

**BS EN ISO 15512:2016**



**BSI Standards Publication**

# **Plastics — Determination of water content (ISO 15512:2016)**



**National foreword**

This British Standard is the UK implementation of EN ISO 15512:2016. It supersedes BS EN ISO 15512:2014 which is withdrawn.

The UK participation in its preparation was entrusted to Technical Committee PRI/21, Testing of plastics.

A list of organizations represented on this committee can be obtained on request to its secretary.

This publication does not purport to include all the necessary provisions of a contract. Users are responsible for its correct application.

© The British Standards Institution 2016.  
Published by BSI Standards Limited 2016

ISBN 978 0 580 92336 4

ICS 83.080.01

**Compliance with a British Standard cannot confer immunity from legal obligations.**

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 November 2016.

**Amendments/corrigenda issued since publication**

| Date | Text affected |
|------|---------------|
|------|---------------|

---

English Version

## Plastics - Determination of water content (ISO 15512:2016)

Plastiques - Dosage de l'eau (ISO 15512:2016)

Kunststoffe - Bestimmung des Wassergehaltes (ISO 15512:2016)

This European Standard was approved by CEN on 29 September 2016.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the CEN-CENELEC Management Centre or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the CEN-CENELEC Management Centre has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and United Kingdom.



EUROPEAN COMMITTEE FOR STANDARDIZATION  
COMITÉ EUROPÉEN DE NORMALISATION  
EUROPÄISCHES KOMITEE FÜR NORMUNG

**CEN-CENELEC Management Centre: Avenue Marnix 17, B-1000 Brussels**

## European foreword

This document (EN ISO 15512:2016) has been prepared by Technical Committee ISO/TC 61 “Plastics” in collaboration with Technical Committee CEN/TC 249 “Plastics” the secretariat of which is held by NBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2017, and conflicting national standards shall be withdrawn at the latest by May 2017.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. CEN [and/or CENELEC] shall not be held responsible for identifying any or all such patent rights.

This document supersedes EN ISO 15512:2014.

According to the CEN-CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

### Endorsement notice

The text of ISO 15512:2016 has been approved by CEN as EN ISO 15512:2016 without any modification.

# Contents

Page

|                                                                    |           |
|--------------------------------------------------------------------|-----------|
| <b>Foreword</b>                                                    | <b>v</b>  |
| <b>Introduction</b>                                                | <b>vi</b> |
| <b>1 Scope</b>                                                     | <b>1</b>  |
| <b>2 Normative references</b>                                      | <b>1</b>  |
| <b>3 Method A — Extraction with anhydrous methanol</b>             | <b>1</b>  |
| 3.1 Principle                                                      | 1         |
| 3.2 Reagents                                                       | 2         |
| 3.3 Apparatus                                                      | 2         |
| 3.4 Preparation of test sample                                     | 2         |
| 3.4.1 Granules or powder                                           | 2         |
| 3.4.2 Finished articles                                            | 2         |
| 3.5 Procedure                                                      | 3         |
| 3.5.1 Precautions                                                  | 3         |
| 3.5.2 Preparation of test portions                                 | 3         |
| 3.5.3 Determination                                                | 3         |
| 3.6 Expression of results                                          | 3         |
| 3.7 Precision                                                      | 4         |
| <b>4 Method B1 — Water vaporization using a heating tube oven</b>  | <b>4</b>  |
| 4.1 Principle                                                      | 4         |
| 4.2 Reagents                                                       | 4         |
| 4.3 Apparatus                                                      | 5         |
| 4.4 Preparation of test sample                                     | 7         |
| 4.5 Procedure                                                      | 7         |
| 4.5.1 Precautions                                                  | 7         |
| 4.5.2 Preparation of apparatus                                     | 7         |
| 4.5.3 Equipment check                                              | 7         |
| 4.5.4 Determination                                                | 8         |
| 4.6 Expression of results                                          | 8         |
| 4.7 Precision                                                      | 9         |
| <b>5 Method B2 — Water vaporization using a heated sample vial</b> | <b>9</b>  |
| 5.1 Principle                                                      | 9         |
| 5.2 Reagents                                                       | 9         |
| 5.3 Apparatus                                                      | 10        |
| 5.4 Preparation of test sample                                     | 11        |
| 5.5 Procedure                                                      | 12        |
| 5.5.1 Preparation of apparatus                                     | 12        |
| 5.5.2 Equipment check                                              | 12        |
| 5.5.3 Determination                                                | 12        |
| 5.6 Expression of results                                          | 14        |
| 5.7 Precision                                                      | 14        |
| <b>6 Method C — Manometric method</b>                              | <b>15</b> |
| 6.1 Principle                                                      | 15        |
| 6.2 Reagents                                                       | 15        |
| 6.3 Apparatus                                                      | 15        |
| 6.4 Preparation of test sample                                     | 16        |
| 6.5 Procedure                                                      | 16        |
| 6.5.1 Equipment                                                    | 16        |
| 6.5.2 Outgassing                                                   | 17        |
| 6.5.3 Calibration                                                  | 17        |
| 6.5.4 Determination                                                | 19        |
| 6.6 Expression of results                                          | 20        |
| 6.7 Precision                                                      | 20        |

|                              |                                                                                               |           |
|------------------------------|-----------------------------------------------------------------------------------------------|-----------|
| <b>7</b>                     | <b>Test report</b> .....                                                                      | <b>20</b> |
| <b>Annex A</b> (informative) | <b>Alternative sample preparation methods and titration methods</b> .....                     | <b>21</b> |
| <b>Annex B</b> (informative) | <b>Selection of the optimum heating temperature for the water content determination</b> ..... | <b>22</b> |
| <b>Annex C</b> (normative)   | <b>Determination of the water content of a water standard</b> .....                           | <b>24</b> |
| <b>Bibliography</b> .....    |                                                                                               | <b>25</b> |

## 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](http://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](http://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 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):

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

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 the formula simplifies to  $V \times T$ .