INTERNATIONAL STANDARD

ISO
1628-6

Plastics — Determination of viscosity number and limiting viscosity number —

Part 6:
Methyl methacrylate polymers

Plastiques — Détermination de l'indice de viscosité et de l'indice limite de viscosité —

Partie 6: Polymères de méthacrylate de méthyle

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 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 1628-6 was prepared by Technical Committee ISO/TC 61, *Plastics.*

It cancels and replaces International Standard ISO 1233 : 1975, of which it constitutes a technical revision.

ISO 1628 consists of the following parts, under the general title *Plastics — Determination of viscosity number and limiting viscosity number* [1]:

— *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: Poly(alkylene terephthalates)*

— *Part 6: Methyl methacrylate polymers*

Annexes A and B form an integral part of this part of ISO 1628. Annex C is for information only.

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[1] The general title of ISO 1628-1 is *Guidelines for the standardization of methods for the determination of viscosity number and limiting viscosity number of polymers in dilute solution.*

Plastics — Determination of viscosity number and limiting viscosity number —

Part 6:
Methyl methacrylate polymers

1 Scope

This part of ISO 1628 specifies the conditions to be used for the determination of the viscosity number of methyl methacrylate polymer moulding and extrusion materials in dilute solution.

It applies to methyl methacrylate (MMA) homopolymers and copolymers, and mixtures of the two with a minimum of 80 % (m/m) of MMA and up to 20 % (m/m) of other monomers, with and without additives, as defined in ISO 8257-1.

NOTE — The dilute-solution viscosity of polymers can be also expressed as a limiting viscosity number [η] (see annex B).

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 : 1984, *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 : 1976, *Glass capillary kinematic viscometers — Specification and operating instructions.*

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

ISO 8257-1 : 1987, *Plastics — Poly(methyl methacrylate) (PMMA) moulding and extrusion materials — Part 1: Designation.*

3 Definitions and units

For the purposes of this part of ISO 1628, the following definition applies, in addition to those in clause 3 of ISO 1628-1 : 1984.

viscosity number, V.N.:

$$\text{V.N.} = \frac{1}{c}\left(\frac{\eta - \eta_0}{\eta_0}\right) \qquad \ldots (1)$$

where

V.N. is the viscosity number, in millilitres per gram;

η is the dynamic viscosity of the test solution, in millipascal seconds;

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

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

NOTE — If the solvent and solution densities ϱ_0 and ϱ are substantially equal, η in equation (1) may be replaced by v and η_0 by v_0, where v and v_0 are the kinematic viscosities, in square millimetres per second, of the solution and the solvent, respectively (see clause 10).

4 Principle

Determination of the efflux time at 25 °C ± 0,05 °C of a solvent and of a polymer solution, having a specified concentration, in that solvent.

Calculation of the viscosity number and the limiting viscosity number from the above measurements and the known concentration of the solution.

5 Measurements

The data needed to calculate the function defined in clause 3 are obtained by means of a capillary tube viscometer, as explained in clause 4 of ISO 1628-1 : 1984.

The efflux time of a liquid is related to its viscosity by the Poiseuille-Hagenbach-Couette equation:

$$v = \frac{\eta}{\varrho} = k(t - \Delta t) = kt - \frac{A}{t} \qquad \ldots (2)$$

where

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

t is the efflux time, in seconds;

Δt is the kinetic energy correction (Hagenbach correction), in seconds;

ϱ is the density, in grams per cubic centimetre, of the liquid;

A is a kinetic energy correction parameter, as defined in ISO 1628-1.

NOTE — The kinetic energy correction Δt becomes insignificant if it is less than 0,2 % of t. For Ubbelohde type OC apparatus, Δt can be neglected if the efflux time t is greater than 270 s; for micro-Ubbelohde type MI apparatus, Δt can be neglected if t is greater than 70 s.

6 Apparatus (see also ISO 1628-1 : 1984, clause 5)

6.1 Capillary viscometer: suspended-level Ubbelohde type OC apparatus with a capillary diameter of 0,36 mm and measuring bulb of volume 2 ml, in accordance with ISO 3105, or **micro-Ubbelohde type MI viscometer**, with a capillary diameter of 0,40 mm and measuring bulb of volume 0,75 ml.

Any viscometer listed in ISO 3105 may be used, provided it gives results equivalent to those obtained with the specified viscometers, which shall be used in the event of a dispute.

The calibration of the viscometer used shall be checked by the method specified in annex A.

6.2 Timing device, with an accuracy of 0,1 s for the Ubbelohde type OC viscometer and 0,02 s for the type MI viscometer.

6.3 Thermostatic bath, capable of being maintained at a temperature of 25 °C ± 0,05 °C.

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

6.5 Balance, accurate to 0,1 mg.

6.6 Sintered-glass filter funnel, with a maximum pore diameter of 40 μm (porosity grade P 40 as defined in ISO 4793).

6.7 Shaking machine.

7 Solvent and test solution (see also ISO 1628-1 : 1984, clause 6)

7.1 Solvent

Chloroform, recognized analytical grade, stabilized with less than 1 % (V/V) of ethanol, kept in a brown bottle and stored in the dark.

7.2 Sample

It is important that the sample is representative of the material to be tested.

7.3 Test solution concentration

The solution concentration shall be 2,6 g of polymer per litre of solution.

7.4 Preparation of the test solution

Weigh, to the nearest 0,1 mg, 0,26 g ± 0,005 g of the sample into the volumetric flask (6.4). Add 50 ml of chloroform (7.1) and dissolve the sample completely with shaking. Then make up to the mark with chloroform at 20 °C ± 1 °C and shake once more briefly.

With polymers containing viscosity-increasing additives requiring modifications to the solution preparation process, such modifications shall be agreed upon by the interested parties.

For materials containing pigments and/or special additives, the quantity weighed into the volumetric flask shall be increased so that the test solution contains 0,26 g of MMA homopolymers and copolymers per 100 ml.

8 Temperature of measurement

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

9 Procedure (see also 1628-1 : 1984, clause 8)

Using the sintered-glass filter (6.6), introduce the chloroform solvent or the test solution directly into the tube of the viscometer.

Determine the efflux times of the solvent and the test solution at 25 °C ± 0,05 °C in three to five passages through the same viscometer. If the maximum difference between the efflux times exceeds 0,2 s for a Ubbelohde type OC viscometer or 0,03 s for a type MI viscometer, repeat the test with the same test solution after cleaning the viscometer with the solvent and drying it.

The above procedure may be suitably modified if a viscometer other than the Ubbelohde as described in ISO 3105 is used.

10 Expression of results (see also ISO 1628-1 : 1984, clause 9)

Calculate the viscosity number V.N., expressed in millilitres per gram, by means of the following equation (this equation is in accordance with ISO 1628-1, but includes the kinetic energy correction, which is necessary in order to obtain accurate results with the viscometer sizes specified):

$$V.N. = \frac{1}{c}\left(\frac{v}{v_0} - 1\right) = \frac{1}{c}\left(\frac{t - \Delta t}{t_0 - \Delta t_0} - 1\right) \qquad \dots (3)$$

where

v is the viscosity of the test solution;

v_0 is the viscosity of the solvent;

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

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

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

Δt is the kinetic energy correction for t (see annex A);

Δt_0 is the kinetic energy correction for t_0 (see annex A).

The equation for the viscosity number given in ISO 1628-1 :

$$V.N. = \frac{t - t_0}{t_0 c} \qquad \dots (4)$$

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

11 Test report

See ISO 1628-1 : 1984, clause 10.

3

Annex A

(normative)

Calibration of the viscometer

A.1 Experience has shown that the required high accuracy of the viscosity number determination of MMA homopolymer and copolymer solutions cannot always be achieved with commercially available Ubbelohde viscometers because of minor irregularities in the capillary. It is therefore advisable to check the accuracy of the viscometer by means of calibrating liquids whose kinematic viscosity [1] is known.

A.2 Viscometer constant k

Using n-undecane of recognized analytical grade at 25 °C, make three to five passages with the same viscometer and determine the mean efflux time t in accordance with ISO 3105 : 1976, clause 5. Calculate the viscometer constant k from the equation:

$$k = \frac{v}{t} \qquad \ldots \text{(A.1)}$$

where v for n-undecane at 25 °C is 1,467 5 mm²/s.

NOTE — The density ϱ of n-undecane at 25 °C is 0,736 5 g/ml.

A.3 Kinetic energy corrections Δt_0 and Δt

The energy correction can be neglected if it is less than 0,2 % of the efflux time.

The Ubbelohde type OC viscometer complies with this requirement if the efflux time is greater than 270 s (ASTM viscometer).

The micro-Ubbelohde type MI viscometer complies if the efflux time is greater than 70 s.

If the efflux time is less than specified above, a kinetic energy correction shall be made for the viscometer, using the following low-viscosity liquids as standards:

1. Dichloromethane (recognized analytical grade)
   $v_{25 °C} = 0,314\ 2$ mm²/s
   $\varrho_{25 °C} = 1,316\ 3$ g/ml

2. Trichloroethylene (recognized analytical grade)
   $v_{25 °C} = 0,369\ 3$ mm²/s
   $\varrho_{25 °C} = 1,455\ 5$ g/ml

3. Tetrachloroethylene (recognized analytical grade)
   $v_{25 °C} = 0,525\ 7$ mm²/s
   $\varrho_{25 °C} = 1,614\ 4$ g/ml

Measure the efflux time of each standard liquid in three to five passages and determine the kinetic energy correction Δt_{si} for each liquid using the equation

$$\Delta t_{si} = t_{si} - \frac{v_i}{k} \qquad \ldots \text{(A.2)}$$

where

t_{si} is the efflux time, in seconds, of the ith standard liquid;

v_i is the kinematic viscosity, in square millimetres per second, of the ith standard liquid;

k is the viscometer constant, in square millimetres per second squared.

Linear interpolation, using the following equations, between the two efflux times t_{s1} and t_{s2} for the standard liquids $i = 1$ and $i = 2$ (see figure A.1) gives the kinematic energy correction Δt_0 for the efflux time t_0 of the solvent (chloroform):

$$\Delta t_0 = \Delta t_{s1} - K_{12}\left(\frac{1}{t_{s1}} - \frac{1}{t_0} \right) \qquad \ldots \text{(A.3)}$$

$$K_{12} = \frac{\Delta t_{s1} - \Delta t_{s2}}{\dfrac{1}{t_{s1}} - \dfrac{1}{t_{s2}}} = \frac{\Delta t_{s1} - \Delta t_0}{\dfrac{1}{t_{s1}} - \dfrac{1}{t_0}} \qquad \ldots \text{(A.4)}$$

In the same manner, linear interpolation, using the following equations, between t_{s2} and t_{s3} gives Δt for the efflux time t of the test solution:

$$\Delta t = \Delta t_{s2} - K_{23}\left(\frac{1}{t_{s2}} - \frac{1}{t} \right) \qquad \ldots \text{(A.5)}$$

$$K_{23} = \frac{\Delta t_{s2} - \Delta t_{s3}}{\dfrac{1}{t_{s2}} - \dfrac{1}{t_{s3}}} = \frac{\Delta t_{s2} - \Delta t}{\dfrac{1}{t_{s2}} - \dfrac{1}{t}} \qquad \ldots \text{(A.6)}$$