SLOVENSKI STANDARD
SIST EN ISO 1628-2:2000

01-maj-2000

Polimerni materiali - Določanje viskoznosti polimerov v razredčenih raztopinah s kapilarnimi viskozimetri - 2. del: Polivinilklorid (ISO 1628-2:1998)

Plastics - Determination of the viscosity of polymers in dilute solution using capillary viscometers - Part 2: Poly(vinyl chloride) resins (ISO 1628-2:1998)

Kunststoffe - Bestimmung der Viskosität von Polymeren in verdünnter Lösung unter Verwendung von Kapillarviskosimetern - Teil 2: Vinylchlorid-Polymere (ISO 1628-2:1998)

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83.080.20 Plastomeri Thermoplastic materials

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English version

Plastics - Determination of the viscosity of polymers in dilute solution using capillary viscometers - Part 2: Poly(vinyl chloride) resins (ISO 1628-2:1998)

Kunststoffe - Bestimmung der Viskosität von Polymeren in verdünnter Lösung unter Verwendung von Kapillarviskosimetern - Teil 2: Vinylchlorid-Polymere (ISO 1628-2:1998)

This European Standard was approved by CEN on 13 December 1998.

CEN members are bound to comply with the CEN/CENELEC Internal Regulations which stipulate the conditions for giving this European Standard the status of a national standard without any alteration. Up-to-date lists and bibliographical references concerning such national standards may be obtained on application to the Central Secretariat or to any CEN member.

This European Standard exists in three official versions (English, French, German). A version in any other language made by translation under the responsibility of a CEN member into its own language and notified to the Central Secretariat has the same status as the official versions.

CEN members are the national standards bodies of Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and United Kingdom.

EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Central Secretariat: rue de Stassart, 36 B-1050 Brussels

Foreword

The text of the International Standard ISO 1628-2:1998 has been prepared by Technical Committee ISO/TC 61 "Plastics" in collaboration with Technical Committee CEN/TC 249 "Plastics", the secretariat of which is held by IBN.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by June 1999, and conflicting national standards shall be withdrawn at the latest by June 1999.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Czech Republic, Denmark, Finland, France, Germany, Greece, Iceland, Ireland, Italy, Luxembourg, Netherlands, Norway, Portugal, Spain, Sweden, Switzerland and the United Kingdom.

NOTE FROM CEN/CS: The foreword is susceptible to be amended on reception of the German language version. The confirmed or amended foreword, and when appropriate, the normative annex ZA for the references to international publications with their relevant European publications will be circulated with the German version.

Endorsement notice

The text of the International Standard ISO 1628-2:1998 was approved by CEN as a European Standard without any modification.

INTERNATIONAL STANDARD

ISO 1628-2

Second edition
1998-12-01

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)

Reference number
ISO 1628-2:1998(E)

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 1628-2 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee 9, *Thermoplastic materials*.

This second edition cancels and replaces the first edition (ISO 1628-2:1988) which has been modified to include:

— the determination of the K-value;

— a limit on the volatile-matter content of resins that can be tested using this part of ISO 1628;

— revised viscometer specifications;

— a reference viscometer;

— a precision statement.

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*

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— *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*

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

Part 2:
Poly(vinyl chloride) resins

1 Scope

This part of ISO 1628 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 part of ISO 1628 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.

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 may not have the same molecular mass.

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 part of ISO 1628 will only give values for reduced viscosity and *K*-value that are comparable when the concentrations of the solutions used are identical.

Limiting viscosity number is not used for PVC resins.

The experimental procedures described in this part of ISO 1628 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 may 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 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 1042:1998, *Laboratory glassware — One-mark volumetric flasks.*

ISO 1269:1980, *Plastics — Homopolymer and copolymer resins of vinyl chloride — Determination of volatile matter (including water).*

ISO 1628-1:1998, *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.*

3 Definitions

The terms used in this part of ISO 1628 are defined in ISO 1628-1:1998, clause 3, and, in particular, definitions 3.3.3 (reduced viscosity) and 3.3.6 (K-value).

4 Principle

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 $2{,}06 \times 10^{-6}$ m^2 s^{-1} and $2{,}33 \times 10^{-6}$ m^2 s^{-1} (2,06 mm^2 s^{-1} and 2,33 mm^2 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

The apparatus required to carry out viscosity measurements on polymers in dilute solution is described in ISO 1628-1:1998, clause 5. In addition, the following particular items are required for the procedures described in this part of ISO 1628:

6.1 Viscometer: From the viscometers described in subclause 5.1 of ISO 1628-1:1998, model 1C, with a capillary diameter of 0,77 mm ± 2 %, from table B.4 of ISO 3105:1994, 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.

6.2 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.

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

6.4 Mechanical agitator, equipped with a heating device to keep the flask (6.2) 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.

6.5 Analytical balance, accurate to 0,1 mg.

6.6 Temperature-regulated bath, capable of being set at 25,0 °C ± 0,5 °C in steps of 0,1 °C and maintaining a stability of ± 0,05 °C around the set temperature.

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

6.8 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 1628-1:1998, clause 6.

Prepare a solution with a concentration of 5 g/l ± 0,1 g/l at 25 °C ± 1 °C, as follows:

Weigh, to the nearest 0,2 mg, 0,250 g ± 0,005 g of resin and transfer it quantitatively to the 50 ml flask (6.2). 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 (6.4). 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 °C ± 1 °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 ± 0,1 %.

If a mass of 0,250 g ± 0,000 25 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 will require the amounts of solvent and test portion taken to be determined by experiment, and may 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 subclause 6.2 of ISO 1628-1:1998. 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.