
**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)*

ISO 1628-5:1998

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Foreword

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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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International Organization for Standardization
Case postale 56 • CH-1211 Genève 20 • Switzerland
Internet central@iso.ch
X.400 c=ch; a=400net; p=iso; o=isocs; s=central

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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 $25 \pm 0,05$ °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