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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

Plastics — Polyamides (PA) — Determination of water content

Plastiques — Polyamides (PA) — Détermination de la teneur en eau

Reference number
ISO 960:1988 (E)

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 960 was prepared by Technical Committee ISO/TC 61, *Plastics*.

It cancels and replaces ISO Recommendation R 960 : 1969, of which it constitutes a technical revision.

Plastics — Polyamides (PA) — Determination of water content

1 Scope

1.1 This International Standard specifies methods for the determination of the water content of polyamides and copolyamides as granules and finished articles. The methods are applicable for the determination of water contents down to 0,01 % (*m/m*). The water content is of importance in connection with the processing of PA, during which it should have a low value to prevent degradation, and for checking the moisture content of test specimens and finished articles.

1.2 Four alternative methods are specified in this International Standard.

Method A is an extraction method using anhydrous methanol and titration of the water by the Karl Fischer method. It is applicable to granules having a maximum size of 4 mm × 4 mm × 3 mm and can be used for all polyamides and copolyamides.

Method B is a method of extraction by melting under vacuum and titration of the water by the Karl Fischer method. It may be applied to granules or pieces of mouldings but not to fine powders (particle size less than 400 µm). For low water content (less than 0,1 % (*m/m*)) and precise determination, it is necessary to make a correction for the water of polycondensation¹⁾. This method is not recommended when, during the determination, due to a change in molecular mass, additional water is formed or existing water is bound, except when appropriate correction can be made.

Method C is a method of extraction by dissolving in a mixture of 3-methylphenol and toluene and titration of the water by the Karl Fischer method. It yields results close to those obtained by method A. It has the advantage of being applicable to granules and powders of all sizes and to finished articles with little or no reduction in size.

NOTE — The three methods A, B and C differ in the procedure for isolating the water, which is determined in all three cases by the Karl Fischer method.

Method D is a manometric method. The water content is determined from the pressure increase after evaporation of the water under vacuum. This method cannot be used for polyamides polymerized with strong acids.

1.3 Method C is the reference method.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards listed below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 760 : 1978, *Determination of water — Karl Fischer method (General method)*.

ISO 3146 : 1985, *Plastics — Determination of melting behaviour (melting temperature or melting range) of semi-crystalline polymers*.

3 Method A — Extraction with anhydrous methanol

3.1 Principle

Extraction of a test portion with anhydrous methanol and determination of the extracted water by 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 % (*m/m*).

3.2.2 Karl Fischer reagent (see 6.2).

3.3 Apparatus

Ordinary laboratory apparatus and

3.3.1 Glass flasks, of capacity 250 ml, provided with ground glass or rubber stoppers.

1) See KLINE, G.M., *Analytical Chemistry of Polymers. Intersc. Pub.*, Part 1, p. 282 (1958).

3.3.2 Conical titration flasks, of capacity 150 ml, 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 Tubes, straight, containing calcium chloride, or other means of drying, with ground joints.

3.3.5 Means for heating the flasks (3.3.2), electrically, by hot air, or by any other suitable means.

3.3.6 Automatically filling pipettes, of capacity 50 ml (see figure 1).

3.3.7 Woolf bottles, with two tubes.

3.3.8 Calcium chloride tubes, curved or U-shaped.

3.3.9 Rubber bulb.

3.3.10 Pipette, of capacity 10 ml.

3.3.11 Desiccator, containing calcium chloride or another suitable drying agent.

3.3.12 Balance, accurate to 0,2 mg.

3.3.13 Apparatus for determining water content by the Karl Fischer method, in accordance with ISO 760.

3.4 Preparation of test sample

3.4.1 Granules

Place a representative test sample of approximately 100 g in a perfectly dry glass flask (3.3.1) and immediately close the flask with either a ground glass or a rubber stopper.

NOTE — It is desirable to predry the container in an oven and to cool it above a suitable water absorbent, for example silica gel.

3.4.2 Finished articles

Cut or saw the test sample into pieces of a few millimetres dimensions. Proceed quickly to minimize moisture absorption. Store the test sample as specified in 3.4.1.

3.5 Number of tests

Carry out two determinations for each sample. Use test portions of 10 to 15 g, depending on the estimated water content.

3.6 Procedure

3.6.1 Carefully dry the apparatus.

3.6.2 Weigh the test portion to the nearest 1 mg into a conical titration flask fitted with a ground glass stopper (3.3.2); let m be the mass, in grams, of this test portion. Measure 50 ml of anhydrous methanol (3.2.1) with an automatically filling pipette (3.3.6) and add it to 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.

3.6.3 Unstopper the flasks and quickly attach them to the reflux condensers (3.3.3) fitted on top with the calcium chloride tubes (3.3.4). Boil the contents of the conical flasks under reflux for 3 h and leave for 45 minutes to cool to room temperature. Separate the flasks from the condensers, quickly stopper them and place them in the desiccator.

3.6.4 Titrate the contents of each flask with the Karl Fischer reagent as indicated in clause 6.

4 Method B — Extraction by melting under vacuum

4.1 Principle

Melting of a test portion under vacuum at a temperature approximately 30 °C above the melting point of the resin and determination of the amount of water collected by the Karl Fischer method.

4.2 Reagents

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

4.2.1 Methanol, anhydrous, having a water content less than 0,1 % (m/m).

4.2.2 Karl Fischer reagent (see 6.2).

4.3 Apparatus

Ordinary laboratory apparatus, and

4.3.1 Vacuum melting unit, capable of maintaining a pressure of less than 500 Pa*). A four-directional unit, given as an example (see figure 2), comprises the following parts:

4.3.1.1 Heating block, made up of an insulated aluminium cylinder equipped with an electric heating collar having approximately 800 W power and 160 mm height. The temperature of the cyl-

*) 500 Pa = 5 mbar $\approx$ 4 mmHg