INTERNATIONAL STANDARD

ISO 1628-1

Fourth edition
2021-02

# 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*

Reference number
ISO 1628-1:2021(E)

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Published in Switzerland

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# Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 249, *Plastics,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fourth edition cancels and replaces the third edition (ISO 1628-1:2009), which has been technically revised. It also incorporates the Amendment 1 ISO 1628-1:2009/Amd 1:2012.

The main changes compared to the previous edition are as follows:

- ISO 3205 (withdrawn) has been deleted from Clause 2;
- the figure keys have been revised;
- nominal viscometer constant has been added to Table 1;

A list of all parts in the ISO 1628 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

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

## Part 1:
## General principles

## 1 Scope

This document 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.

This document is used to develop standards for measuring the viscosities in solution of individual types of polymer. It is also used to measure and report the viscosities of polymers in solution for which no separate standards exist.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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, *Glass capillary kinematic viscometers — Specifications and operating instructions*

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

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

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 80000-1, ISO 80000-4 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

### 3.1 Terms related to any liquid

**3.1.1**
**viscosity**
property 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 formula

$$\tau = \eta \dot{\gamma}$$

where

$\tau$ is the shear stress;

$\eta$ 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 to entry: The dimensions of viscosity are $\mathrm{ML}^{-1}\mathrm{T}^{-1}$.

Note 2 to entry: The units of viscosity are Pa·s.

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

Note 4 to entry: 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.

### 3.1.2
**viscosity/density ratio**
**kinematic viscosity**
$v$
ratio defined by the formula

$$v=\frac{\eta}{\rho}$$

where $\rho$ is the density of the fluid at the temperature at which the viscosity is measured

Note 1 to entry: The dimensions of kinematic viscosity are $\mathrm{L}^2\mathrm{T}^{-1}$.

Note 2 to entry: The units of kinematic viscosity are $\mathrm{m}^2 \cdot \mathrm{s}^{-1}$.

Note 3 to entry: 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.

## 3.2 Terms related to polymer solutions

### 3.2.1
**relative viscosity**
**viscosity ratio**
$\eta_r$
ratio of the viscosity of the polymer solution (of stated concentration) $\eta$ and the viscosity of the pure solvent $\eta_0$, at the same temperature:

$$\eta_\mathrm{r}=\frac{\eta}{\eta_0}$$

Note 1 to entry: The ratio has no dimensions.

### 3.2.2
**relative viscosity increment**
**viscosity ratio increment and specific viscosity**
viscosity ratio minus one

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

Note 1 to entry: The increment has no dimensions.

### 3.2.3
### reduced viscosity
### viscosity number

*I*

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

$$I = \frac{\eta - \eta_0}{\eta_0 c}$$

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

Note 2 to entry: The units of reduced viscosity are $m^3/kg$.

Note 3 to entry: 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 4 to entry: 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 is necessary.

### 3.2.4
### inherent viscosity
### logarithmic viscosity number

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

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

Note 1 to entry: The dimensions and units are the same as those given in 3.2.3.

Note 2 to entry: 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 is necessary.

### 3.2.5
### intrinsic viscosity
### limiting viscosity number

[η]

limiting value of the reduced viscosity or of the *inherent viscosity* (3.2.4) at infinite dilution:

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

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

Note 1 to entry: The dimensions and units are the same as those given in 3.2.3.

Note 2 to entry: The effect of the shear rate on the functions defined in 3.2.1 to 3.2.5 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 can be defined at the limiting (preferably infinitely small) value of the shear rate.

### 3.2.6
### *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:

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

Note 1 to entry: According to H. Fikentscher[1], *k* is calculated as follows:

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

and therefore

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

where

$\eta_\mathrm{r}=\frac{\eta}{\eta_0}$ is the viscosity ratio (see 3.2.1);

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

Note 2 to entry: A limiting viscosity number $[\eta]_\mathrm{k}$ can be calculated from $k$:

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

## 4 Principle

The data needed for the evaluation of the functions defined in 3.2 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 formula as shown in Formula (1):

$$v=\frac{\eta}{\rho}=Ct-\left(\frac{A}{t^2}\right) \tag{1}$$

where

$v$ is the viscosity/density ratio ;

$C$ is a constant of the viscometer;

$A$ is a parameter of the kinetic-energy correction;

$\rho$ is the density of the liquid;

$t$ is the efflux time.

For the purposes of this document, 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, Formula (1) can be reduced to Formula (2):

$$v=\frac{\eta}{\rho}=Ct \tag{2}$$

Moreover, if the solution concentrations are limited so that the solvent density $\rho_0$ and that of the solution $\rho$ 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 described in Annex B.

## 5 Apparatus

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

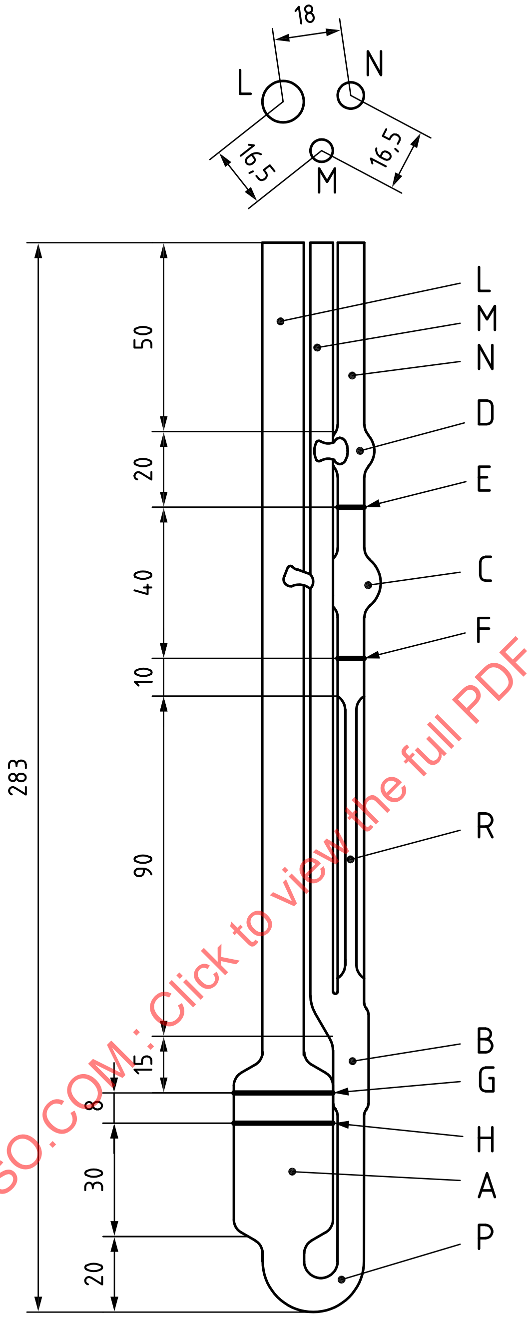
The use of a viscometer having the dimensions given in Figure 1 or Figure 2 is strongly recommended. Furthermore, it is strongly recommended that the size of the viscometer be chosen from among those listed in Table 1. The choice is determined by the viscosity/density ratio of the solvent at the temperature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

Other types of viscometer listed in ISO 3105 may 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, equivalent results with larger sizes of capillary than those listed for the appropriate solvent viscosity/density ratio in Table 1 can be obtained.

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

Dimensions in millimetres



**Key**

| | | | |
|---|---|---|---|
| A | lower reservoir 26 mm internal diameter | L | mounting tube 11 mm internal diameter |
| B | suspended level bulb | M | lower vent tube 6 mm internal diameter |
| C | timing bulb | N | upper vent tube 7 mm internal diameter |
| D | upper reservoir | P | connecting tube |
| E and F | timing marks | R | working capillary |
| G and H | filling marks | | |

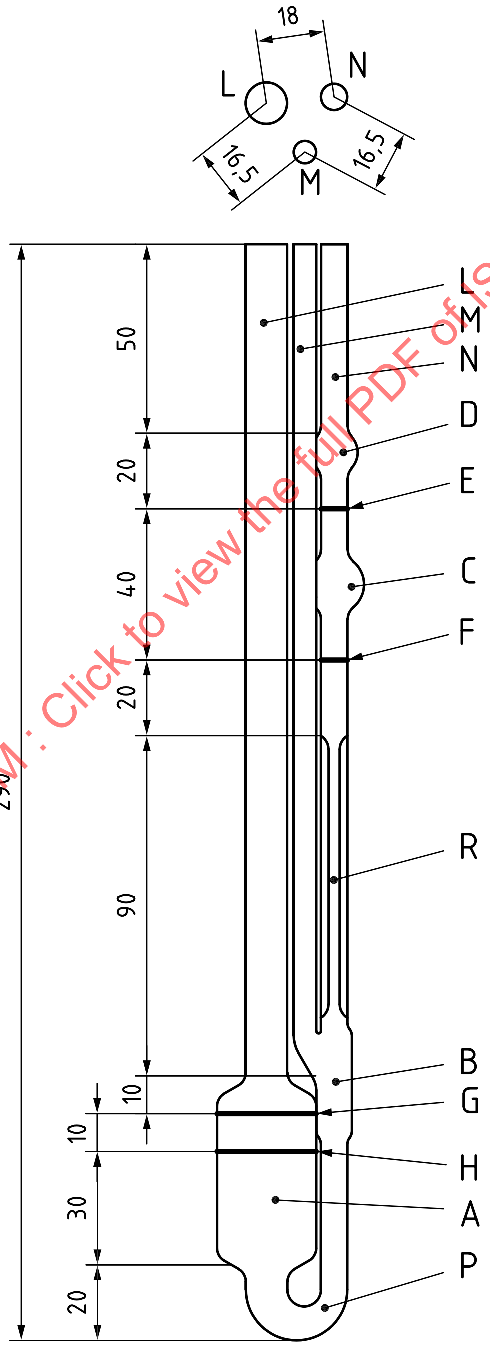
**Figure 1 — Ubbelohde viscometer**

**5.3 Thermostatic bath,** transparent liquid or vapour bath of a size such that, during the measurement, all sections containing test liquid are at least 20 mm below the surface of the bath medium and at least 20 mm away from all boundaries of the bath tank.

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 K 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



NOTE For key, see Figure 1.

Figure 2 — DIN Ubbelohde viscometer

**5.4 Temperature-measuring device,** thermometer, reading to 0,02 °C in the range in which it will be used and in a known state of calibration, is suitable.

**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.

**Table 1 — Ubbelohde viscometers recommended for the determination of the dilute-solution viscosity of polymers**

| Viscosity/density ratio of solvent at temperature of measurement | Nominal viscometer constant | Ubbelohde conforming to ISO 3105 | | DIN Ubbelohde conforming to ISO 3105 | |
|---|---|---|---|---|---|
| $\text{mm}^2\cdot\text{s}^{-1}$ | $\text{mm}^2\cdot\text{s}^{-2}$ | Size No. | Inside diameter of tube R[a] mm | Size No. | Inside diameter of tube R[a] mm |
| 0,15 to 0,30 | 0,001 | 0 | 0,24 | 0 | 0,36 |
| 0,31 to 0,50 | 0,003 | 0C | 0,36 | 0c | 0,47 |
| 0,51 to 0,75 | 0,005 | 0B | 0,46 | 0a | 0,53 |
| 0,76 to 1,50 | 0,01 | 1 | 0,58 | I | 0,63 |
| 1,51 to 2,50 | 0,03 | 1C | 0,77 | Ic | 0,84 |
| 2,51 to 5,00 | 0,05 | 1B | 0,88 | Ia | 0,95 |
| 5,01 to 15,00 | 0,1 | 2 | 1,03 | II | 1,13 |

[a] The tolerance of the inside diameter of tube R is ±2 %.

## 6 Solutions

### 6.1 Preparation

The dissolution of the test sample of polymer in the solvent shall give a “true” solution, essentially free of microgels and associated macromolecules. Polymer degradation shall also be minimized. For these reasons, it is necessary for the dissolution procedure to be exactly defined and it is recommended that the following factors be specified:

a) the solvent and its pretreatment, if any;

b) the apparatus and the method of agitation;

c) the temperature range within which the system is maintained during the preparation of the solution;

d) the time interval necessary for the complete dissolution of the polymer without degradation, or at constant degradation;

e) the stabilizer and/or the protective atmosphere used;

f) the conditions of filtration of the solution, if applicable;

g) visual homogeneity of the solution and expected nature/composition of the filter residue.

## 6.2 Concentration

Where no standard exists, careful consideration shall be given to the choice of solvent and the solution concentration. The solution concentration shall be chosen so that the ratio of the efflux time of the solution to the efflux time of the solvent is at least 1,2 and less than 2,0.

NOTE A lower limit of 1,2 ensures sufficient precision of the measured difference in efflux times. The upper limit of 2,0 prevents shear effects and non-linearity of the viscosity number in relation to concentration that can occur at higher molecular masses.

More than one concentration can therefore be used for a given polymer/solvent system, depending on the molecular mass of the polymer under test.

The concentration is preferably expressed in kg/m$^3$ of solution or as the multiple 10$^3$ kg/m$^3$, i.e. g/cm$^3$.

# 7 Temperature of measurement

The temperature shall be chosen with due regard to sufficient solubility and other technical requirements, but kept constant for any particular polymer/solvent system. The temperature tolerance shall be specified. A temperature of 25 °C ± 0,05 °C shall be chosen whenever possible. If another temperature is used, use the temperature that was agreed between the parties concerned and stated in the test report.

# 8 Procedure

## 8.1 General

Measure the efflux times for the solution and the solvent successively in the same viscometer, using the procedure described in 8.2 and 8.3.

## 8.2 Preparing and charging the viscometer

**8.2.1** Maintain the bath at the specified test temperature.

**8.2.2** Charge the dry, clean viscometer (the cleaning procedure is specified in Annex A) by tilting it about 30° from the vertical and pouring sufficient liquid through tube L (see Figure 1 or Figure 2) so that, when the viscometer is returned to the vertical, the meniscus is between the filling marks G and H. Avoid trapping air bubbles in the viscometer. The initial filling may be carried out away from the bath.

**8.2.3** Mount the viscometer in a holder in the bath, ensuring that tube N is vertical. Allow time for the charged viscometer to reach the temperature of the bath. Usually, 15 min will suffice if the measurement is to be made at 25 °C. At higher temperatures, longer times is necessary. Unnecessary delays should be avoided as it is found that the most consistent results are obtained shortly after temperature equilibrium is attained with a freshly charged viscometer.

**8.2.4** This procedure shall also be followed when a measured amount of solvent is added to a solution, already contained in the viscometer, in order to create a more dilute solution for additional determinations. The additional solvent shall be maintained at the specified test temperature prior to use.

**8.2.5** In automated equipment, the viscometer is fixed in the vertical position within a temperature-controlled bath and the apparatus is designed to fill the viscometer with liquid in this position. The bath shall be maintained at the specified test temperature and an equilibration time selected in accordance with 8.2.3.

### 8.3 Efflux time measurement

See Figure 1 or Figure 2.

Close tube M and apply suction to tube N, or pressure to tube L, until the liquid reaches a level about 5 mm above mark E. Hold the liquid at this level by closing tube N.

Open tube M so that the liquid drops away from the lower end of capillary tube R.

When the liquid is clear of the end of the capillary and the lower end of tube M, open tube N. Measure the efflux time to the nearest 0,2 s as the time taken for the bottom of the meniscus to pass from the top edge of mark E to the top edge of mark F. For polymer solutions containing small amounts of finely divided pigments, such as carbon black, it shall be necessary to view the top of the meniscus. Where large concentrations of pigments are involved, it shall be necessary to centrifuge the solution before proceeding.

The opening and closing of the tubes may be carried out conveniently by means of taps or clips on clean flexible tubing attached to the ends of the tubes, making sure that no fluff or other contaminant enters the tube.

The first flow shall not be taken into account for measuring the efflux time. Repeat the measurement of efflux time immediately, without emptying or recharging, until two successive efflux times agree to within 0,25 %. Take the mean of these two values as the efflux time.

If two successive determinations of the mean efflux time of the solvent differ by more than 0,4 s, clean the viscometer as specified in Annex A.

The efflux time for any “new” solution, created by the addition of solvent to an existing solution held in the viscometer, shall be determined as a separate liquid, in accordance with this procedure.

## 9 Expression of results

### 9.1 Reduced viscosity and intrinsic viscosity

Results are preferably expressed as a reduced viscosity.

The reduced viscosity $I$, in the units $10^{-3}$ m$^3$/kg, i.e. cm$^3$/g, is calculated using Formula (3):

$$I = \frac{t-t_0}{t_0 c} \tag{3}$$

$t$ is the efflux time of the solution, in seconds;

$t_0$ is the efflux time of the solvent, in seconds, through the same viscometer;

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

The results can also be expressed as an intrinsic viscosity $[\eta]$, for example in order to compare copolymers of different average molecular masses for which it has been necessary to use different concentrations (see 6.2).

The intrinsic viscosity shall be calculated from the values of inherent viscosity or reduced viscosity obtained for concentrations c1, c2, c3 ..., in the approximate ratio 1 : 2 : 3 ..., by a graphical method consisting of plotting the inherent-viscosity values or the reduced-viscosity values (on the ordinate axis) against the concentration (on the abscissa) and extrapolating the curve to zero concentration. The intrinsic viscosity is read off the ordinate axis.

It is recommended that the method of least squares be used for analysing the experimental values.

The intrinsic viscosity [$\eta$] can also be calculated from one value of the reduced viscosity, for instance by using Formula (4):

$$[\eta]=\frac{I}{1+k'cI} \tag{4}$$

where $k'$ is an experimental constant, usually between 0,2 and 0,3, which must be determined previously by measuring the reduced viscosity at two different concentrations and applying Formula (5):

$$k'=\frac{I_2-I_1}{I_1 I_2 (c_2-c_1)} \tag{5}$$

In the calculation of $k'$, it should be preferable to use several concentrations and plot $I$ against $c$, drawing a straight line through the points and selecting two positions on this line to give values for substitution in the formula for $k'$.

### 9.2 *K*-value

The *K*-value can be calculated from the relationship shown as Formula (6):

$$\text{K-value}=1\,000\ \text{k} \tag{6}$$

where $k$ is defined by the formulae given in 3.2.6.

## 10 Test report

The test report shall contain the following particulars:

a) a reference to this document, i.e. ISO 1628-1:2021, and the particular standard for the polymer tested, if one exists;

b) all details necessary for identification of the material tested, including type, source and manufacturer’s code number;

c) the concentration of material in the solution used, a description of the solvent and details of the preparation of the solution;

d) details of the viscometer used;

e) the temperature of the test;

f) the test results;

g) the date of the test.

# Annex A
(normative)

# Cleaning of apparatus

It is essential that all apparatus coming into contact with the liquid under test is scrupulously clean. Any impurities, such as dust, traces of liquid or filaments in the viscometer, will cause false results to be obtained.

Before the test, clean the viscometer and all other apparatus used (glass vessels, pipettes, sintered-glass filters, rubber tubing, etc.). A suitable detergent may be adequate. Aqua regia (3 volumes concentrated hydrochloric acid and 1 volume concentrated nitric acid) may be required to remove inorganic residues from the glassware, and a suitable solvent may be required to remove oil and grease. Following this, dry the glass apparatus and then clean it with specially prepared chromic acid solution. Leave this solution in contact with the glassware overnight at a minimum temperature of 20 °C. For more active cleaning, warm the chromic acid gently on a water bath.

Remove the chromic acid and rinse at least five times with distilled or deionized water. Dry in an electric oven at a temperature no higher than 100 °C or rinse at least five times with acetone that has been distilled, dried and filtered, drying the apparatus with a slow stream of filtered dry air or, preferably, with a vacuum line.

Between successive determinations of the efflux times of samples of a similar nature, the viscometer shall be cleaned by draining and thoroughly rinsing with a suitable volatile solvent that has been distilled and filtered. Dry the apparatus with a slow stream of filtered dry air, with a vacuum line, or in an electric oven at a temperature no higher than 100 °C. The effectiveness of the cleaning can be checked by establishing that the efflux times with a given solvent and viscometer remain constant.

If the next solution to be measured is of a polymer of the same type and of similar viscosity, it is permissible to drain the viscometer, wash it with the next solution to be measured and then fill it with a further quantity of this solution.

It is recommended that viscometers used for silicone fluids and fluorocarbons be reserved exclusively for use with these fluids and that these viscometers be subject to calibration checks at frequent intervals. Great care shall be taken to ensure that these fluids do not contaminate other apparatus, either directly or indirectly.

**WARNING — Particular care should be taken when using aqua regia and chromic acid. Chromic acid is toxic. Appropriate action should also be taken to ensure the safe disposal of these acids. Goggles and protective gloves should be worn. Any splashes should be washed off the skin immediately with plenty of cold water. Inhalation of the vapours should be avoided.**

# Annex B
(informative)

# Notes on sources of error

## B.1 General

Guidance is given in this document on the determination of the viscometric properties of polymers in dilute solution as defined in Clause 3. The measurement of these properties can usually be carried out with better reproducibility than that attained in absolute measurements of viscosity since many of the instrumental errors are approximately the same for both solvent and solution, and are effectively cancelled.

## B.2 Principle of measurements

The relative viscosity and the other parameters defined in Clause 3 are calculated from the efflux times of solution and solvent. The liquid flows, under the force of gravity, through the capillary of a suspended-level Ubbelohde viscometer. The efflux times are taken to be proportional to the viscosity/density ratio of the fluids (see Clause 4). This is equivalent to stating that the kinetic energy and other errors are negligible and that the density of the solvent differs negligibly from the density of the solutions.

## B.3 Sources of error

The most important sources of error in capillary viscometry are related to

a) surface tension,

b) capillary end effects,

c) drainage effects,

d) viscous heat effects,

e) deviation of the viscometer from the vertical,

f) hydrostatic head variations,

g) losses by evaporation,

h) errors in the measurement of the concentration and efflux time,

i) shear effects,

j) errors in temperature stability and measurement,

k) kinetic energy.

Fortunately, most of these effects can be considered negligible for the proposed procedures. Errors due to surface tension, end effects and drainage effects are quite small in tests with organic solvents in the Ubbelohde viscometer, when relative measurements of viscosity are concerned[2],[3]. Viscous heat effects are negligible when the capillary viscometer is operated by gravity. Errors due to deviation from the vertical and variations in the hydrostatic head are usually very small with this type of viscometer.

Solvent losses by evaporation and concentration errors depend on the particular test procedure and have to be taken into account.