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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.

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 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.

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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 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 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 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.

— **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.

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— **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

4.1 Principle

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

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).

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