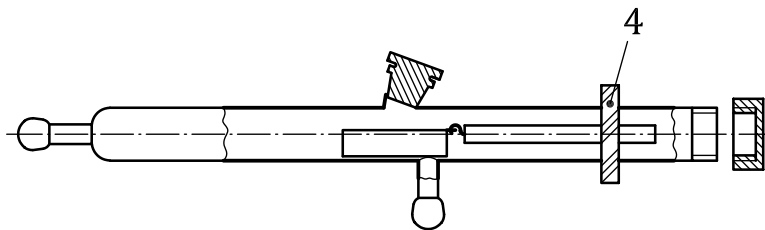
Figure 1 — Flowchart of a system used for the determination of water content using method B1

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a) Push-bar type



b) Magnet type

Key

- | | |
|----------------|---------------------|
| 1 sample boat | 3 sample-boat inlet |
| 2 sample inlet | 4 magnet |

Figure 2 — Heating tubes

5.4 Preparation of test sample

The test material can be in any form, such as granules, moulding powder, fabricated shapes, or moulded items.

Cut fabricated shapes and moulded items to a suitable size. Granules shall be smaller than 4 mm × 4 mm × 3 mm.

Take a representative sample of ≤10 g. Due to the small size of the sample, care shall be taken to ensure that it is in fact representative.

5.5 Procedure

5.5.1 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.

5.5.2 Preparation of apparatus

Refer to [Figure 1](#) for the following procedure.

Assemble the water vaporizer ([5.3.2](#)) and coulometric Karl Fischer titrator ([5.3.1](#)) as directed in the manufacturer's instructions. Fill the absorption tubes with desiccating agent ([5.3.2](#)) as directed in the manufacturer's instructions.

Pour approximately 200 ml (adjust for the container size) of the anode solution ([5.2.1](#)) into the generator cell and approximately 10 ml of the cathode solution ([5.2.2](#)) into the cathode solution cell ([5.3.1](#)) (adjust these amounts for the container size). The surface of the cathode solution shall be below the level of the anode solution to prevent backflow contamination of the cathode solution.

NOTE A universal single anode coulometric reagent is applied in diaphragm-free titration cells. Titration cells with diaphragm require the use of a separate anode and cathode solution.

Start the titration cell. If the cell potential shows a negative value, indicating that the anode solution contains an excess of iodine, add 50 µl to 200 µl (adjust for the container size) of the neutralization solution ([5.2.4](#)).

Disconnect the tube connecting the vaporizer unit to the titration cell. Set the carrier gas flow to 200 ml/min to 300 ml/min and heat the oven to the desired temperature to remove any residual water from the vaporizer ([5.3.2](#)).

Remove any residual water from the walls e.g. by gently swirling the cell. If possible, stir the solution for 1 min in the "TITRATION" mode to dry and stabilize the inner atmosphere.

Reconnect the tube from the vaporizer unit to the titration cell. Keep the carrier-gas flow on during the whole titration. The instrument is then ready for sample analysis.

5.5.3 Equipment checks

To check if the coulometric Karl Fischer titrator is functioning properly, carry out a determination with a known amount of water as follows. With the analyser stabilized and in the "READY" position, start the titration process and carefully inject 5 µl of distilled water into the cell using a 10 µl syringe ([5.3.3](#)). When the titration process is finished, record the reading. This reading should be (5 000 ± 250) µg.

To check if the whole system is functioning properly, carry out a determination with any available certified water standard for Karl Fischer ovens. Examples are:

- sodium tartrate dihydrate with 15,6 % ± 0,5 % water content;