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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

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

Reference number
ISO 1628-4:1999(E)

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 1628-4 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.

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}.

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·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·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.

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.

7 Measurement temperature

The measurement temperature shall be 25 °C ± 0,1 °C.

8 Procedure (see also clause 3 and ISO 1628-1:1998, clause 8)

Pour pure solvent into the viscometer (5.1) through a P1,6 filter crucible (5.8).

Determine the flow time of the solvent three to five times at 25 °C ± 0,1 °C. The individual measurements shall not differ from their mean value by more than 0,2 %.

Repeat the procedure using the sample solution.

9 Calculation of results (see also ISO 1628-1:1998, clause 9)

Calculate the viscosity number, expressed in ml/g, using

a) the method described in A.3;

or

b) the following equation:

$$\mathrm{VN} = \frac{1}{c}\left(\frac{\nu}{\nu_0} - 1\right)$$

$$= \frac{1}{c}\left(\frac{t - \Delta t}{t_0 - \Delta t_0} - 1\right)$$

where

ν is the kinematic viscosity of the solution, in $\mathrm{m^2 \cdot s^{-1}}$;

ν_0 is the kinematic viscosity of the solvent, in $\mathrm{m^2 \cdot s^{-1}}$;

t is the arithmetic mean of the flow times for the solution, in s;

t_0 is the arithmetic mean of the flow times for the solvent, in s;

c is the concentration of the solution, in $\mathrm{g \cdot ml^{-1}}$;

Δt is the kinetic-energy correction for t, provided by the manufacturer of the capillary, in s;

Δt_0 is the kinetic-energy correction for t_0, provided by the manufacturer of the capillary, in s.

NOTE This equation conforms to the relationship defined in ISO 1628-1, but additionally takes the kinetic-energy correction into account. This correction is necessary in order to obtain accurate results with the capillaries specified.

Equation (12) given in ISO 1628-1:1998, which, using the symbol VN instead of I, reads

$$\mathrm{VN} = \frac{t - t_0}{t_0 c}$$

can be applied with sufficient accuracy if a viscometer with a comparatively thin capillary is used since the kinetic-energy correction term constitutes less than 0,2 % of the efflux time.

Calculate the relative viscosity of the solution η_{rel} (dimensionless) as follows:

$$\eta_{\text{rel}} = \frac{\nu}{\nu_0}$$

$$= \frac{t - \Delta t}{t_0 - \Delta t_0}$$

where

ν is the kinematic viscosity of the solution, in $m^2 \cdot s^{-1}$;

ν_0 is the kinematic viscosity of the solvent, in $m^2 \cdot s^{-1}$;

t is the arithmetic mean of the flow times for the solution, in s;

t_0 is the arithmetic mean of the flow times for the solvent, in s;

Δt is the kinetic-energy correction for t, provided by the manufacturer of the capillary, in s;

Δt_0 is the kinetic-energy correction for t_0, provided by the manufacturer of the capillary, in s.

Clauses A.1 and A.2 describe how to determine the correction terms Δt and Δt_0.

10 Test report

This document (test certificate) is described in ISO 1628-1:1998, clause 10.

Annex A

(normative)

Instrument-calibration procedures

A.1 Checking the accuracy of the viscometer

Experience has shown that the high level of accuracy necessary when determining the viscosity number of polycarbonate solutions cannot always be reached by commercially available Ubbelohde viscometers. The reason for this lies in small irregularities in the capillaries. It is therefore necessary to check the accuracy of the capillaries used with the aid of suitable reference materials. There are two ways of doing this:

a) Use of calibration liquids of known kinematic viscosity

The following materials are recommended[1]:

0	Dichloromethane (analytical grade)	$\nu_{0,\ 25{,}00\ °\mathrm{C}} = 0{,}314\,2\ \mathrm{mm^2 \cdot s^{-1}}$ $\rho_{0,\ 25{,}00\ °\mathrm{C}} = 1{,}316\,3\ \mathrm{g \cdot ml^{-1}}$
1	Trichloroethylene (analytical grade)	$\nu_{0,\ 25{,}00\ °\mathrm{C}} = 0{,}369\,3\ \mathrm{mm^2 \cdot s^{-1}}$ $\rho_{0,\ 25{,}00\ °\mathrm{C}} = 1{,}455\,5\ \mathrm{g \cdot ml^{-1}}$
2	Tetrachloroethylene (analytical grade)	$\nu_{0,\ 25{,}00\ °\mathrm{C}} = 0{,}525\,7\ \mathrm{mm^2 \cdot s^{-1}}$ $\rho_{0,\ 25{,}00\ °\mathrm{C}} = 1{,}614\,4\ \mathrm{g \cdot ml^{-1}}$

To conduct the check, determine the efflux time of the three liquids, free from the influence of the time-correction term, carrying out three to five individual measurements under standard conditions (25 °C ± 0,1 °C). The required repeatability of the individual measurements specified in clause 8 is also applicable in this case.

The relative viscosities are calculated in accordance with the equations given in clause 9, as follows:

$$\frac{\nu_1}{\nu_0} = \frac{t_1 - \Delta t_1}{t_0 - \Delta t_0}$$

or

$$\frac{\nu_2}{\nu_0} = \frac{t_2 - \Delta t_2}{t_0 - \Delta t_0}$$

The values obtained experimentally are compared with the true values:

$$\frac{\nu_1}{\nu_0} = 1{,}175$$

or

$$\frac{\nu_2}{\nu_0} = 1{,}673$$

The deviation shall be less than 0,4 % in all cases for the measurements to be considered reliable.

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[1] Bauer, H., and Meerlender, G., Newtonian reference materials with $n < 1\ \mathrm{mm^2 \cdot s^{-1}}$ and their application in extending the range of capillary viscometers, *Rheol. Acta* **21** (1982), pp. 499-501.

b) **Use of certified polycarbonate materials at 5 $g \cdot l^{-1}$**

The method described in a) requires the use of reference materials which are structurally different from polycarbonates. Moreover, as the purity of these materials is often not known exactly at the time of use (due to unsealed bottles, etc.), the use of polycarbonate standards with a known solution viscosity is recommended as an alternative method. It is possible to have various polycarbonate samples tested at standards institutes and then use these samples as standards (with a test certificate from the institute concerned). In this context, the standards shall be carefully chosen so that the entire measurement range is covered by two to four standards. The comparison shall be conducted as per the instructions for normal measurements (see clauses 6 to 8). The results shall conform to those of the standards institute to within ± 0,4 %. The polymer concentration shall be 5 $g \cdot l^{-1}$ in such cases.

c) **Use of certified polycarbonate materials at concentrations other than 5 $g \cdot l^{-1}$**

The method described in b) requires the use of polymer concentrations at the standard concentration of 5 $g \cdot l^{-1}$. In practice, it can be difficult to weigh out 5 $g \cdot l^{-1}$ exactly. Therefore, a compensatory process can also be used, in which the standards [two to four, as in b)] are measured at several concentrations (e.g. five different concentrations between 4 $g \cdot l^{-1}$ and 6 $g \cdot l^{-1}$). In addition, the value for the pure solvent is taken as a blank. The viscosity of the solution at a known concentration, which lies within the range covered by the standard values but differs from these values, is interpolated from the resulting plot [see A.3 c) for procedure].

A.2 Determination of the effective viscometer constant k_{eff} and the effective kinetic-energy correction Δt_{eff}

If there is a discrepancy of more than 0,4 % between the expected and recorded viscosities of the reference standards, the effective viscometer constant k_{eff}, and thus the kinetic-energy correction Δt_{eff}, can be determined in the following way:

$$k_0 = \frac{v_0}{t_0 - \Delta t_0}$$

$$k_1 = \frac{v_1}{t_1 - \Delta t_1}$$

$$k_2 = \frac{v_2}{t_2 - \Delta t_2}$$

$$k_{\text{eff}} = \frac{k_1 + k_2 + k_3}{3}$$

where

v_i is the kinematic viscosity of the calibration liquids [i = 0, 1 and 2; see A.1 a)];

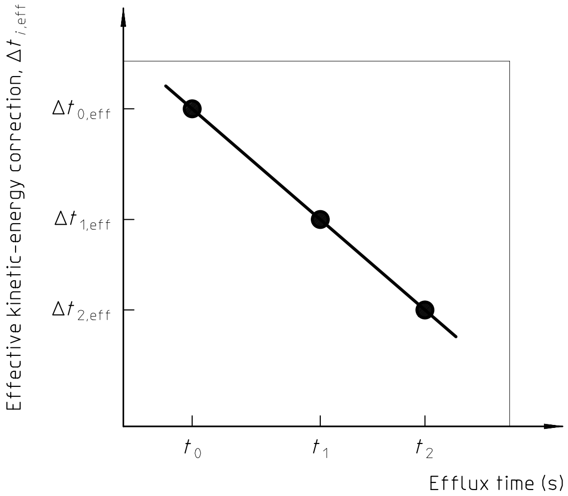
t_i is the corresponding efflux time;

Δt_i is the kinetic-energy correction term for t_i, provided by the manufacturer of the capillary.

The following calculation is carried out:

$$\Delta t_{i,\text{eff}} = t_i - \frac{v_i}{k_{\text{eff}}}$$

and the values of Δt_i corresponding to the individual t_i values are obtained either by plotting a graph or by linear-regression analysis (see figure A.1).



NOTE For meaning of subscripts, see clause A.2.

Figure A.1 — Determination of the effective kinetic-energy correction

A.3 Simplified calibration method

The correlation between the efflux time (primary measured variable) and viscosity number (target variable) can also be obtained directly for viscometers not described in clause A.1:

a) Use of calibration liquids of known kinematic viscosity

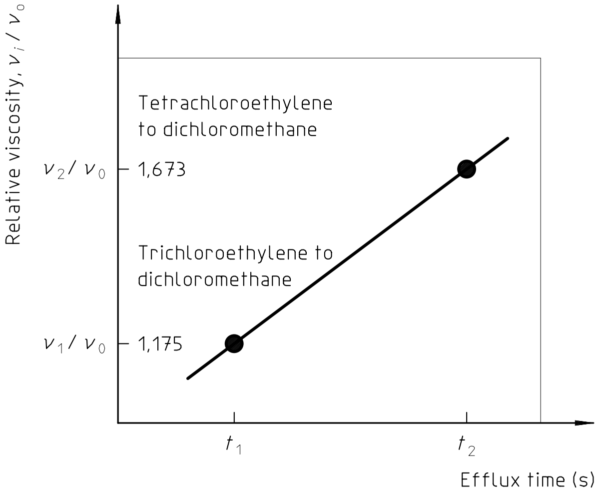
The use of calibration liquids of known kinematic viscosity permits a direct correlation to be made between efflux time and reduced viscosity. No knowledge of the viscometer constant or the kinetic-energy correction is necessary.

The relative viscosities

$$\frac{v_1}{v_0} \text{ and } \frac{v_2}{v_0}$$

of the calibration liquids are plotted against the respective efflux times for the viscometer concerned (see figure A.2). The relative viscosity of a polycarbonate solution of concentration c can be obtained by graphical or numerical interpolation or by linear regression. The viscosity number is calculated as described in clause 9:

$$\mathrm{VN} = \frac{1}{c}\left(\frac{v}{v_0} - 1\right)$$



NOTE For meaning of subscripts, see clause A.2.

Figure A.2 — Determination of the relative viscosity using standards

b) Use of certified polycarbonate materials at 5 g·l^{-1}

This procedure differs from that described in a) only in that the solutions used are polycarbonate solutions at the standard concentration of 5 g·l^{-1}. The polycarbonate standards used shall cover the entire measurement range with two to four interpolation nodes. The relative viscosities of the standards employed shall be known (source: e.g. certified standards institutes).

c) Use of certified polycarbonate materials at concentrations other than 5 g·l^{-1}

The use of several polycarbonate standards of known relative viscosities permits a procedure similar to that described in a) to be followed. The efflux time of each standard (two to four standards are used, covering the whole of the measurement range), is measured at several different concentrations (e.g. five concentrations between 4 g·l^{-1} and 6 g·l^{-1}). In addition, the value for the pure solvent is taken as a blank.

The two-dimensional function

$$\mathrm{VN} = f\left(\frac{t}{t_0}, c\right)$$

describes a plane whose position in space is defined by the ratio $\frac{t}{t_0}$ and the concentration c. The efflux time t and the viscosity number VN are calculated from this two-dimensional function within the defined limits (i.e. within the range of the concentrations and standard viscosities employed).

A correlation suitable for practical use is given by the following equation:

$$\frac{\mathrm{VN}}{c} = m \times \frac{t}{t_0} + b$$

in which m and b are empirical constants determined using the efflux times obtained from measurement of the standard solutions of known viscosity and known concentration.

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