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

**Part 1:
General principles**

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

Partie 1: Principes généraux

ISO 1628-1:1998

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

This second edition cancels and replaces the first edition (ISO 1628-1:1984), which has been revised to include the following changes:

- a) the determination of the K -value;
- b) a procedure for the determination of the efflux times for several solution concentrations by the addition of solvent to a given solution held in the viscometer;
- c) revised viscometer specifications.

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*

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- *Part 4: Polycarbonate (PC) moulding and extrusion materials*
- *Part 5: Thermoplastic polyester (TP) homopolymers and copolymers*
- *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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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.

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2 Normative references

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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 31-0:1992, *Quantities and units — Part 0: General principles*.

ISO 31-3:1992, *Quantities and units — Part 3: Mechanics*.

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

ISO 3205:1976, *Preferred test temperatures*.

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 31-0, while the units appropriate to the properties are given in ISO 31-0 and ISO 31-3.

3.2 Definitions applicable to any liquid

3.2.1 viscosity: The 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, is defined by the Newton equation

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

where

τ is the shear stress;

η is the viscosity;

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

The dimensions of viscosity are: $ML^{-1}T^{-1}$.

The units of viscosity are: Pa·s.

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

NOTE — 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, ν : This ratio is defined by the equation

$$\nu = \frac{\eta}{\rho} \quad \dots(2)$$

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

The dimensions of kinematic viscosity are: L^2T^{-1} .

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

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

3.3 Definitions applicable to polymer solutions

3.3.1 relative viscosity, η_r (also known as viscosity ratio): The 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:

$$\eta_r = \frac{\eta}{\eta_0} \quad \dots(3)$$

The ratio has no dimensions.

3.3.2 relative viscosity increment (also known as viscosity ratio increment and specific viscosity): The viscosity ratio minus one:

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

The increment has no dimensions.

3.3. reduced viscosity, I (also known as viscosity number): The ratio of the viscosity ratio increment to the polymer concentration c in the solution:

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

The dimensions of reduced viscosity are: L^3M^{-1} .

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

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.

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 (also known as logarithmic viscosity number): The ratio of the natural logarithm of the viscosity ratio to the polymer concentration in the solution:

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

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

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, $[\eta]$ (also known as limiting viscosity number): The limiting value of the reduced viscosity or of the inherent viscosity at infinite dilution:

$$[\eta] = \lim_{c \rightarrow 0} \left(\frac{\eta - \eta_0}{\eta_0 c} \right) \quad \dots(7)$$

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

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

NOTE — 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 m³/kg, i.e. 500 cm³/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: A constant independent of the concentration of the polymer solution and peculiar to the polymer sample. It is a measure of the average degree of polymerization.

$$K\text{-value} = 1\,000\,k \quad \dots(8)$$

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

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

and therefore

$$k = \frac{1,5 \lg \eta_r - 1 + \sqrt{1 + \left(\frac{2}{c} + 2 + 1,5 \lg \eta_r \right) 1,5 \lg \eta_r}}{150 + 300\,c} \quad \dots(9)$$

where

$\eta_r = \frac{\eta}{\eta_0}$ = the viscosity ratio (see 3.3.1);

c is the concentration, in 10³ kg/m³, i.e. g/cm³.

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

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

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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:

$$v = \frac{\eta}{\rho} = kt - \left(\frac{A}{t^2} \right) \quad \dots(10)$$

where

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

k 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

$$v = \frac{\eta}{\rho} = kt \quad \text{..(11)}$$

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, 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 is 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.

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 $\pm 0,2$ °C.

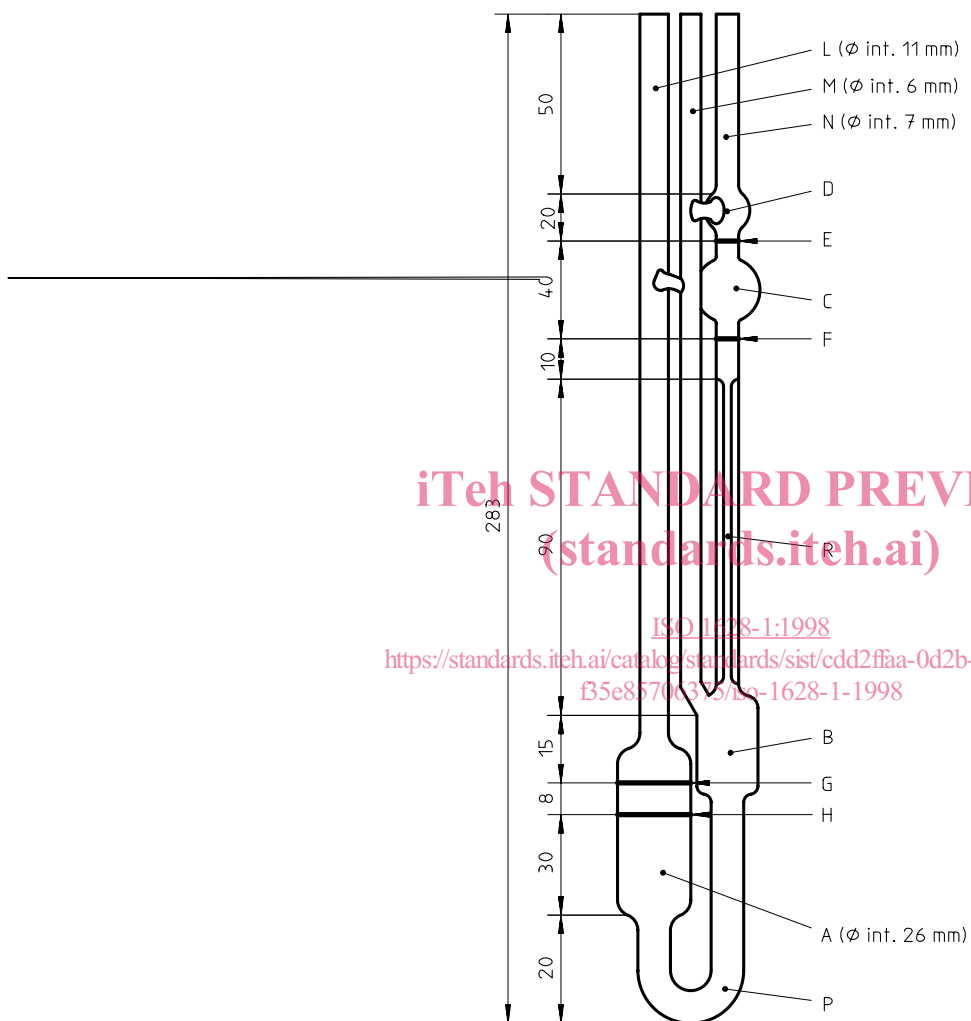
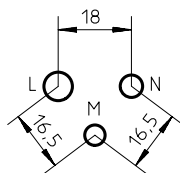
5.4 Temperature-measuring device.

A liquid-in-glass, "total immersion" thermometer, reading to 0,05 °C in the range to 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.

Dimensions in millimetres



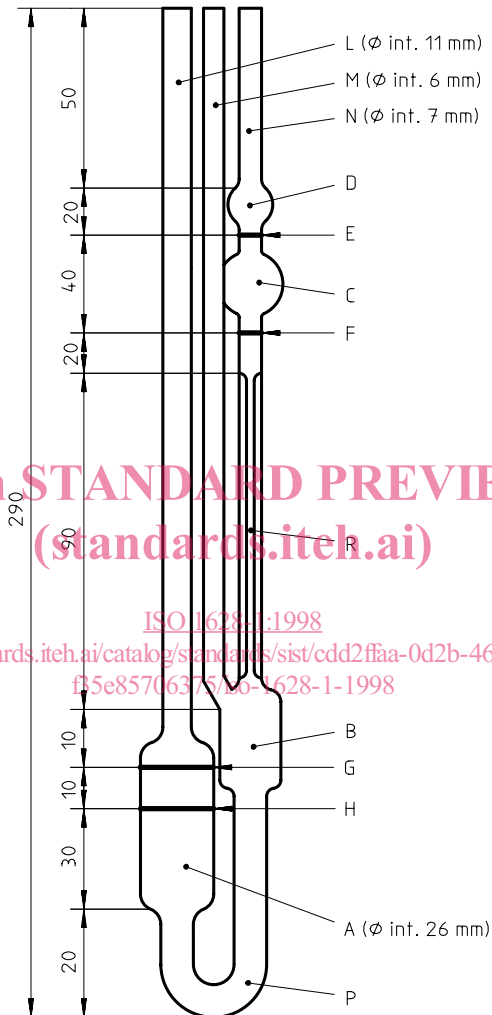
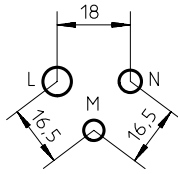
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