International Standard 599

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION●МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ●ORGANISATION INTERNATIONALE DE NORMALISATION

Plastics — Polyamide homopolymers — Determination of matter extractable by boiling methanol

Plastiques — Homopolymères polyamides — Détermination des matières extractibles par le méthanol bouillant

Second edition — 1985-06-15

UDC 678.675 : 543.832 **Ref. No. ISO 599-1985 (E)**

Descriptors : plastics, homopolymers, polyamide, chemical analysis, determination of content, soluble matter, extraction method, carbinols.

Price based on 3 pages

Foreword

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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 599 was prepared by Technical Committee ISO/TC 61, *Plastics*.

ISO 599 was first published in 1979. This second edition cancels and replaces the first edition, of which it constitutes a minor revision.

Printed in Switzerland

Plastics — Polyamide homopolymers — Determination of matter extractable by boiling methanol

1 Scope and field of application

This International Standard specifies a method for the determination of the matter extractable from polyamide homopolymers by boiling methanol.

Polyamides may contain monomers and oligomers and, possibly, additives. Consequently, if it is desired to know the monomer and oligomer contents, a correction has to be made for the amount of co-extracted additives, if any. Since these additives may vary widely in nature, no general instruction as to their determination can be given.

Knowledge of the methanol-extractable matter content of polyamides may be required for the determination of the viscosity number of polyamides (see ISO 307).

2 References

ISO 307, *Plastics — Polyamides — Determination of viscosity number.*

ISO 565, *Test sieves — Woven metal wire cloth, perforated plate and electroformed sheet — Nominal sizes of openings.*

3 Principle

Extraction of ground polyamide homopolymers with methanol at its boiling point. Careful evaporation of the solvent. Drying of the residue at moderate temperatures, and weighing.

4 Definition

For the purposes of this International Standard, the following definition applies :

methanol-extractable matter : The substances extracted from a sample of polyamide by boiling methanol, under the specified conditions.

5 Apparatus and materials

5.1 Mill, for reducing the sample to the required grain size.

A mill in which the sample is cut between rotating and stationary blades is preferred. Large pieces can be reduced in size with a pair of shears before they are fed to the mill.

5.2 Sieves, with aperture size of 710 µm and 500 µm respectively, complying with the requirements of ISO 565.

5.3 Balance, accurate to 0,000 1 g.

5.4 Extraction apparatus that will accommodate an extraction crucible or porous ceramic thimble containing the test portion.

The apparatus shall be of such a design that the crucible or thimble is heated by the rising methanol vapour.

Examples of appropriate extraction apparatus according to this principle are :

a) — extraction flask, of capacity 250 ml;

— extraction chamber to accommodate the extraction crucible so that it is enveloped on all sides by the rising methanol vapour and that the condensed methanol drips through it continuously;

— glass triangle to support the crucible;

— reflux condenser;

— sintered glass filter crucible grade P 100 (pore size index 40 to 100 µm), of capacity 30 ml;

— porcelain filter-plate of slightly smaller diameter than the crucible, with holes of diameter 0,4 mm;

b) — extraction flask, of capacity 250 ml;

— jacketed Soxhlet extractor;

— reflux condenser;

— sintered glass filter crucible grade P 100 (pore size index 40 to 100 µm), of capacity 30 ml, or a porous ceramic thimble of similar porosity and capacity. The dimensions shall be such that the crucible or thimble can be satisfactorily accommodated in the Soxhlet apparatus;

— porcelain filter-plate of slightly smaller diameter than the crucible or thimble, as appropriate, with holes of diameter 0,4 mm.

5.5 Appropriate heating device for the extraction apparatus.

5.6 Distillation apparatus, fitted with a Vigreux or equivalent distillation column of length at least 400 mm, or

5.7 Rotating evaporator, connected to an aspirator.

5.8 Water bath.

5.9 Vacuum drying cabinet, equipped with an aspirator or other means to achieve a pressure between 1 and 2,5 kPa*.

NOTE — Pressures lower than 1 kPa shall not be applied to avoid loss of caprolactam from the extract obtained with PA 6.

5.10 Desiccator, containing a drying agent such as calcium chloride, phosphorus(V) oxide or silica gel.

5.11 Methanol, of recognized analytical grade.

5.12 Solid carbon dioxide, if necessary.

6 Preparation of test sample

Take a representative sample of the polymer and grind it in the mill (5.1). Grind the material in small portions at a time, to prevent undue heat development above about 40 °C, letting the mill cool down in between. Solid carbon dioxide (5.12) may be ground with the polymer to prevent heat build-up. With a larger mill, having a greater heat capacity, these precautions may not be required. Collect the fraction that passes through the sieve (5.2) with mesh opening 710 µm but not through the one with mesh opening 500 µm.

7 Procedure

7.1 Test portion

Weigh, to the nearest 0,01 g, 5 ± 0,5 g (mass m_0) of the test sample into the filter crucible or porous thimble (see 5.4).

NOTES

1 If the methanol-extractable matter content is expected to be less than 0,5 % (m/m), the mass of the test portion (7.1) should be increased to obtain a residue of at least 0,025 g.

2 Polyamides may contain a small amount of water, forming part of the mass of the test portion (m_0). The water content is not accounted for in the calculation of the methanol-extractable matter content since its effect is small, compared with the variance of the determination.

7.2 Determination

Clean and dry the extraction flask (see 5.4), allow it to cool in the desiccator (5.10) for between 30 and 40 min and then weigh it to the nearest 0,000 2 g (mass m_1).

Cover the test portion (7.1) with the filter-plate, pour about 50 ml of the methanol (5.11) into the extraction flask, place the crucible or thimble containing the test portion in the extraction chamber and fit the condenser to the chamber. Heat the methanol in the flask to boiling. If the apparatus described under 5.4 a) is used, adjust the rate of reflux to 1 or 2 drops per second and ensure that the drops fall within the crucible. If a Soxhlet extractor as described under 5.4 b) is used, adjust the heating so that there are five to eight siphonings per hour.

Extract for a period of 3 h ± 5 min and allow the extractor to cool to ambient temperature, overnight if necessary.

Detach the extraction flask with its contents and continue according to either of the following procedures (see note 1) :

— Using the distillation apparatus (5.6) :

Carefully evaporate the methanol down to approximately 20 ml on the water bath (5.8). Place the flask in the vacuum drying cabinet (5.9) and carefully evaporate the residual methanol at ambient temperature in vacuo.

— Using the rotating evaporator (5.7) :

Connect the flask to the rotating evaporator. Evaporate the methanol in vacuo (see note 2) at a temperature between 25 and 35 °C.

Finally, in either case, clean and dry the outside of the flask, and then place it in the vacuum drying cabinet, maintained at 40 ± 2 °C, for 4 h. Allow the flask to cool in the desiccator for between 30 and 40 min and weigh it to the nearest 0,000 2 g. Repeat the weighing after the flask has been dried for another 2 h at 40 ± 2 °C. If the difference between the results of the two weighings is more than 2 % of the mass of the extract, continue the drying until the difference between two successive weighings is less than 2 % of the mass of the extract.

Carry out two determinations.

NOTES

1 For high methanol-extractable matter contents, the drying time may be prolonged if a rotating evaporator is not used.

2 If the rotating evaporator is used, frothing of the extract occasionally takes place. As this can lead to a loss of some of the extract, the determination should be repeated if frothing occurs.

8 Expression of results

The methanol-extractable matter content is given, as a percentage by mass, by the formula

$$\frac{m_2 - m_1}{m_0} \times 100$$

where

m_0 is the mass, in grams, of the test portion;

m_1 is the mass, in grams, of the flask;

m_2 is the mass, in grams, of the flask and residue.

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* 1 kPa = 7,5 mmHg = 1 kN/m^2