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Plastics — Determination of water content

Plastiques — Dosage de l'eau

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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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For an explanation on 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 the following URL: Foreword – Supplementary information

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

This fourth edition cancels and replaces the third edition (ISO 15512:2014), of which it constitutes a minor revision to update information in 6.2.1.

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 have to, 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 International Standard is intended to be followed strictly.

The temperature settings for the vaporization method are not specified in this International 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 has to 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 interlaboratory comparability.

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 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 %.

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

1.2 Four alternative methods are specified in this International Standard.

- **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 mm × 4 mm × 3 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 of the collected water. It can be used for all plastics and is applicable to granules smaller than 4 mm × 4 mm × 3 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 mm × 4 mm × 3 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.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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 Titration medium, methanol anhydrous, having a water content less than 0,1 % by mass. Other solvents can be used if shown to be comparable.

3.2.2 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.

3.3 Apparatus

Ordinary laboratory apparatus and the following.

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

3.3.2 Conical titration flasks, approximately 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 suitable drying agent.

3.3.5 Heaters, suitable for the conical titration flasks (3.3.2).

3.3.6 Desiccator, containing a suitable desiccant.

3.3.7 Analytical balance, accurate to 0,2 mg.

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

3.4 Preparation of test sample

3.4.1 Granules or powder

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 and then cool it over a suitable desiccant.

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.

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.

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 of anhydrous methanol (3.2.1) 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 (3.3.6) 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 (3.3.8) to titrate the contents of each flask with Karl Fischer reagent (3.2.2).

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

3.6 Expression of results

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

$$w = \frac{\left(V_1 - V_2\right) \times T}{m} \times 100 \qquad (1)$$

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 the formula simplifies to $V \times T$.

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 to a subsequent revision.

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

4 Method B1 — Water vaporization using a heating tube oven

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:

$$I_2 + SO_2 + H_2O \rightarrow 2HI + SO_3$$

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

$$2I^- \rightarrow I_2 + 2e^-$$

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.

4.2.1 Anode solution, containing iodide ions to generate iodine in the reaction mixture, 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.

4.2.3 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.

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 Nitrogen gas (N_2), 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 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 may require a diaphragm for the best possible accuracy. This can be checked with the equipment supplier.

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 flow meter, and carrier-gas drying tubes containing desiccant.

4.3.3 Microsyringe, with capacity of 10 µl, calibrated.

4.3.4 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.

4.3.5 Suitable desiccant, for the carrier-gas drying tubes (4.3.2).

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

4.3.7 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.