
Polimerni materiali - Določanje viskoznosti polimerov v razredčenih raztopinah s kapilarnimi viskozimetri - 2. del: Polivinilklorid (ISO/FDIS 1628-2:2020)

Plastics - Determination of the viscosity of polymers in dilute solution using capillary viscometers - Part 2: Poly(vinyl chloride) resins (ISO/FDIS 1628-2:2020)

Plastiques - Détermination de la viscosité des polymères en solution diluée à l'aide de viscosimètres à capillaires - Partie 2: Résines de poly (chlorure de vinyle) (ISO/FDIS 1628-2:2020)

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

Part 2: Poly(vinyl chloride) resins

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

Partie 2: Résines de poly(chlorure de vinyle)

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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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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 9, *Thermoplastic materials*, 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 third edition cancels and replaces ISO 1628-2:1998, of which it constitutes a minor revision.

The change compared to the previous edition is as follows:

— [Clause 2](#) has been updated.

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 2: Poly(vinyl chloride) resins

1 Scope

1.1 This document specifies conditions for the determination of the reduced viscosity (also known as viscosity number) and *K*-value of PVC resins. It is applicable to resins in powder form which consist of homopolymers of the monomer vinyl chloride and copolymers, terpolymers, etc., of vinyl chloride with one or more other monomers, but where vinyl chloride is the main constituent. The resins may contain small amounts of unpolymerized substances (e.g. emulsifying or suspending agents, catalyst residues, etc.) and other substances added during the course of the polymerization. This document is not applicable, however, to resins having a volatile-matter content in excess of 0,5 % ± 0,1 %, when determined in accordance with ISO 1269. In addition to this, it is not applicable to resins which are not entirely soluble in cyclohexanone.

1.2 The reduced viscosity and *K*-value of a particular resin are related to its molecular mass, but the relationship varies depending on the concentration and type(s) of other monomer(s) present. Hence, homopolymers and copolymers having the same reduced viscosity or *K*-value might not have the same molecular mass.

1.3 The values determined for reduced viscosity and *K*-value, for a particular sample of PVC resin, are influenced differently by the concentration of the solution chosen for the determination. Hence the use of the procedures described in this document only gives values for reduced viscosity and *K*-value that are comparable when the concentrations of the solutions used are identical.

1.4 Limiting viscosity number is not used for PVC resins.

1.5 The experimental procedures described in this document can also be used to characterize the polymeric fraction obtained during the chemical analysis of a PVC composition. However, the values calculated for the reduced viscosity and *K*-value in these circumstances might not indicate the actual values for the resin used to produce the composition because of the impure nature of the recovered polymer fraction.

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 1042, *Laboratory glassware — One-mark volumetric flasks*

ISO 1628-1:2009, *Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 1: General principles*

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

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3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1628-1 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/>

4 Principle

4.1 A test portion is dissolved in a solvent. The reduced viscosity and the *K*-value are calculated from the efflux times for the solvent and the solution in a capillary tube viscometer.

5 Materials

5.1 Cyclohexanone, having a viscosity/density ratio (kinematic viscosity) between $2,06 \times 10^{-6} \text{ m}^2 \text{ s}^{-1}$ and $2,33 \times 10^{-6} \text{ m}^2 \text{ s}^{-1}$ ($2,06 \text{ mm}^2 \text{ s}^{-1}$ and $2,33 \text{ mm}^2 \text{ s}^{-1}$) at 25 °C. The specified boiling point shall be 155 °C. Store the solvent in the dark in a dark-coloured bottle fitted with a ground-glass stopper. Check the kinematic viscosity before use.

6 Apparatus

6.1 The apparatus required to carry out viscosity measurements on polymers in dilute solution are in accordance with ISO 1628-1:2009, Clause 5. In addition, the following particular items are required for the procedures described in this document.

6.2 Viscometer. From the viscometers described in ISO 1628-1:2009, 5.1, model 1C, with a capillary diameter of $0,77 \text{ mm} \pm 2 \%$, from ISO 3105:1994, Table B.4, shall be used as the reference viscometer.

Other viscometers described in ISO 1628-1 may be used provided the correlation between the chosen viscometer and the reference viscometer has been established over the range of reduced viscosities and *K*-values to be measured, and the results are corrected accordingly.

6.3 Graduated flask (one-mark volumetric flask), class A, as specified in ISO 1042, with a volume of 50 ml.

NOTE The use of a flask calibrated at a temperature of 20 °C, as specified in ISO 1042, causes a systematic error which can, however, be neglected.

6.4 Filter funnel, with fritted-glass filter disc of medium porosity (pore size 40 µm to 50 µm), or **glass funnel** with paper filter.

6.5 Mechanical agitator, equipped with a heating device to keep the flask (6.3) and its contents at a temperature between 80 °C and 85 °C.

As an alternative, a rotary agitator or shaker may be placed in an oven at a temperature between 80 °C and 85 °C.

6.6 Analytical balance, accurate to 0,1 mg.

6.7 Temperature-regulated bath, capable of being set at $25,0 \text{ °C} \pm 0,5 \text{ °C}$ in steps of 0,1 °C and maintaining a stability of $\pm 0,05 \text{ °C}$ around the set temperature.

6.8 Thermometer, with a sensitivity of 0,05 °C.

6.9 Time-measuring device, with a sensitivity of 0,1 s.

7 Sampling

Take a sample which is representative of the resin whose properties are to be determined and large enough for at least two determinations.

8 Number of determinations

Carry out two complete determinations, starting each with a fresh test portion.

9 Procedure

9.1 Preparation of solution

General requirements for the dissolution of polymer in solvent are given in ISO 1628-1:2009, Clause 6. Prepare a solution with a concentration of $5 \text{ g/cm}^3 \pm 0,1 \text{ g/cm}^3$ at $25 \text{ °C} \pm 1 \text{ °C}$, as follows.

Weigh, to the nearest 0,2 mg, $0,250 \text{ g} \pm 0,005 \text{ g}$ of resin and transfer it quantitatively to the 50 ml flask (6.3). Add about 40 ml of cyclohexanone (5.1) to the flask, swirling the flask by hand to prevent coagulation or the formation of lumps. Continue dissolution by agitating for 1 h between 80 °C and 85 °C using the agitator (6.5). Check visually that dissolution is complete. If gelatinized particles are still visible, start again with a new portion of the resin. Cool the solution to $25 \text{ °C} \pm 1 \text{ °C}$ and make up to the mark with cyclohexanone at the same temperature. Mix the solution thoroughly by shaking.

Determine the actual concentration of the solution to an accuracy of $\pm 0,1 \%$.

If a mass of $0,250 \text{ g} \pm 0,000 25 \text{ g}$ is taken and made up to 50 ml of solution as described above, Table 1 can be used to read off the reduced viscosity and *K*-value from the ratio of the efflux time of the solution to that of the solvent (the so-called viscosity ratio).

Alternative methods for the preparation of the solution may be used, for example the addition of a measured volume of solvent to a measured mass of test portion, provided that the values obtained for the reduced viscosity and *K*-value can be shown to be equivalent to those obtained with the method of solution preparation described above. Such alternative methods of solution preparation requires the amounts of solvent and test portion taken to be determined by experiment, and can also require compensation for loss of solvent by evaporation during the dissolution process.

With resins having *K*-values greater than 85, the ratio of the efflux time of the solution to that of the solvent will exceed the maximum value of 2,0, which is contrary to the requirement specified in ISO 1628-1:2009, 6.2. In order to ensure uniformity of testing for PVC, this non-conformity shall be ignored, and all currently available resins tested using the same test-portion mass.

9.2 Determination of efflux times

The procedure is described in ISO 1628-1:2009, Clause 8.

The temperature of the thermostat (6.7) shall be set such that the actual temperature which is measured by the thermometer (6.8) lies in the range $25 \text{ °C} \pm 0,5 \text{ °C}$. The measured temperature shall be stable to $-0,05 \text{ °C}$ around the temperature at which the thermostat has been set.

When filling the viscometer, filter the solvent and the solution using a filter funnel or a glass funnel and paper filter (see 6.4).

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Particular care shall be taken over viscometer cleaning, which shall be based on the procedure described in ISO 1628-1:2009, Annex A. Efflux times with the control solvent cyclohexanone shall remain constant to within 0,2 s for a given viscometer. With the solution, repeat the measurement of the efflux time until two successive measurements differ by less than 0,25 %. Always discard the first efflux time reading.

NOTE This is a manual procedure. Proprietary equipment is available which will organize the charging of the viscometer with solution and solvent and measure the respective efflux times automatically. The use of such equipment is included in the scope of this document provided that all the procedures and verification checks described above are followed by the automated procedure.

10 Expression of results

10.1 Reduced viscosity

Calculate the reduced viscosity, I , for each test portion as specified in ISO 1628:2009, Clause 9, using [Formula \(1\)](#):

$$I = \frac{t - t_0}{t_0 c} \quad (1)$$

where

t and t_0 are the efflux times, in seconds, of the solution and solvent, respectively;

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

Calculate the reduced viscosity I for the sample as the mean value of the two individual values obtained in the two determinations, expressing the result to the nearest whole number. If the I values obtained in the two determinations deviate by more than $\pm 0,4 \%$ from the mean value, these results shall be rejected and further determinations carried out with fresh test portions.

If the solution concentration is $5 \text{ g/cm}^3 \pm 0,005 \text{ g/cm}^3$, it is more convenient to read off the values of I from [Table 1](#), expressing I in $(\text{m}^3/\text{kg}) \cdot 10^{-3}$, i.e. cm^3/g , rounded to the first place of decimals.

10.2 K-value

For each test portion, calculate the K-value as specified in ISO 1628-1:2009, Clause 9, using [Formula \(2\)](#):

$$K = \frac{1,5 \lg \eta_r - 1 + \sqrt{1 + \left(\frac{2}{c} + 2 + 1,5 \lg \eta_r \right) 1,5 \lg \eta_r}}{150 + 300c} \times 1000 \quad (2)$$

where

$\eta_r = \frac{\eta}{\eta_0} = \frac{t}{t_0}$ is the ratio of the viscosities (efflux times) of the solution and solvent;

t and t_0 are the efflux times, in seconds, of the solution and solvent, respectively;

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

Calculate the K-value for the sample as the mean value of the two individual K-values obtained in the two determinations, expressing the result to the first place of decimals. If the K-values obtained in the two determinations deviate by more than $\pm 0,4 \%$ from the mean value, these results shall be rejected and further determinations carried out with fresh test portions.

If the solution concentration is $5 \text{ g/cm}^3 \pm 0,005 \text{ g/cm}^3$, it is more convenient to read off the K-value from [Table 1](#), rounding to the second place of decimals.