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)

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*

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.*

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

Part 2:

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. (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 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.

9.2 Determination of efflux times

The procedure is described in ISO 1628-1:1998, clause 8.

The temperature of the thermostat (see 6.6) shall be set such that the actual temperature which is measured by the thermometer (6.7) lies in the range 25 °C ± 0,5 °C. The measured temperature shall be stable to ± 0,05 °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 6.3).

Particular care shall be taken over viscometer cleaning, which shall be based on the procedure described in ISO 1628-1:1998, 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 part of ISO 1628 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 1628-1:1998, clause 9, using the equation

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

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 grams per millilitre.

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 ± 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 g/l ± 0,005 g/l, it is more convenient to read off the values of I from table 1, expressing I in (m^3/kg) × 10^{-3}, i.e. ml/g, rounded to the first place of decimals.

10.2 *K*-value

For each test portion, calculate the K-value as specified in ISO 1628-1:1998, clause 9, using the equation

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

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 grams per millilitre.

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 ± 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 g/l ± 0,005 g/l, it is more convenient to read off the K-value from table 1, rounding to the second place of decimals.

11 Precision

Interlaboratory trials conducted on three resins in 11 laboratories on four different dates gave the following results for the repeatability standard deviation s_r (within the same laboratory) and the reproducibility standard deviation s_R (among different laboratories):

	K-value		
	approx. 50	approx. 70	approx. 90
s_r	0,132	0,115	0,120
s_R	0,420	0,291	0,495

	Reduced viscosity		
	approx. 61	approx. 124	approx. 227
s_r	0,313	0,458	0,742
s_R	0,984	1,202	3,042

12 Test report

The test report shall include the following information:

a) a reference to this part of ISO 1628;

b) all details necessary for complete identification of the material under test;

c) the reduced viscosity and/or K-value of the resin sample;

d) any difference between the type of viscometer used and the reference viscometer specified in this part of ISO 1628;

e) the date of the test.

Table 1 — Conversion of viscosity ratio (VR) to reduced viscosity (*I*) and *K*-value

Unit for reduced viscosity: $(m^3/kg) \times 10^{-3}$, i.e. ml/g
Concentration of resin in solution = 5 g/ml

VR	*I*	*K*	VR	*I*	*K*	VR	*I*	*K*
1,195	39,0	39,74	1,237	47,4	44,02	1,279	55,8	47,87
1,196	39,2	39,85	1,238	47,6	44,12	1,280	56,0	47,95
1,197	39,4	39,95	1,239	47,8	44,22	1,281	56,2	48,04
1,198	39,6	40,06	1,240	48,0	44,31	1,282	56,4	48,13
1,199	39,8	40,17	1,241	48,2	44,41	1,283	56,6	48,21
1,200	40,0	40,27	1,242	48,4	44,50	1,284	56,8	48,30
1,201	40,2	40,38	1,243	48,6	44,60	1,285	57,0	48,38
1,202	40,4	40,49	1,244	48,8	44,69	1,286	57,2	48,47
1,203	40,6	40,59	1,245	49,0	44,79	1,287	57,4	48,55
1,204	40,8	40,70	1,246	49,2	44,88	1,288	57,6	48,64
1,205	41,0	40,80	1,247	49,4	44,98	1,289	57,8	48,72
1,206	41,2	40,91	1,248	49,6	45,07	1,290	58,0	48,81
1,207	41,4	41,01	1,249	49,8	45,16	1,291	58,2	48,89
1,208	41,6	41,12	1,250	50,0	45,26	1,292	58,4	48,98
1,209	41,8	41,22	1,251	50,2	45,35	1,293	58,6	49,06
1,210	42,0	41,33	1,252	50,4	45,44	1,294	58,8	49,15
1,211	42,2	41,43	1,253	50,6	45,53	1,295	59,0	49,23
1,212	42,4	41,53	1,254	50,8	45,63	1,296	59,2	49,32
1,213	42,6	41,64	1,255	51,0	45,72	1,297	59,4	49,40
1,214	42,8	41,74	1,256	51,2	45,81	1,298	59,6	49,48
1,215	43,0	41,84	1,257	51,4	45,90	1,299	59,8	49,57
1,216	43,2	41,94	1,258	51,6	45,99	1,300	60,0	49,65
1,217	43,4	42,05	1,259	51,8	46,09	1,301	60,2	49,73
1,218	43,6	42,15	1,260	52,0	46,18	1,302	60,4	49,81
1,219	43,8	42,25	1,261	52,2	46,27	1,303	60,6	49,90
1,220	44,0	42,35	1,262	52,4	46,36	1,304	60,8	49,98
1,221	44,2	42,45	1,263	52,6	46,45	1,305	61,0	50,06
1,222	44,4	42,55	1,264	52,8	46,54	1,306	61,2	50,14
1,223	44,6	42,65	1,265	53,0	46,63	1,307	61,4	50,23
1,224	44,8	42,75	1,266	53,2	46,72	1,308	61,6	50,31
1,225	45,0	42,85	1,267	53,4	46,81	1,309	61,8	50,39
1,226	45,2	42,95	1,268	53,6	46,90	1,310	62,0	50,47
1,227	45,4	43,05	1,269	53,8	46,99	1,311	62,2	50,55
1,228	45,6	43,15	1,270	54,0	47,07	1,312	62,4	50,63
1,229	45,8	43,25	1,271	54,2	47,16	1,313	62,6	50,71
1,230	46,0	43,34	1,272	54,4	47,25	1,314	62,8	50,79
1,231	46,2	43,44	1,273	54,6	47,34	1,315	63,0	50,87
1,232	46,4	43,54	1,274	54,8	47,43	1,316	63,2	50,95
1,233	46,6	43,64	1,275	55,0	47,52	1,317	63,4	51,03
1,234	46,8	43,73	1,276	55,2	47,60	1,318	63,6	51,11
1,235	47,0	43,83	1,277	55,4	47,69	1,319	63,8	51,19
1,236	47,2	43,93	1,278	55,6	47,78	1,320	64,0	51,27

Table 1 (*continued*)

VR	*I*	*K*	VR	*I*	*K*	VR	*I*	*K*
1,321	64,2	51,35	1,371	74,2	55,14	1,421	84,2	58,59
1,322	64,4	51,43	1,372	74,4	55,21	1,422	84,4	58,65
1,323	34,6	51,51	1,373	74,6	55,28	1,423	84,6	58,72
1,324	34,8	51,59	1,374	74,8	55,35	1,424	84,8	58,79
1,325	65,0	51,67	1,375	75,0	55,42	1,425	85,0	58,85
1,326	65,2	51,75	1,376	75,2	55,49	1,426	85,2	58,92
1,327	65,4	51,83	1,377	75,4	55,57	1,427	85,4	58,98
1,328	65,6	51,91	1,378	75,6	55,64	1,428	85,6	59,05
1,329	65,8	51,98	1,379	75,8	55,71	1,429	85,8	59,11
1,330	66,0	52,06	1,380	76,0	55,78	1,430	86,0	59,18
1,331	66,2	52,14	1,381	76,2	55,85	1,431	86,2	59,24
1,332	66,4	52,22	1,382	76,4	55,92	1,432	86,4	59,31
1,333	66,6	52,29	1,383	76,6	55,99	1,433	86,6	59,37
1,334	66,8	52,37	1,384	76,8	56,06	1,434	86,8	59,44
1,335	67,0	52,45	1,385	77,0	56,13	1,435	87,0	59,50
1,336	67,2	52,53	1,386	77,2	56,20	1,436	87,2	59,57
1,337	67,4	52,60	1,387	77,4	56,27	1,437	87,4	59,63
1,338	67,6	52,68	1,388	77,6	56,34	1,438	87,6	59,70
1,339	67,8	52,76	1,389	77,8	56,41	1,439	87,8	59,76
1,340	68,0	52,83	1,390	78,0	56,48	1,440	88,0	59,82
1,341	68,2	52,91	1,391	78,2	56,55	1,441	88,2	59,89
1,342	68,4	52,99	1,392	78,4	56,62	1,442	88,4	59,95
1,343	68,6	53,06	1,393	78,6	56,69	1,443	88,6	60,02
1,344	68,8	53,14	1,394	78,8	56,76	1,444	88,8	60,08
1,345	69,0	53,21	1,395	79,0	56,83	1,445	89,0	60,14
1,346	69,2	53,29	1,396	79,2	56,90	1,446	89,2	60,21
1,347	69,4	53,37	1,397	79,4	56,97	1,447	89,4	60,27
1,348	69,6	53,44	1,398	79,6	57,04	1,448	89,6	60,33
1,349	69,8	53,52	1,399	79,8	57,11	1,449	89,8	60,40
1,350	70,0	53,59	1,400	80,0	57,17	1,450	90,0	60,46
1,351	70,2	53,67	1,401	80,2	57,24	1,451	90,2	60,52
1,352	70,4	53,74	1,402	80,4	57,31	1,452	90,4	60,59
1,353	70,6	53,82	1,403	80,6	57,38	1,453	90,6	60,65
1,354	70,8	53,89	1,404	80,8	57,45	1,454	90,8	60,71
1,355	71,0	53,96	1,405	81,0	57,51	1,455	91,0	60,78
1,356	71,2	54,04	1,406	81,2	57,58	1,456	91,2	60,84
1,357	71,4	54,11	1,407	81,4	57,65	1,457	91,4	60,90
1,358	71,6	54,19	1,408	81,6	57,72	1,458	91,6	60,96
1,359	71,8	54,26	1,409	81,8	57,79	1,459	91,8	61,03
1,360	72,0	54,33	1,410	82,0	57,85	1,460	92,0	61,09
1,361	72,2	54,41	1,411	82,2	57,92	1,461	92,2	61,15
1,362	72,4	54,48	1,412	82,4	57,99	1,462	92,4	61,21
1,363	72,6	54,55	1,413	82,6	58,05	1,463	92,6	61,27
1,364	72,8	54,63	1,414	82,8	58,12	1,464	92,8	61,34
1,365	73,0	54,70	1,415	83,0	58,19	1,465	93,0	61,40
1,366	73,2	54,77	1,416	83,2	58,26	1,466	93,2	61,46
1,367	73,4	54,85	1,417	83,4	58,32	1,467	93,4	61,52
1,368	73,6	54,92	1,418	83,6	58,39	1,468	93,6	61,58
1,369	73,8	54,99	1,419	83,8	58,45	1,469	93,8	61,64
1,370	74,0	55,06	1,420	84,0	58,52	1,470	94,0	61,70