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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

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

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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 normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 62, *Plastics — Determination of water absorption*.

ISO 760:1978, *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

3.5.3.1 Carefully dry the apparatus.

3.5.3.2 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.

3.5.3.3 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.

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

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3.6 Expression of results

3.6.1 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.

3.6.2 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.

3.6.3 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.

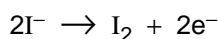
4 Method B — Water vaporization

4.1 Principle

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 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.

4.2 Reagents

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

4.2.1 Anode solution, containing iodide ions (to generate iodine in the reaction mixture), pyridine (or another base such as dipyrldylpropane, alkylaminopyridine, imidazole or diethanolamine), sulfur dioxide and methanol (or another suitable organic solvent), prepared in accordance with the equipment manufacturer's instructions.

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.

NOTE For the anode (4.2.1) and cathode solutions (4.2.2), hydranal Karl Fischer reagents are recommended, as these reagents do not contain pyridine, are non-toxic, are very stable, and have no offensive odour.

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

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

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

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

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

4.3 Apparatus

Ordinary laboratory apparatus and the following:

4.3.1 Coulometric Karl Fischer titrator, consisting of a control unit, a titration-cell assembly (see Figure 1) (equipped with a generator cell, a cathode solution cell, dual platinum sensing electrodes and a magnetic stirrer). 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.

4.3.2 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 flowmeter and carrier-gas drying tubes containing desiccant.

1) SICAPENT[®] is a trade name of MERCK.

4.3.3 **Microsyringe**, capacity 10 µl, calibrated.

4.3.4 **Sample boat**: aluminium foil may be used to hand fashion a disposable boat large enough to contain the sample and small enough to fit into the heating tube.

4.4 **Preparation of sample**

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

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

4.4.3 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.

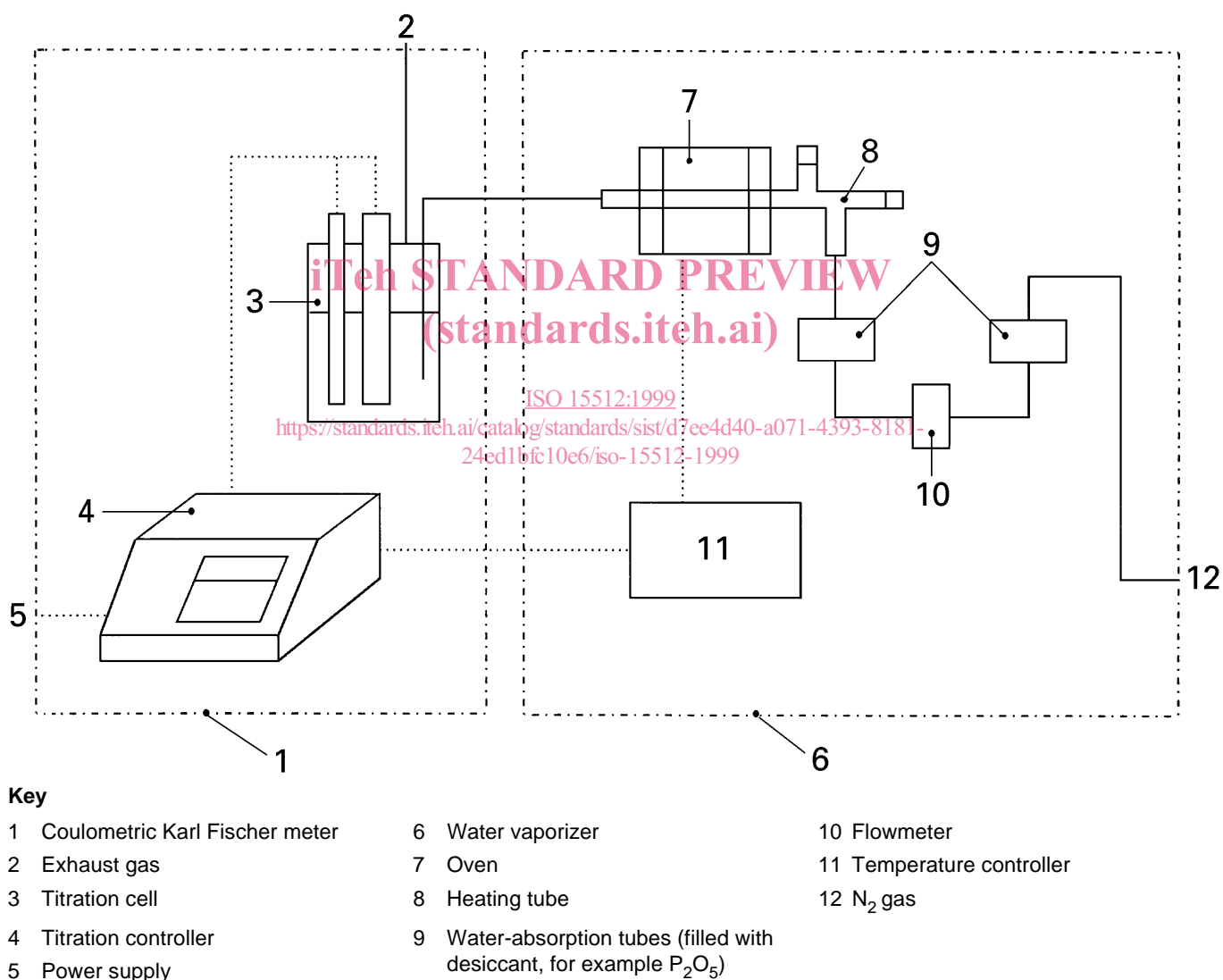


Figure 1 — Flow chart of system used for the determination of water content using method B