INTERNATIONAL STANDARD

ISO 1628-1

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

Part 1:
General principles

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

Partie 1: Principes généraux

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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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. 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.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 1628-1 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

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This third edition cancels and replaces the second edition (ISO 1628-1:1998), of which it constitutes a minor revision intended primarily to correct an error in Subclause 9.1, paragraph 4 (starting: “The intrinsic viscosity shall be calculated from”), where, in line 2, “intrinsic-viscosity values” has been replaced by “inherent-viscosity” values.

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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*

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

Part 1:
General principles

1 Scope

This part of ISO 1628 defines the general conditions for the determination of the reduced viscosity, intrinsic viscosity and K-value of organic polymers in dilute solution. It defines the standard parameters that are applied to viscosity measurement, and can be used to develop standards for measuring the viscosities in solution of individual types of polymer. It can also be used to measure and report the viscosities of polymers in solution for which no separate standards exist.

2 Normative references

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The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3105:1994, *Glass capillary kinematic viscometers — Specifications and operating instructions*

ISO 3205, *Preferred test temperatures*

ISO 80000-1, *Quantities and units — Part 1: General*[1)]

ISO 80000-4, *Quantities and units — Part 4: Mechanics*

3 Definitions

3.1 Dimensions and units

The dimensions of properties defined in this part of ISO 1628 are expressed in terms of L for length, M for mass and T for time in accordance with ISO 80000-1, while the units appropriate to the properties are given in ISO 80000-1 and ISO 80000-4.

3.2 Definitions applicable to any liquid

3.2.1
viscosity
viscosity of a fluid sheared between two parallel plates, one of which moves relative to the other in uniform rectilinear motion in its own plane, defined by the Newton equation

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1) To be published. (Revision of ISO 31-0:1992)

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$$\tau = \eta \dot{\gamma} \qquad (1)$$

where

τ is the shear stress;

η is the viscosity;

$\dot{\gamma}$ is the velocity gradient or rate of shear, given by $\frac{\mathrm{d}v}{\mathrm{d}z}$ where v is the velocity of one plane relative to the other and z the coordinate perpendicular to the two planes

NOTE 1 The dimensions of viscosity are $\mathrm{ML}^{-1}\mathrm{T}^{-1}$.

NOTE 2 The units of viscosity are Pa·s.

NOTE 3 For practical use, the sub-multiple 10^{-3} Pa·s is more convenient.

NOTE 4 Viscosity is usually taken to mean “Newtonian viscosity”, in which case the ratio of shearing stress to velocity gradient is constant. In non-Newtonian behaviour, which is the usual case with high-polymer solutions, the ratio varies with the shear rate. Such ratios are often called “apparent viscosities” at the corresponding shear rate.

3.2.2
viscosity/density ratio
kinematic viscosity
v
ratio defined by the equation

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$$v = \frac{\eta}{\rho} \qquad (2)$$

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where ρ is the density of the fluid at the temperature at which the viscosity is measured

NOTE 1 The dimensions of kinematic viscosity are $\mathrm{L}^2\mathrm{T}^{-1}$.

NOTE 2 The units of kinematic viscosity are $\mathrm{m}^2\cdot\mathrm{s}^{-1}$.

NOTE 3 For practical use, the sub-multiple 10^{-6} $\mathrm{m}^2\cdot\mathrm{s}^{-1}$, i.e. $\mathrm{mm}^2\cdot\mathrm{s}^{-1}$, is more convenient.

3.3 Definitions applicable to polymer solutions

3.3.1
relative viscosity
η_r
ratio of the viscosity of the polymer solution (of stated concentration) η and the viscosity of the pure solvent η_0, at the same temperature:

$$\eta_\mathrm{r} = \frac{\eta}{\eta_0} \qquad (3)$$

NOTE 1 Also known as viscosity ratio.

NOTE 2 The ratio has no dimensions.

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3.3.2
relative viscosity increment

viscosity ratio minus one:

$$\left(\frac{\eta}{\eta_0}\right)-1=\frac{\eta-\eta_0}{\eta_0} \qquad (4)$$

NOTE 1 Also known as viscosity ratio increment and specific viscosity.

NOTE 2 The increment has no dimensions.

3.3.3
reduced viscosity

I

ratio of the viscosity ratio increment to the polymer concentration c in the solution:

$$I=\frac{\eta-\eta_0}{\eta_0 c} \qquad (5)$$

NOTE 1 Also known as viscosity number.

NOTE 2 The dimensions of reduced viscosity are $L^3 M^{-1}$.

NOTE 3 The units of reduced viscosity are m^3/kg.

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NOTE 4 For practical use, the sub-multiple 10^{-3} m^3/kg, i.e. cm^3/g, is more convenient and the commonly quoted numerical values for reduced viscosity (viscosity number) use these practical units.

NOTE 5 The reduced viscosity is usually determined at low concentration (less than 5 kg/m^3, i.e. 0,005 g/cm^3), except in the case of polymers of low molar mass, for which higher concentrations may be necessary.

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3.3.4
inherent viscosity

ratio of the natural logarithm of the viscosity ratio to the polymer concentration in the solution:

$$\frac{\ln\left(\frac{\eta}{\eta_0}\right)}{c} \qquad (6)$$

NOTE 1 Also known as logarithmic viscosity number.

NOTE 2 The dimensions and units are the same as those given in 3.3.3.

NOTE 3 The inherent viscosity is usually determined at low concentration (less than 5 kg/m^3, i.e. 0,005 g/cm^3), except in the case of polymers of low molar mass, for which higher concentrations may be necessary.

3.3.5
intrinsic viscosity

$[\eta]$

limiting value of the reduced viscosity or of the inherent viscosity at infinite dilution:

$$[\eta] = \lim_{c \to 0} \left(\frac{\eta - \eta_0}{\eta_0 c} \right)$$

$$[\eta] = \lim_{c \to 0} \frac{\ln \left(\dfrac{\eta}{\eta_0} \right)}{c} \tag{7}$$

NOTE 1 Also known as limiting viscosity number.

NOTE 2 The dimensions and units are the same as those given in 3.3.3.

NOTE 3 The effect of the shear rate on the functions defined in 3.3.1 to 3.3.5 has been neglected, since this effect is usually negligible for values of the reduced viscosity, inherent viscosity and intrinsic viscosity less than 0,5 m^3/kg, i.e. 500 cm^3/g. Strictly speaking, all these functions should be defined at the limiting (preferably infinitely small) value of the shear rate.

3.3.6
K-value

constant, independent of the concentration of the polymer solution and peculiar to the polymer sample, which is a measure of the average degree of polymerization:

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$$K\text{-value} = 1\,000k \tag{8}$$

NOTE 1 According to H. Fikentscher[2], k is calculated as follows:

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$$\lg \eta_\text{r} = \left(\frac{75k^2}{1 + 150kc} + k \right) 100c$$

and therefore

$$k = \frac{1{,}5 \lg \eta_\text{r} - 1 + \sqrt{1 + \left(\dfrac{2}{c} + 2 + 1{,}5 \lg \eta_\text{r} \right) 1{,}5 \lg \eta_\text{r}}}{150 + 300c} \tag{9}$$

where

$\eta_\text{r} = \dfrac{\eta}{\eta_0}$ = the viscosity ratio (see 3.3.1);

c is the concentration, in 10^3 kg/m^3, i.e. g/cm^3.

NOTE 2 A limiting viscosity number $[\eta]_k$ can be calculated from k:

$$[\eta]_k = 230{,}3 \left(75k^2 + k \right)$$

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4 Principle

The data needed for the evaluation of the functions defined in 3.3 are obtained by means of a capillary-tube viscometer. The efflux times of a given volume of solvent t_0 and of solution t are measured at fixed temperature and atmospheric-pressure conditions in the same viscometer. The efflux time of a liquid is related to its viscosity by the Poiseuille-Hagenbach-Couette equation:

$$\nu = \frac{\eta}{\rho} = Ct - \left(\frac{A}{t^2}\right) \tag{10}$$

where

ν is the viscosity/density ratio (see 3.2.2);

C is a constant of the viscometer;

A is a parameter of the kinetic-energy correction;

ρ is the density of the liquid;

t is the efflux time.

For the purposes of this part of ISO 1628, the kinetic energy correction $\left(\frac{A}{t^2}\right)$ shall be regarded as negligible when it is less than 3 % of the viscosity of the solvent. Hence Equation (10) can be reduced to

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$$\nu = \frac{\eta}{\rho} = Ct \tag{11}$$

Moreover, if the solution concentrations are limited so that the solvent density ρ_0 and that of the solution ρ differ by less than 0,5 %, the viscosity ratio $\frac{\eta}{\eta_0}$ will be given by the so-called "efflux time ratio" $\frac{t}{t_0}$.

The need for these constraints, and the consequences of not observing them, is developed in Annex B.

5 Apparatus

5.1 Capillary viscometer, of the suspended-level Ubbelohde type.

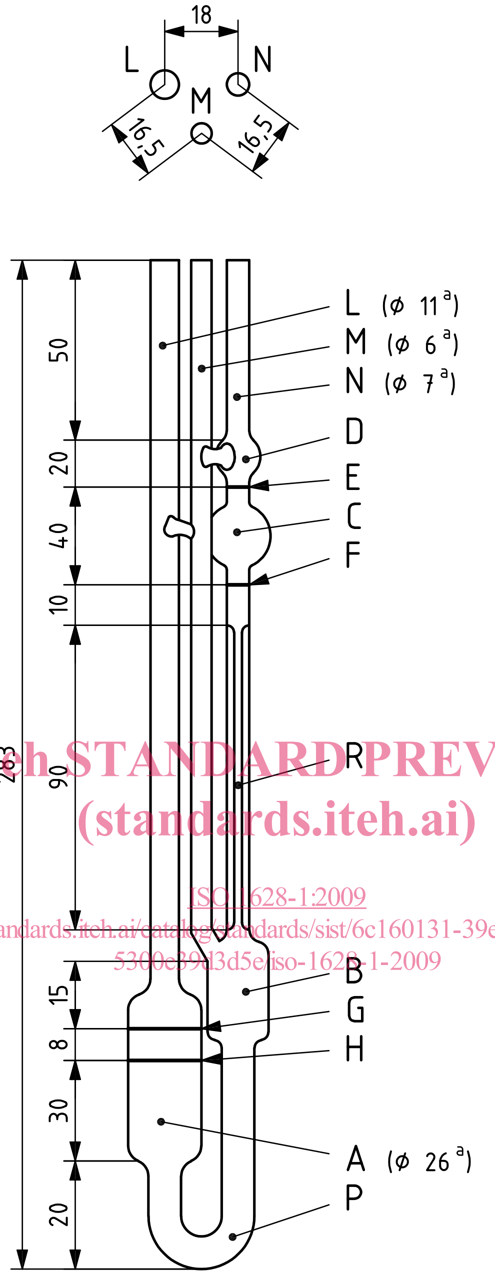
The use of a viscometer having the dimensions given in Figure 1 or 2 is strongly recommended. Furthermore, it is strongly recommended that the size of the viscometer be chosen from among those listed in Table 1. The choice is determined by the viscosity/density ratio of the solvent at the temperature of the measurement, as indicated in Table 1. The next-smaller viscometer can also be used.

Other types of viscometer listed in ISO 3105 can be used, provided they give results equivalent to those given by the particular size of Ubbelohde viscometer chosen on the basis of the criteria specified in the preceding paragraph. In cases of dispute, an Ubbelohde viscometer shall be used.

With automated apparatus, fitted with special timing devices, it may be possible to obtain equivalent results with larger sizes of capillary than those listed for the appropriate solvent viscosity/density ratio in Table 1.

5.2 Viscometer holder, suitable to hold the viscometer firmly in the thermostatic bath (5.3) in the vertical position.

Dimensions in millimetres



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Graduation marks: E and F

Filling marks: G and H

[a] Internal diameter.

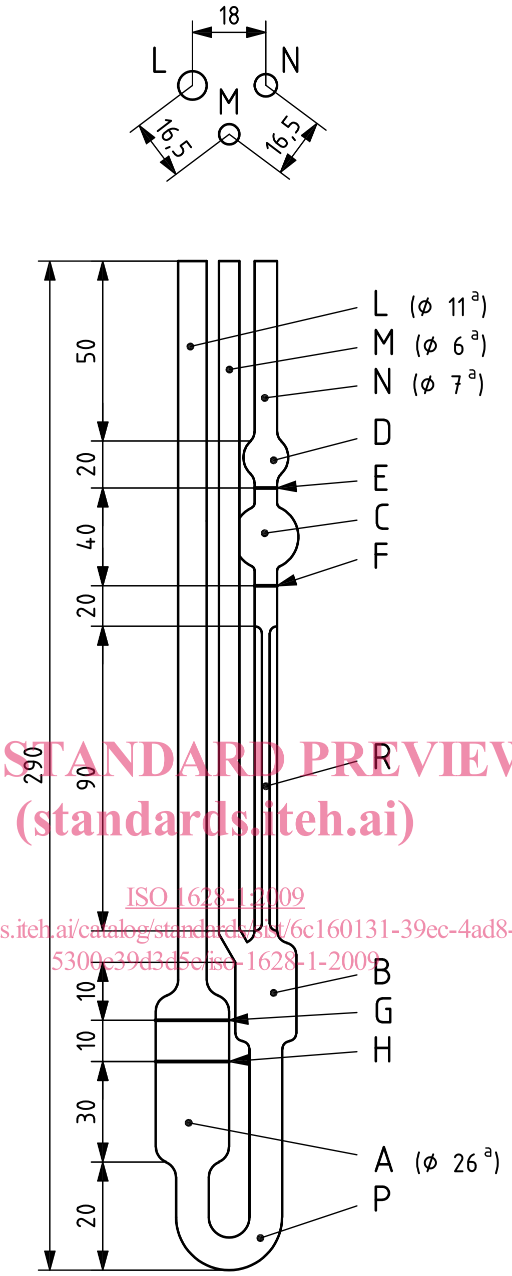
Figure 1 — Ubbelohde viscometer

5.3 Thermostatic bath, holding a transparent liquid or vapour and of such depth that, during the measurement, no portion of the test liquid will be less than 20 mm below the surface of the bath medium or less than 20 mm above the bottom of the bath.

The temperature control shall be such that, within the range 25 °C to 100 °C, the temperature of the bath does not vary from the specified temperature by more than 0,05 °C over the length of the viscometer, or between the viscometers if several determinations are carried out simultaneously.

At temperatures higher than 100 °C, the tolerance shall be ± 0,2 °C.

Dimensions in millimetres



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Graduation marks: E and F

Filling marks: G and H

[a] Internal diameter.

Figure 2 — DIN Ubbelohde viscometer

5.4 Temperature-measuring device.

A liquid-in-glass "total immersion" thermometer, reading to 0,05 °C in the range in which it will be used and in a known state of calibration, is suitable. Other thermometric devices of at least equal precision may be used.

5.5 Timing device.

Any timing device may be used providing that it can be read to 0,1 s and that its speed is constant to 0,1 % over 15 min.