INTERNATIONAL STANDARD

ISO 1628-4

Second edition
1999-03-01

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

Part 4:
Polycarbonate (PC) moulding and extrusion materials

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

Partie 4: Matériaux polycarbonates (PC) pour moulage et extrusion

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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 1628-4 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 9, *Thermoplastic materials*.

This second edition cancels and replaces the first edition (ISO 1628-4:1986), 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 conditions*

— *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 forms an integral part of this part of ISO 1628.

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

Part 4:
Polycarbonate (PC) moulding and extrusion materials

1 Scope

This part of ISO 1628 describes the conditions necessary for the determination of the viscosity number (also known as the reduced viscosity) and the relative viscosity of polycarbonates in dilute solution.

It can be used for pure polycarbonates and blends with other polymers, as well as mixtures of both, with or without fillers, as defined in ISO 7391-1.

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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 currently valid International Standards.

ISO 1628-1:1998, *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 4793:1980, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

ISO 7391-1:1996, *Plastics — Polycarbonate (PC) moulding and extrusion materials — Part 1: Designation system and basis for specifications.*

ISO 7391-2:1996, *Plastics — Polycarbonate (PC) moulding and extrusion materials — Part 2: Preparation of test specimens and determination of properties.*

3 Definitions and units

The viscosity number VN is defined as

$$\mathrm{VN} = \frac{\eta - \eta_0}{\eta_0 c}$$

$$= \frac{v\rho - v_0 \rho_0}{v_0 \rho_0 c}$$

$$= \frac{v \dfrac{\rho}{\rho_0} - v_0}{v_0 c}$$

$$= \frac{v - v_0}{v_0 c}$$

where

η is the dynamic viscosity of the solution, in Pa·s;

η_0 is the dynamic viscosity of the solvent, in Pa·s;

ρ is the density of the solution, in kg·m^{-3};

ρ_0 is the density of the solvent, in kg·m^{-3};

$v = \dfrac{\eta}{\rho}$ is the kinematic viscosity of the solution, in m^2·s^{-1};

$v_0 = \dfrac{\eta_0}{\rho_0}$ is the kinematic viscosity of the solvent, in m^2·s^{-1};

c is the concentration of the solution, in g·ml^{-1}.

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The units of VN are ml·g^{-1}.

Due to the fact that there is only a slight difference between the density of the solution ρ_0 and that of the solvent ρ, η can be replaced by v in the formula for calculating the reduced viscosity.

The relative viscosity of the solution η_{rel} is defined as

$$\eta_{\mathrm{rel}} = \frac{v}{v_0}$$

where

$v = \dfrac{\eta}{\rho}$ is the kinematic viscosity of the solution, in m^2·s^{-1};

$v_0 = \dfrac{\eta_0}{\rho_0}$ is the kinematic viscosity of the solvent, in m^2·s^{-1}.

The relative viscosity is a dimensionless quantity.

4 Principle (see also ISO 1628-1:1998, clause 4)

The kinematic viscosity ν is calculated from the following equation:

$$\nu = \frac{\eta}{\rho} = k(t - \Delta t)$$

where

k is the viscometer constant, in $ml^2 \cdot s^{-2}$;

t is the efflux time, in s;

Δt is the kinetic-energy correction, in s;

ρ is the density of the solution, in $kg \cdot m^{-3}$.

NOTE Contrary to the instructions in ISO 1628-1:1998, clause 4, the kinetic-energy correction can only be ignored if it is not more than 0,2 % of the efflux time t.

5 Apparatus (see also ISO 1628-1:1998, clause 5)

5.1 Viscometer:

a) Ubbelohde capillary viscometer, capillary size number 0C, capillary diameter 0,36 mm, receiver flask 2 ml, as specified in ISO 3105.

b) Other viscometers listed in ISO 3105, provided that the same values are obtained as with the viscometer specified above.

c) When using automatic viscometers with suitable automatic timing devices (see below), identical results are obtained even if capillaries with a larger diameter (e.g. 0,58 mm) are used (cf. ISO 1628-1, table 1), which means that other capillaries can be used in conjunction with this type of apparatus.

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In cases of doubt, a viscometer conforming to the requirements given in a) shall be used.

The viscometer shall be calibrated by the method described in annex A.

5.2 Timing device, capable of being read to the nearest 0,1 s and accurate to within ± 0,1 % over a 15-minute period, except when using automatic viscometers with larger-diameter capillaries [see 5.1, item c)] when the timing device shall be capable of being read to the nearest 0,01 s and be accurate to within ± 0,1 % over a 15-minute period.

5.3 Thermostatic bath, operated at 25 °C.

Temperature fluctuations may not exceed ± 0,1 °C.

5.4 Volumetric flasks, volume 100 ml at the temperature of calibration, fitted with a ground-glass or plastic stopper giving an airtight seal.

5.5 Analytical balance, accurate to 0,1 mg.

5.6 Drying oven, operated at 110 °C.

5.7 Petri dishes.

5.8 Sintered-glass filter crucible, porosity class P1,6 (see ISO 4793).

5.9 Sintered-glass filter crucible, porosity class P4 (see ISO 4793).

5.10 Filter aid, e.g. kieselguhr or diatomaceous earth.

5.11 Laboratory shaker.

5.12 Laboratory centrifuge.

6 Solvent and preparation of test solution (see also ISO 1628-1:1998, clause 6)

6.1 Solvent

Dichloromethane, of recognized analytical purity, or equivalent.

6.2 Sampling

Carry out sampling in such a way that the sample taken is representative of the whole material.

6.3 Concentration of solution

The concentration of polycarbonate in the solution shall be 5 g/l.

6.4 Preparation of the test solution

6.4.1 Non-reinforced samples containing little or no pigment/additive

Using the analytical balance (5.5), weigh, to the nearest 0,1 mg, 500 mg of the test material into a 100 ml volumetric flask (5.4). Add approximately 70 ml of dichloromethane (6.1) and place on the shaker (5.11) until completely dissolved. Make up to the mark with dichloromethane at the calibration temperature, and shake once more to homogenize the solution.

When testing materials containing small amounts of pigments and/or additives (see below), increase the mass of the sample in proportion to the amount of pigment or additive present so that the resulting concentration of pure polycarbonate is 5 g/l.

NOTE 1 This correction is only necessary if the pigment and/or additive content is more than 1 %. Soluble dyes, pigments and/or additives at concentrations of less than 1 % will not affect the result of the determination.

NOTE 2 Higher concentrations of pigments/additives or very intense dyes (preventing optical measurements using photo-electric cells) affect the measurement so much that these components must be removed from the test solution with the help of a filter aid (5.10) or centrifuge (5.12). The instructions given in 6.4.2 are applicable to the treatment of the sample in such cases.

NOTE 3 When using an automatic viscometer with computerized control equipment [5.1 c)], the amount of polymer weighed out can differ from the set value by up to 10 %, as long as the polymer concentration, which will also differ from the set value, is taken into account when calculating the result [see A.3 c)].

6.4.2 Glass-fibre-reinforced samples and/or samples with high pigment/additive contents

Weigh approximately 5 g of the material under test into a 100 ml volumetric flask (5.4). Add approximately 70 ml of dichloromethane and place on the shaker until completely dissolved. The sample can be crushed mechanically before this step to increase the speed of dissolution.

Allow the insoluble components (glass fibres, pigments, etc.) to settle out and filter the solution through a P4 filter crucible (5.9) into a Petri dish (5.7). Place the Petri dish in the drying oven (5.6) operated at 110 °C to evaporate off the dichloromethane. Leave the film which remains in the drying oven until it reaches constant mass (1 h to 10 h, depending on the thickness of the film). Prepare a solution from the dried film using the procedure described in 6.4.1.

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