SLOVENSKI STANDARD
SIST EN ISO 307:2000

01-maj-2000

Polimerni materiali - Poliamidi - Določanje viskoznostnega števila (ISO 307:1994)

Plastics - Polyamides - Determination of viscosity number (ISO 307:1994)

Kunststoffe - Polyamide - Bestimmung der Viskositätszahl (ISO 307:1994)

Plastiques - Polyamides - Détermination de l'indice de viscosité (ISO 307:1994)

Ta slovenski standard je istoveten z: **EN ISO 307:1997**

ICS:

83.080.20 Plastomeri Thermoplastic materials

SIST EN ISO 307:2000 **en**

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN ISO 307

November 1997

ICS 83.080.20

Descriptors: see ISO document

English version

Plastics - Polyamides - Determination of viscosity number (ISO 307:1994)

Plastiques - Polyamides - Détermination de l'indice de viscosité (ISO 307:1994)

Kunststoffe - Polyamide - Bestimmung der Viskositätszahl (ISO 307:1994)

This European Standard was approved by CEN on 16 October 1997.

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 Central Secretariat 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 Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

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

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Foreword

The text of the International Standard from Technical Committee ISO/TC 61 "Plastics" of the International Organization for Standardization (ISO) has been taken over as an European Standard by Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

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of an identical text or by endorsement, at the latest by May 1998, and conflicting national standards shall be withdrawn at the latest by May 1998.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

Endorsement notice

The text of the International Standard ISO 307:1994 has been approved by CEN as a European Standard without any modification.

NOTE: Normative references to International Standards are listed in annex ZA (normative).

Annex ZA (normative)
Normative references to international publications with their relevant European publications

This European Standard incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this European Standard only when incorporated in it by amendment or revision. For undated references the latest edition of the publication referred to applies.

Publication	Year	Title	EN	Year
ISO 3451-4	1986	Plastics - Determination of ash - Part 4: Polyamides	EN ISO 3451-4	1995

INTERNATIONAL STANDARD

ISO 307

Third edition
1994-09-01

Plastics — Polyamides — Determination of viscosity number

Plastiques — Polyamides — Détermination de l'indice de viscosité

Reference number
ISO 307:1994(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 voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

International Standard ISO 307 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This third edition cancels and replaces the second edition (ISO 307:1984), of which it constitutes a technical revision.

Annex A forms an integral part of this International Standard.

International Organization for Standardization
Case Postale 56 • CH-1211 Genève 20 • Switzerland

Printed in Switzerland

Plastics — Polyamides — Determination of viscosity number

1 Scope

This International Standard specifies a method for the determination of the viscosity number of dilute solutions of polyamides in certain specified solvents.

The method is applicable to the polyamides designated PA 6, PA 66, PA 69, PA 610, PA 612, PA 11, PA 12 and PA MXD6 as defined in ISO 1874-1, as well as to copolyamides and other polyamides that are soluble in one of the specified solvents under the specified conditions.

The method is not applicable to polyamides produced by anionic polymerization of lactams or produced with crosslinking agents; such polyamides are normally insoluble in the specified solvents.

The viscosity number is determined by the general procedure specified in ISO 1628-1, observing the particular conditions specified in this International Standard.

The determination of the viscosity number of a polyamide provides a measure of the relative molecular mass of the polymer.

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 indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 472:1988, *Plastics — Vocabulary.*

ISO 599:1985, *Plastics — Polyamide homopolymers — Determination of matter extractable by boiling methanol.*

ISO 960:1988, *Plastics — Polyamides (PA) — Determination of water content.*

ISO 1042:1983, *Laboratory glassware — One-mark volumetric flasks.*

ISO 1628-1:1984, *Guidelines for the standardization of methods for the determination of viscosity number and limiting viscosity number of polymers in dilute solution — Part 1: General conditions.*

ISO 1874-1:1992, *Plastics — Polyamide (PA) moulding and extrusion materials — Part 1: Designation.*

ISO 3105:—[1], *Glass capillary kinematic viscometers — Specifications and operating instructions.*

ISO 3451-4:1986, *Plastics — Determination of ash — Part 4: Polyamides.*

ISO 6427:1992, *Plastics — Determination of matter extractable by organic solvents (conventional methods).*

ASTM D 789:1986, *Standard test methods for determination of relative viscosity, melting point, and moisture content of polyamide (PA).*

3 Definitions

For the purposes of this International Standard, the definitions given in ISO 1628-1 and the following definition apply.

1) To be published. (Revision of ISO 3105:1976)

3.1 viscosity number (of a polymer): The value given by the formula

$$\left(\frac{\eta}{\eta_0} - 1\right)\frac{1}{\varrho_P}$$

where

η is the viscosity of a solution of the polymer in a specified solvent;

η_0 is the viscosity of the solvent, expressed in the same units as η;

ϱ_P is the concentration, in grams per millilitre, of the polymer in the solution.

The viscosity number is usually expressed in millilitres per gram.

4 Principle

The times of flow of a solvent and a solution of the polyamide at a concentration of 0,005 g/ml in the solvent are measured at 25 °C, the same viscometer being used for both measurements. The viscosity number is calculated from these measurements and from the known concentration of the solution.

5 Reagents and materials

Use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

WARNING — Avoid contact with the skin and inhalation of any vapours of the solvents and cleaning liquids.

5.1 Solvents

5.1.1 Sulfuric acid, 96 % (*m/m*) ± 0,15 % (*m/m*) solution.

For the determination of the concentration of commercial sulfuric acid (95 % to 97 %) and adjustment to 96,0 %, see annex A.

5.1.2 Formic acid, 90 % (*m/m*) ± 0,15 % (*m/m*) solution.

The solvent shall be stored in a brown glass bottle. Its concentration shall be checked at least every 2 weeks. It shall not contain more than 0,2 % acetic acid or methyl formate.

5.1.3 *m*-Cresol, meeting the following specifications:

appearance: clear and colourless

m-cresol content: 99 % (*m/m*) min.

o-cresol content: 0,3 % (*m/m*) max.

water content: 0,13 % (*m/m*) max.

m-Cresol of the required purity can be obtained by distillation of chemically pure *m*-cresol, preferably *in vacuo*.

To avoid oxidation, nitrogen shall preferably be used for pressure compensation. Its purity may be checked by gas chromatography. The solvent shall be stored in a brown glass bottle.

5.2 Cleaning liquids

5.2.1 Chromic acid solution, prepared by mixing equal volumes of sulfuric acid (ϱ = 1,84 g/ml) and a saturated solution of potassium dichromate. If required, the chromic acid solution may be replaced by other, equally effective cleaning liquids.

5.2.2 Acetone, freshly distilled.

6 Apparatus

6.1 Vacuum drying cabinet, pressure less than 100 kPa.

6.2 Balance, accurate to 0,1 mg.

6.3 Volumetric flask, capacity 50 ml, complying with the requirements of ISO 1042, fitted with a ground-glass stopper.

6.4 Shaking apparatus or **magnetic stirrer**.

6.5 Sintered-glass filter, with a pore size between 40 µm and 100 µm (grade P 100), or **stainless-steel sieve**, with apertures of about 0,075 mm^2.

6.6 Viscometer, of the suspended-level Ubbelohde type, complying with the requirements of ISO 3105. The essential dimensions of the viscometer are shown in figure 1. For use with the formic acid solution (5.1.2), the inside diameter of the capillary shall be 0,58 mm ± 2 % (complying with the requirements of size No. 1 of ISO 3105). For use with the sulfuric acid solution (5.1.1) or *m*-cresol (5.1.3), the inside diameter of the capillary shall be 1,03 mm ± 2 %

(complying with the requirements of size No. 2 of ISO 3105).

Other types of viscometer listed in ISO 3105 may be used, provided that the results are equivalent to those of the Ubbelohde viscometers specified above. However, in cases of dispute, an Ubbelohde viscometer shall be used.

6.7 Thermostatic bath, capable of being maintained at 25 °C ± 0,05 °C.

6.8 Stop-watch, accurate to 0,1 s.

6.9 Centrifuge.

7 Preparation of test samples

7.1 General

Polyamide test samples for the determination of the viscosity number shall be completely soluble in the solvents mentioned, and the additives contained in them (reinforcement fibres, flame-retardants and modifiers) shall not interfere with the viscosity measurement, or it shall be possible to separate the additives quantitatively from the solution as with glass and carbon fibres, for instance.

If the content of these additives is greater than 2 % (*m/m*), it shall be determined quantitatively to allow the exact test portion size to be calculated (see table 1).

Exception: an extracted sample if it contains extractable additives (see 7.2).

7.2 Samples with extractable ingredients

The ground polymer remaining after extraction in accordance with ISO 6427 or ISO 599 shall be dried by heating for 3 h at 100 °C under vacuum and used as the test sample.

7.3 Samples with auxiliaries (non-hydrolyzable and without ash) that are insoluble in hydrochloric acid, e.g. polymer modifiers, carbon fibres and certain flame-retardants

Preliminary tests shall be carried out to determine whether the sample is completely soluble in the solvent to be used, i.e. that it does not form particles of gel.

The fraction of polymer modifier and other additives that are insoluble in hydrochloric acid is determined from the hydrolysis residue (this method will form the subject of a future International Standard) and the correct amount of polyamide sample to be weighed out is calculated using equation 8.2.

NOTE 1 Experience indicates that the usual polymer modifiers for polyamides, e.g. ethylene copolymers and EP rubber, are so finely dispersed in the solution of the sample that the solution viscosity is practically unaffected, provided that the solution is free from gel particles.

If carbon fibres that can be detected in the hydrolysis residue under an optical microscope are present, they shall be filtered off from the solution of the polyamide sample by means of a filter crucible with a glass frit.

7.4 Samples with auxiliaries that are soluble in dilute hydrochloric acid and/or cannot be determined from the ash owing to oxide formation, e.g. carbonates, metal powders and certain pigments

The residue on dissolution in 90 % formic acid is determined as follows:

Shake or stir the polyamide sample for several hours at room temperature in about 10 times its own volume of 90 % formic acid using the procedure described in 10.2.

Separate off the solid residue by filtration.

Carefully wash the residue with 90 % formic acid and then with acetone. Subsequently dry for 3 h at 100 °C under a vacuum. Weigh.

7.5 Samples with auxiliaries with determinable ash, e.g. glass fibres and silicates

The ash is determined by the method specified in ISO 3451-4, and the amount of polyamide sample to be weighed out is calculated using equation 8.2. When necessary, filter the solution of the sample through a filter crucible with a glass frit before making the measurement.

NOTE 2 Any glass fibres of the usual dimensions contained in the sample will sediment completely after 3 h to 4 h. In such cases, the test solution can be decanted for the measurement and thus does not need to be filtered.