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INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 960

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PLASTICS

DETERMINATION OF THE WATER CONTENT IN POLYAMIDES

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BRIEF HISTORY

The ISO Recommendation R 960, *Plastics – Determination of the water content in polyamides*, was drawn up by Technical Committee ISO/TC 61, *Plastics*, the Secretariat of which is held by the American National Standards Institute (ANSI).

Work on this question led to the adoption of a Draft ISO Recommendation.

In March 1967, this Draft ISO Recommendation (No. 1004) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies :

Australia	Hungary	Romania
Belgium	India	South Africa, Rep. of
Brazil	Iran	Spain
Bulgaria	Ireland	Sweden
Canada	Israel	Switzerland
Chile	Italy	Turkey
Czechoslovakia	Japan	U.A.R.
Finland	Korea, Dem. P. Rep. of	United Kingdom
France	Netherlands	U.S.A.
Germany	New Zealand	U.S.S.R.
Greece	Poland	

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No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in February 1969, to accept it as an ISO RECOMMENDATION.

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ISO Recommendation | R 960 | February 1969

PLASTICS

DETERMINATION OF THE WATER CONTENT IN POLYAMIDES

1. SCOPE

1.1 This ISO Recommendation describes three alternative methods for determining the water content in polyamides and polyamide copolymers.

1.2 The water content is of importance when evaluating results found with the following methods of test :

(*a*) determination of the viscosity number of polyamide resins in dilute solution (see ISO Recommendation R 307);

(*b*) determination of the percentage of extractable materials in polyamides (see ISO Recommendation R 599);

(*c*) determination of the viscosity ratio of polyamide in concentrated solution (see ISO Recommendation R 600).

The knowledge of the water content is also of importance when controlling processes and converting polyamides.

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

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2. FIELDS OF APPLICATION OF THE THREE METHODS

2.1 **Method A** is an extraction method using anhydrous methanol.

It is applicable to granules having a maximum size of 4 mm × 4 mm × 3 mm and can be used for all polyamides and copolymers of polyamides insoluble in methanol.

2.2 **Method B** is a method of extraction by melting under vacuum.

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 %) and precise determinations, a correction for the water of polycondensation should be made.*

This method is not recommended when, during the determination, due to a variation of the molecular mass, additional water is formed or existing water is bound, except when appropriate correction can be made.

2.3 **Method C** is a method of extraction by dissolving in metacresol.

It yields results close to the results obtained by Method A. It has the advantage of not being connected with polyamide granulation.

* See KLINE. Analytical chemistry of polymers. Intersc. Pub., 1958, p. 282.

3. METHOD A – EXTRACTION USING ANHYDROUS METHANOL

3.1 Principle

The granules are extracted with anhydrous methanol and the water extracted is determined by the Karl Fischer method.

3.2 Reagents

3.2.1 *Anhydrous methanol*, analytical grade, water content less than 0.1 %.

3.2.2 *Karl Fischer reagent* (see section 6).

3.3 Apparatus

3.3.1 *Glass flasks* with ground glass or rubber stoppers, 250 ml.

3.3.2 *Conical titration flasks*, 150 ml, with standard ground necks equipped with stoppers.

3.3.3 *Reflux condenser* with ground neck to fit on the flasks (3.3.2) and on the tubes (3.3.4).

3.3.4 *Straight calcium chloride tubes* or other means of drying, with ground joints.

3.3.5 *Means for heating the flasks* (3.3.2), electrically or by hot air.

3.3.6 *Automatically filling pipettes*, 50 ml (see Fig. 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*, 10 ml.

3.3.11 *Calcium chloride desiccator.*

3.3.12 *Balance*, accurate to 0.0001 g.

3.3.13 *Apparatus for determining water content by the Karl Fischer method* (see section 6).

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3.4 Preparation of sample

A representative sample of approximately 100 g should be placed in a perfectly dry 250 ml glass flask (3.3.1) which should be stoppered immediately with either a ground glass stopper or a rubber stopper.

3.5 Number of tests and test portions

Two determinations should be carried out for each sample.

Test portions of 10 or 15 g should be used according to the estimated water content.

3.6 Procedure

The apparatus to be used in the determination should be dried thoroughly.

Boil 50 ml of the Karl Fischer reagent for one hour; pour off the reagent and leave the apparatus to cool to room temperature.

In order to avoid the entrance of moist air, a water bath should not be used.

Weigh the test portion in a 150 ml ground glass stoppered conical titration flask (3.3.2) to the nearest 0.001 g. Let M be the mass, in grammes, of this test portion. Measure 50 ml of anhydrous methanol (3.2.1) with an automatically filling pipette (3.3.6) and add to the 150 ml conical flask containing the test portion. At the same time, add 50 ml anhydrous methanol (3.2.1) to another conical flask for a blank test. Stopper the flasks.

Keep the stoppered flasks in the desiccator (3.3.11) awaiting the continuation of the test.

Unstopper the flasks and rapidly join them to the reflux condensers (3.3.3) fitted on top with calcium chloride tubes (3.3.4).

Boil the contents of the conical flasks under reflux for 3 hours and leave for 45 minutes to cool to room temperature.

Separate the flasks from the condensers and quickly stopper them. Then place them in the desiccator.

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

4. METHOD B – EXTRACTION BY MELTING UNDER VACUUM

4.1 Principle

The polyamides are melted under vacuum at a temperature approximately 30 °C above their melting point and the amount of water collected is determined by the Karl Fischer method.

4.2 Reagents

4.2.1 *Methanol* containing less than 0.1 % water.

4.2.2 *Karl Fischer reagent* (see section 6).

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4.3 Apparatus

4.3.1 *Vacuum melting unit* (capable of maintaining a pressure of 4 mmHg absolute or less). A four-directional unit given as an example is made up of the following parts (see Fig. 2; the reference letters are taken from the figure):

(*a*) *Heating block* made up of an insulated aluminium cylinder equipped with an electric heating collar having approximately 800 W power and standing 160 mm high. The temperature of the cylinder is adjusted to approximately 30 °C above the melting point of the test resin as determined by the method described in ISO Recommendation R . . . *, *Plastics – Determination of the melting point of polyamides*, and maintained within ± 5 °C. This cylinder, which is 200 mm in height and 160 mm in diameter, has a centre hole for a thermometer to be installed, and four holes 170 mm deep, 25 mm in diameter, and spaced at regular intervals in relation to its vertical axis.

(*b*) *Four traps* (P) in 6 mm × 8 mm U-shaped glass tubes in accordance with Figure 2. These parts are inserted in a 1 litre Dewar flask containing a mixture of methanol or acetone and carbon dioxide snow (making it possible to obtain a temperature of approximately −78 °C).

(*c*) *Rubber vacuum tubing.*

(*d*) *Two glass stopcocks* (R_1 and R_2), for each direction.

(*e*) *Four heat-proof 24/40 internally ground glass tubes* (A), 22 mm in diameter and 220 mm over-all length (tube plus ground portion), to contain the test tubes mentioned below, and to be inserted in the four holes provided in the cylinder.

(*f*) *Four 24/40 externally ground heat-proof glass fittings* (B).

* At present Draft ISO Recommendation No. 1218.

4.3.2 *Four glass test tubes*, 16 mm in diameter and 160 mm long, which contain the test portions.

4.3.3 *Apparatus* for producing a pressure less than 4 mmHg in the traps.

4.3.4 *Manometer* permitting the measurement of this pressure.

4.3.5 *Glass flask* with ground glass or rubber stopper, 250 ml.

4.3.6 *Conical titration flasks*, 150 ml, with standard ground necks.

4.3.7 *Automatic burette.*

4.3.8 *Hot-air oven* giving a temperature of at least 100 °C.

4.3.9 *Desiccators* capable of containing all of the glassware needed in the determination.

4.3.10 *Balance*, accurate to 0.0001 g.

4.3.11 *Apparatus for determining water content by the Karl Fischer method* (see section 6).

4.4 Preparation of test sample

A representative sample of approximately 100 g should be placed in a perfectly dry 250 ml glass flask (4.3.5), which should be stoppered immediately with either a ground glass stopper or a rubber stopper.

4.5 Number of tests and test portions

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Two determinations should be made.

The test portions used should be

– 10 g when the water content is estimated as less than 0.5 %,

– 2 g when the water content is estimated as higher than 0.5 %.

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4.6 Procedure

4.6.1 Depending on the vacuum melting unit (4.3.1) available, one or several simultaneous different determinations may be carried out.

In the case of the apparatus described as an example in clause 4.3, the procedure to be followed is indicated below. For any other apparatus it would be best to follow the procedure taking the same general precautions.

4.6.2 The glass accessories required for the determination are removed from the oven (4.3.8), where they are permanently placed to shelter them from moisture, and placed in the desiccators (4.3.9) until completely cooled.

Weigh the test portion in a calibrated test tube (4.3.2) to the nearest 0.001 g. Let M be the mass, in grammes, of this test portion.

Seal the test tube with a completely dry rubber stopper and replace in the desiccator.

Place the trap P in the tubing and insert in the Dewar flask. The trap should be subjected to a vacuum by attaching the vacuum apparatus (4.3.3) to the tubing end and opening stopcock R_2, stopcock R_1 being closed.

Wait 5 minutes in order that the trap is at a temperature of –78 °C.

Place the test tube, after removal of its stopper, in a ground tube A.

Close the ground tube A with a glass fitting B and join to the trap by rubber vacuum tube and stopcock R_1.

Open the stopcock R_1 and ascertain the proper vacuum by reading the manometer (4.3.4).

Place the ground tube for 30 minutes in the heating block previously brought to the required temperature (30 °C above the melting point as determined by the method described in ISO Recommendation R . . .*, *Plastics – Determination of the melting point of polyamides*) while the vacuum apparatus is continuously functioning.

Close stopcocks R_2 and R_1 and shut off the vacuum apparatus.

Remove the ground tube from the heating block and from the tubing.

Remove the trap from the Dewar flask and leave to regain room temperature while keeping the two stopcocks R_1 and R_2 closed.

Dry the outside of the trap carefully and disconnect the vacuum tubes.

Place in a desiccator one of the titration flasks of the apparatus for determining the percentage of water according to the Karl Fischer method.

One of the ends of the trap being inserted in the titration flask, add 10 ml of anhydrous methanol (4.2.1) to the other end by means of an automatic burette (4.3.7). Evacuation should be quickly carried out by continuously rinsing the water in the trap while taking care to scour all of the sides of the tube with methanol.

Separate the U-shaped tube from the reagent bottle and carry out the determination without waiting, as indicated in section 6.

Carry out a blank test on a further 10 ml of the methanol (4.2.1) used to wash the trap.

5. METHOD C – DETERMINATION BY DISSOLVING IN METACRESOL

5.1 Principle

The polyamide is dissolved in boiling metacresol and the percentage of water in the solution is determined by the Karl Fischer method; the addition of toluene prevents oxidation and lowers the boiling temperature.

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5.2 Reagents

5.2.1 *Anhydrous metacresol*, water content less than 0.015 %.

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5.2.2 *Anhydrous toluene*, water content less than 0.005 %.

5.2.3 *Anhydrous methanol.*

5.2.4 *Karl Fischer reagent* (see section 6).

5.3 Apparatus

5.3.1 *Glass flask* with ground glass or rubber stopper, 250 ml.

5.3.2 *Conical titration flask*, 250 ml, with standard ground neck.

5.3.3 *Reflux condenser*, with ground neck to fit on the flask (5.3.2) and on the tube (5.3.4).

5.3.4 *Tube* with silica-gel, anhydrous calcium chloride, or other effective means of drying.

5.3.5 *Electrical or hot-air heating.*

5.3.6 *Graduated burette*, 100 ml.

5.3.7 *Pipette*, 10 ml.

5.3.8 *Balance*, accurate to 0.0001 g.

5.3.9 *Fragmented pumice stone.*

5.3.10 *Apparatus for determining water content by the Karl Fischer method* (see section 6).

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* At present Draft ISO Recommendation No. 1218.