SLOVENSKI STANDARD
SIST ISO 1628-4:1996

01-junij-1996

Polimerni materiali - Določanje viskoznostnega števila in mejnega viskoznostnega števila - 4. del: Polikarbonatni materiali (PC) za oblikovanje in ekstrudiranje

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 extruction

Ta slovenski standard je istoveten z: **ISO 1628-4:1986**

ICS:

83.080.10	Duromeri	Thermosetting materials
83.080.20	Plastomeri	Thermoplastic materials

SIST ISO 1628-4:1996 **en**

International Standard 1628/4

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION•МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ•ORGANISATION INTERNATIONALE DE NORMALISATION

Plastics — Determination of viscosity number and limiting viscosity number — Part 4: Polycarbonate (PC) moulding and extrusion materials

Plastiques — Détermination de l'indice de viscosité et de l'indice limite de viscosité — Partie 4: Matériaux à mouler et à extruder à base de polycarbonate (PC)

First edition — 1986-06-15

UDC 678.6'5 : 678.01 : 532.13 **Ref. No. ISO 1628/4-1986 (E)**

Descriptors : plastics, polymers, carbonates, moulding materials, extrusions, tests, determination, viscosity.

Price based on 5 pages

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.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 1628/4 was prepared by Technical Committee ISO/TC 61, *Plastics*.

Users should note that all International Standards undergo revision from time to time and that any reference made herein to any other International Standard implies its latest edition, unless otherwise stated.

Printed in Switzerland

Plastics — Determination of viscosity number and limiting viscosity number — Part 4: Polycarbonate (PC) moulding and extrusion materials

1 Scope and field of application

This part of ISO 1628 defines particular conditions for determination of the viscosity number of polycarbonate moulding and extrusion materials in dilute solution.

It applies to polycarbonate homopolymers and copolymers and mixtures of the two, with and without filler, as defined in ISO 7391/1.

2 References

ISO 1628/1, *Plastics — 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 3105, *Glass capillary kinematic viscosimeters — Specification and operating instructions.*

ISO 4793, *Laboratory sintered (fritted) filters — Porosity grading, classification and designation.*

ISO 7391, *Plastics — Polycarbonate (PC) moulding and extrusion materials*

— *Part 1: Designation.*

— *Part 2: Preparation of test specimens and determination of properties.*

3 Definitions and units (see ISO 1628/1, clause 3)

viscosity number, V.N.

$$\text{V.N.} = \frac{\eta - \eta_0}{\eta_0 \, c} = \frac{\nu \varrho - \nu_0 \varrho_0}{\nu_0 \varrho_0 \, c} \approx \frac{\nu - \nu_0}{\nu_0 \, c}$$

expressed in millilitres per gram,

where

η is the dynamic viscosity, in pascal seconds, of the solution;

η_0 is the dynamic viscosity, in pascal seconds, of the solvent;

ϱ is the density, in kilograms per cubic metre, of the solution;

ϱ_0 is the density, in kilograms per cubic metre, of the solvent;

$\nu = \frac{\eta}{\varrho}$ is the kinematic viscosity, in metres squared per second, of the solution;

$\nu_0 = \frac{\eta_0}{\varrho_0}$ is the kinematic viscosity, in metres squared per second, of the solvent;

c is the concentration, in grams per millilitre, of polycarbonate in the solution.

NOTE — Because of the small differences between the densities of the solution and solvent, η may be replaced by ν in the formula for V.N.

4 Measurements (see ISO 1628/1, clause 4)

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

where

k is a constant of the viscometer, in millimetres squared per second squared;

t is the efflux time, in seconds;

$\Delta t = \frac{A}{kt}$ is the kinetic energy correction time, in seconds;

ϱ is the density, in grams per cubic millimetre.

NOTE — Contrary to the instruction in ISO 1628/1, clause 4, the kinetic energy correction time Δt may be neglected only if it is less than 0,2 % of t.

5 Apparatus (see ISO 1628/1, clause 5)

5.1 Capillary viscometer, suspended-level Ubbelohde type OC, with capillary diameter of 0,36 mm and measuring bulb volume of 2 ml, according to ISO 3105.

Any other viscometer listed in ISO 3105 may be used, provided that it gives equivalent results to the specified viscometer, which shall be used for reference in case of dispute.

The calibration of the viscometer used shall be checked according to the method given in the annex, clause A.1.

5.2 Timing-device, which can be read to the nearest 0,1 s and has a rate constant to ± 0,1 % over 15 min.

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

5.4 One-mark volumetric flask, of capacity 100 ml, provided with a ground glass stopper.

5.5 Balance, accurate to 0,1 mg.

5.6 Drying cabinet, capable of being maintained at 110 °C.

5.7 Petri dishes.

5.8 Glass filter crucibles, grade P 1,6 (see ISO 4793).

5.9 Glass filter crucibles, grade P 4 (see ISO 4793).

5.10 Diatomaceous filter aid material.

5.11 Shaking machine.

5.12 Centrifuge.

6 Solvent and solution (see also ISO 1628/1, clause 6)

6.1 Solvent

Dichloromethane, redistilled, boilling range 39 to 41 °C, refractive index within the range of 1,423 to 1,425.

6.2 Sampling

The sample shall be representative of the material under test.

6.3 Solution concentration

The concentration shall be 5 g of resin per litre of solution.

6.4 Preparation of solution

6.4.1 PC material without fillers

Weigh, to the nearest 0,1 mg, 0,5 ± 0,01 g of the sample, into the one-mark volumetric flask (5.4). Add 70 ml of the dichloromethane (6.1) and dissolve the sample with agitation. Then dilute to the mark with dichloromethane at 20 ± 1 °C or weigh in as much solvent as is necessary to reach the concentration of 0,5 g/100 ml, and briefly agitate again.

For pigmented materials and/or materials containing special additives, the quantity weighed into the one-mark volumetric flask shall be increased so that the solution will contain 0,5 g PC per 100 ml (see the notes).

NOTES

1 This correction is necessary for a pigment or additive content greater than 0,5 % (*m*/*m*) [in most cases the pigment content is below 0,5 % (*m*/*m*)]. Dissolved dyestuffs and pigments under 0,5 % (*m*/*m*) will not interfere with the measurement.

2 To prevent contamination of the capillary in the case of solutions with higher pigment contents, it may be advisable to handle those materials as given under 6.4.2. To remove the additives, the diatomaceous filter aid material (5.10) or the centrifuge (5.12) may be used.

6.4.2 PC materials with fillers

Dissolve approximately 5 g of the sample in approximately 70 ml of the dichloromethane (6.1) in a round-bottomed flask with continuous stirring. To ensure complete dissolution, the material may be ground into fine particles.

When the filler, for example glass fibre, has settled, filter the solution through a grade P 4 glass filter crucible (5.9) into Petri dishes (5.7). Evaporate the dichloromethane under a fume hood and dry the resulting films in the drying cabinet (5.6) at 110 °C to constant mass (1 to 10 h, depending on film thickness). Prepare a solution of the dried film in accordance with 6.4.1.

7 Temperature of measurement

The temperature shall be 25 ± 0,05 °C.

8 Procedure (see clause 3 and ISO 1628/1, clause 8)

Introduce the dichloromethane solvent into the viscometer (5.1) using the glass filter crucible grade P 1,6 (5.8).

Determine the efflux times of the solvent at 25 ± 0,05 °C in three to five passes through the same viscometer. The range of efflux times shall not exceed 0,01 %.

Repeat the procedure with the test solution.

9 Expression of results (see ISO 1628/1, clause 9)

Calculate the viscosity number, expressed in millilitres per gram,

a) by the method given in the annex, clause A.3, or

b) by means of the following equation:

$$\text{V.N.} = \frac{1}{c}\left(\frac{\nu}{\nu_0} - 1\right)$$

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

where

ν is the kinematic viscosity of the solution, in metres squared per second;

ν_0 is the kinematic viscosity of the solvent, in metres squared per second;

t is the arithmetic mean of the readings obtained for the efflux time, in seconds, of the solution;

t_0 is the arithmetic mean of the readings obtained for the efflux time, in seconds, of the solvent;

c is the concentration, in grams per millilitre, of the solution;

Δt is the kinetic energy correction time for t as given by the manufacturer of the viscometer, in seconds;

Δt_0 is the kinetic energy correction time for t_0 as given by the manufacturer of the viscometer, in seconds.

NOTE — This equation is in accordance with ISO 1628/1, but introduces the kinetic energy correction time. This is necessary in order to obtain accurate results with the viscometer size specified.

Equation (10) for the viscosity number given in ISO 1628/1

$$\text{V.N.} = \frac{t - t_0}{t_0\, c}$$

can be used to give results of similar accuracy if a viscometer with a suitable smaller capillary diameter is used so that the kinetic energy correction time is less than 0,2 % of the efflux time.

To verify the kinetic energy correction times Δt and Δt_0, see the annex, clauses A.1 and A.2, respectively.

10 Test report

See ISO 1628/1, clause 10.

Annex

Calibration procedures

(This annex forms an integral part of the Standard.)

A.1 Verification of the accuracy of the viscometer

Experience has shown that the required high accuracy of viscosity number determination of PC solutions cannot always be achieved with commercially available Ubbelohde viscometers because of small irregularities of the capillary. It is therefore advisable to check the accuracy of measurement with the help of calibrating liquids of known kinematic viscosity.

The following liquids are recommended [1]:

0 — Dichloromethane (analytical purity) $\nu_{0,\ 25{,}00\ °C} = 0{,}314\ 2\ mm^2/s$; $\varrho_{0,\ 25{,}00\ °C} = 1{,}316\ 3\ g/ml$

1 — Trichloroethylene (analytical purity) $\nu_{1,\ 25{,}00\ °C} = 0{,}369\ 3\ mm^2/s$; $\varrho_{1,\ 25{,}00\ °C} = 1{,}455\ 5\ g/ml$

2 — Tetrachloroethylene (analytical purity) $\nu_{2,\ 25{,}00\ °C} = 0{,}525\ 7\ mm^2/s$; $\varrho_{2,\ 25{,}00\ °C} = 1{,}614\ 4\ g/ml$

Determine the kinetic energy corrected efflux time of these liquids at 25 ± 0,05 °C in three to five passes through the viscometer to be used. The range of efflux times shall not exceed 0,1 %.

Calculate the viscosity ratios according to clause 9 as

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

and

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

Comparing these values with the "true" values

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

and

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

the deviation should be less than 0,4 %.

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

If the deviation between the expected and the measured viscosity ratio of the calibrating liquids is more than 0,4 %, the effective capillary constant k_{eff} and the kinetic energy correction time $\Delta t_{i,eff}$ may be determined by the following method.

Calculate k_{eff} of the viscometer used as the arithmetic mean of three equations

$$k_0 = \frac{\nu_0}{t_0 - \Delta t_0}$$

$$k_1 = \frac{\nu_1}{t_1 - \Delta t_1}$$

$$k_2 = \frac{\nu_2}{t_2 - \Delta t_2}$$

$$k_{eff} = \frac{k_0 + k_1 + k_2}{3}$$

where

ν_i is the kinematic viscosity of the calibrating liquids (0, 1 or 2 respectively) mentioned in clause A.1;

t_i is the corresponding efflux time;

Δt_i is the kinetic energy correction time for t_i as given by the manufacturer of the viscometer.

Calculate

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

and plot the resulting $\Delta t_{i,eff}$ values against the corresponding efflux times, as in figure 1.

The appropriate Δt_i corresponding to a certain t_i can be determined by interpolation from this plot.

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