SLOVENSKI STANDARD
SIST ISO 1628-5:2000

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Polimerni materiali - Določanje viskoznosti polimerov v razredčenih raztopinah s kapilarnimi viskozimetri - 5. del: Plastomerni poliestri (TP) homo- in kopolimerov

Plastics -- Determination of the viscosity of polymers in dilute solution using capillary viscometers -- Part 5: Thermoplastic polyester (TP) homopolymers and copolymers

Plastiques -- Détermination de la viscosité des polymères en solution diluée à l'aide de viscosimètres à capillaires -- Partie 5: Homopolymères et copolymères des polyesters thermoplastiques (TP)

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INTERNATIONAL STANDARD

ISO 1628-5

Second edition
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Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers —

Part 5:
Thermoplastic polyester (TP) homopolymers and copolymers

Plastiques — Détermination de la viscosité des polymères en solution diluée à l'aide de viscosimètres à capillaires —

Partie 5: Homopolymères et copolymères des polyesters thermoplastiques (TP)

Reference number
ISO 1628-5:1998(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 1628-5 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This second edition cancels and replaces the first edition (ISO 1628-5:1986), which has been technically revised.

ISO 1628 consists of the following parts, under the general title *Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers*:

— *Part 1: General principles*

— *Part 2: Poly(vinyl chloride) resins*

— *Part 3: Polyethylenes and polypropylenes*

— *Part 4: Polycarbonate (PC) moulding and extrusion materials*

— *Part 5: Thermoplastic polyester (TP) homopolymers and copolymers*

— *Part 6: Methyl methacrylate polymers*

Annex A of this part of ISO 1628 is for information only.

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Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers —

Part 5:

Thermoplastic polyester (TP) homopolymers and copolymers

1 Scope

This part of ISO 1628 specifies a method for the determination of the viscosity number (also referred to as "reduced viscosity") of dilute solutions of thermoplastic polyesters (TPs) in certain specified solvents. The method is applicable to poly(ethylene terephthalate) (PET), poly(butylene terephthalate) (PBT), poly- (cyclohexylenedimethylene terephthalate) (PCT), and poly(ethylene naphthalate) (PEN), as well as to copolyesters and other polyesters, defined in ISO 7792-1, that are soluble in one of the specified solvents under the specified conditions.

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

The determination of the viscosity number of a thermoplastic polyester 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 part of ISO 1628. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this part of ISO 1628 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 current valid International Standards.

ISO 1628-1:—[1)], *Plastics - Determination of the viscosity of polymers in dilute solution using capillary viscometers - Part 1: General principles.*

ISO 3105:1994, *Glass capillary kinematic viscometers - Specifications and operating instructions.*

ISO 3451-2:1984, *Plastics - Determination of ash - Part 2: Polyalkylene terephthalates.*

ISO 7792-1:1997, *Plastics - Thermoplastic polyester (TP) moulding and extrusion materials - Part 1: Designation system and basis for specifications.*

1) To be published. (Revision of ISO 1628-1:1984)

3 Principle

The times of flow of a solvent and a solution of resin in the solvent at a concentration of 0,005 g / ml are measured at 25 °C by conventional methods. The viscosity number is calculated from these measurements and from the known concentration of the solution. Specific gravity difference and kinetic energy corrections are small in this method and are not applied.

4 Solvents

WARNING - The solvents used are hazardous. Avoid contact with the skin and inhalation of the vapours.

4.1 Phenol/1,2-dichlorobenzene

Weigh out 1 part by mass of phenol (analytical grade) and dissolve in 1 part by mass of 1,2-dichlorobenzene (analytical grade). Work to an accuracy of 1 % or better in the weighings.

4.2 Phenol/1,1,2,2-tetrachloroethane

Weigh out 6 parts by mass of phenol (analytical grade) and dissolve in 4 parts by mass of 1,1,2,2-tetrachloroethane (analytical grade), or weigh out 1 part by mass of phenol and dissolve in 1 part by mass of 1,1,2,2-tetrachloroethane. Work to an accuracy of 1 % or better in the weighings.

4.3 *o*-Chlorophenol, analytical grade.

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

appearance: clear and colorless;
m-cresol content: 99 % (*m* / *m*) min.;
o-cresol content: 0,3 % (*m* / *m*) max.;
water content: 0,1 % (*m* / *m*) max.

NOTE - A solvent of the required purity can be obtained by distillation of chemically pure *m*-cresol, preferably in vacuo, pressure compensation being effected with nitrogen in order to avoid oxidation. The purity of the solvent may be checked by gas chromatography.

4.5 Dichloroacetic acid, analytical grade.

4.6 Phenol/2,4,6-trichlorophenol

Weight out 6 parts by mass of phenol (analytical grade) and dissolve in 4 parts by mass of 2,4,6-trichlorophenol (analytical grade).

NOTE - 2,4,6-trichlorophenol of the required purity can be obtained by distillation of chemically pure 2,4,6-trichlorophenol.

4.7 Storage of the solvents

Protect the solvents from light by storing them, for example, in stoppered brown-glass bottles.

5 Apparatus

5.1 Mill, to reduce the sample to a grain size of about 0,5 mm.

5.2 Volumetric flask, of capacity 100 ml, fitted with a ground-glass stopper.

5.3 Conical flask, of capacity 150 ml, fitted with a ground-glass stopper.

5.4 Burette, graduated in divisions of 0,1 ml.

5.5 Steam bath, or other means to heat the contents of a flask to 90 to 100 °C.

5.6 Oil bath, capable of being controlled at 135 to 140 °C, or other means to heat the contents of a flask to that temperature range.

5.7 Magnetic stirrer, or other equipment to continuously agitate the contents of a stoppered flask.

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

5.9 Viscometer, suspended-level Ubbelohde type, size No. 1B, 1C or 2, in accordance with 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, Ubbelohde viscometers shall be used.

5.10 Stainless-steel sieve, with a nominal aperture size of 63 to 90 µm, or **sintered-glass filter funnel** with 40 to 100 µm pore diameter.

5.11 Analytical balance, accurate to 0,1 mg.

5.12 Stopwatch, accurate to 0,1 s.

5.13 Vacuum dryer, capable of being maintained at about 130 °C under vacuum.

6 Solvent and solution

6.1 Selection of the solvent

6.1.1 The value of the viscosity number of a saturated polyester depends on the solvent used. Six different solvents are described in this part of ISO 1628: phenol / 1,2-dichlorobenzene (4.1), phenol / 1,1,2,2-tetrachloroethane (4.2), *o*-chlorophenol (4.3), *m*-cresol (4.4), dichloroacetic acid (4.5) and phenol / 2,4,6-trichlorophenol (4.6).

The flow times of the solvents shall be determined at least once each day that they are used (see 7.2). If the flow time of a solvent differs by more than 1 % from the initial value at the time of preparation, the solvents shall be discarded and fresh solvent prepared.

The solvent or solvents to be used for a particular saturated polyester are specified below.

6.1.2 For PET, use phenol / 1,2-dichlorobenzene (50 / 50), phenol / 1,1,2,2-tetrachloroethane (50 / 50 or 60 / 40), *o*-chlorophenol or dichloroacetic acid as solvent.

NOTE - Equations for the interconversion of viscosity number determined in these four solvents are presented in annex A, subclause A.3.1.

6.1.3 For PBT and corresponding copolyesters, use phenol / 1,1,2,2-tetrachloroethane (50 / 50 or 60 / 40), *o*-chlorophenol, *m*-cresol, phenol / 1,2-dichlorobenzene (50 / 50) or dichloroacetic acid as solvent.

NOTE - Equations for the interconversion of viscosity number determined in these four solvents are presented in annex A, subclause A.3.2.

6.1.4 For PCT, use phenol / 1,1,2,2-tetrachloroethane (60 / 40) as solvent.

6.1.5 For amorphous PEN, use phenol / 1,1,2,2-tetrachloroethane (60 / 40) as solvent and for crystalline PEN use phenol / 2,4,6-trichlorophenol (60 / 40).

6.1.6 For other TP homopolymers and copolymers, *m*-cresol is the recommended solvent.

6.2 Determination of inorganic materials or other additives in the sample

Contents of inorganic materials or other additives exceeding 0,5 % (*m* / *m*) each shall be taken into account in the preparation of the test solution (6.4).

6.2.1 Determination of the content of inorganic materials

If the sample contains inorganic materials, such as fillers or glass fibres, determine the content in accordance with ISO 3451-2.

6.2.2 Determination of the content of other additives

If the sample contains other additives, such as polyalkenes or flame retardants, determine the content in an appropriate way. Report the procedure(s) used.

NOTE - When a polyester is a compound consisting of many components (flame retardants, fillers, antioxidants, impact modifiers), the analysis of the polyester content is more specific. This can be achieved by hydrolysis of the polyester and subsequent analysis of the monomer(s).

6.3 Sample

The sample shall be representative of the material to be tested. Predry the sample at 120 °C under vacuum for 3 h to avoid a decrease in VN by hydrolysis with residual water.

6.4 Preparation of solution

Use one of the following procedures:

6.4.1 Procedure A

Weigh out, to the nearest 0,2 mg, a test portion m_1 in the range

$$\frac{0,5}{1-[(i+o)/100]} \pm 0,01 \quad \text{grams}$$

where

i is the content of inorganic materials, expressed as a percentage by mass, in the sample, determined in accordance with 6.2.1

o is the content of other additives, expressed as a percentage by mass, in the sample, determined in accordance with 6.2.2.

The corrections for *i* and *o* need only be applied if they exceed 0,5 % (*m* / *m*) each.

Transfer the test portion to the volumetric flask (5.2), add 60 ml of solvent, stopper the flask and warm on the steam bath (5.5), with occasional stirring, until the polymer has dissolved completely. On the steam bath, no degradation of the polymer occurs. If, however, long waiting times are undesirable for other reasons, the dissolution may be accelerated by agitating the flask continuously, for example with the magnetic stirrer (5.7).

Samples of highly crystalline poly(ethylene terephthalate), such as postcondensed moulding chips, which may have crystallinities of around 65 %, will not dissolve on the steam bath, not even with continuous stirring. To dissolve such samples, grind the material in the mill (5.1) and dissolve at a temperature of 135 to 140 °C (5.6) with continuous stirring. At 135 to 140 °C, some degradation of the polymer occurs. Therefore, avoid heating times over 30 min.

After dissolution, cool the flask and its contents to 25 ± 2 °C, make up to 100 ml with solvent held at this temperature, and mix well. If, in dissolving, a magnetic stirrer has been used, first remove it from the solution and rinse it with the make-up solvent, ensuring that all the washings enter the flask. The concentration of the polymer in the solution, expressed in grams per millilitre, used in calculating the viscosity number (clause 8) is given by the formula

$$0,01\ (1-\frac{i+o}{100})\, m_1$$

NOTE - Usually, the concentration will differ slightly from 0,005 g / ml. The effect on the viscosity number can be neglected, however, since, over the range considered, the viscosity relative increment may be considered to be a linear function of the concentration.

6.4.2 Procedure B

Weigh out, to the nearest 0,2 mg, a test portion m_2 in the range 0,4 to 0,6 g.

Transfer the test portion to the conical flask (5.3). Add by burette (5.4), to the nearest 0,1 ml, a volume of solvent, in millilitres, equal to

$$200\ (1-\frac{i+o}{100})\, m_2$$

where *i* and *o* have the same meaning as in 6.4.1.