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Thermoplastics piping systems for non-pressure applications — Unplasticized poly(vinyl chloride) (PVC-U) pipes and fittings — Determination of the viscosity number and K-value

Systèmes de canalisations thermoplastiques pour applications sans pression — Tubes et raccords en poly(chlorure de vinyle) non plastifié (PVC-U) — Détermination de l'indice de viscosité réduite et de la valeur K

Reference number
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Foreword

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The main task of technical committees is to prepare International Standards. 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.

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.

ISO 13229 was prepared by Technical Committee ISO/TC 138, *Plastics pipes, fittings and valves for the transport of fluids*, Subcommittee SC 1, *Plastics pipes and fittings for soil, waste and drainage (including land drainage)*.

Thermoplastics piping systems for non-pressure applications — Unplasticized poly(vinyl chloride) (PVC-U) pipes and fittings — Determination of the viscosity number and K-value

1 Scope

This International Standard specifies a method for the determination of the viscosity number (also known as reduced viscosity) and K-value of an unplasticized poly(vinyl chloride) (PVC) resin derived from a pipe, fitting or compound.

In this International Standard, only the method for isolation (or separation) of the PVC resin is detailed, while the determination of the viscosity number is given in ISO 1628-2.

The presence of other additives or polymers can invalidate this method (see Clause 3).

2 Normative references

The following referenced documents are indispensable for the application 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 1628-2, *Plastics — Determination of the viscosity of polymers in dilute solution using capillary viscometers — Part 2: Poly(vinyl chloride) resins*

3 Principle

A PVC resin, contained in a sample taken from a pipe, a fitting or a compound, is separated from most additives by dissolution in tetrahydrofuran (THF) and precipitation by methanol from a portion of the solution that has been isolated by centrifuging and decantation. The presence of additives in injection-moulding compounds can affect the results for materials for/from injection-moulded fittings.

If other polymers soluble in THF and insoluble in methanol (e.g. PMMA material) are present, this method shall not be used.

The precipitate is used for estimation of the viscosity number and K-value in accordance with ISO 1628-2.

4 Reagents for isolation or separation of the PVC resin

4.1 Tetrahydrofuran (THF), stabilized.

WARNING — It is very important for safety reasons that personal protective clothing be used when applying solvents to the test specimen. The use of solvents in regard to application of this International Standard may be further controlled under national and/or regional legislation. In particular, the THF used shall be collected, stored and sent to solvent recovery.

4.2 Methanol.

5 Apparatus for isolation or separation of the PVC resin

5.1 Glass container, of minimum capacity 100 ml.

5.2 Magnetic stirrer, with an adjustable speed of 0 r/min to 1 200 r/min.

5.3 Glass beaker, of capacity 600 ml, tall form.

5.4 Filter funnel.

5.5 Laboratory filter paper, capable of retaining polymer precipitated in accordance with 6.1.

5.6 Centrifuge with tubes, for 50 ml.

5.7 Vacuum dessicator.

5.8 Water bath, if necessary (see 6.1).

5.9 Pasteur pipette, if necessary (see 6.1).

6 Procedure

6.1 Isolation or separation of the PVC resin

Take approximately 2 g of PVC compound (2,5 g if the filler content is expected to be high), cut, if necessary, from the pipe or fitting tested.

Dissolve it in approximately 50 ml of THF in the glass container (5.1) by stirring. If the dissolution occurs slowly, warm carefully in a water bath (5.8).

The PVC resin shall be completely dissolved before continuing the procedure.

Transfer the solution to a tube of the centrifuge (5.6) and operate the centrifuge for approximately 40 min.

Decant that part of THF solution free of particles into the glass beaker (5.3), if necessary using a pasteur pipette (5.9), without entraining any filler.

In the beaker, precipitate the polymer by carefully adding methanol and stirring, until 10 parts of methanol have been added per part of THF solution.

Filter the suspension using a filter paper (5.5) and wash the precipitate with methanol. Transfer the precipitated polymer, but not the filter paper, to a bowl and dry at 50 °C for at least 12 h in the vacuum dessicator (5.7).

6.2 Determination of the viscosity number

Determine and record the viscosity number, in millilitres per gram, in accordance with ISO 1628-2 using a resin sample of (0,250 ± 0,000 25) g by dissolution in cyclohexanone.

7 Calculation of K-value

Calculate the K-value of the PVC resin using Equation (1):

$$K = \frac{1{,}5\lg\dfrac{t}{t_0} - 1 + \left[1 + \left(402 + 1{,}5\lg\dfrac{t}{t_0}\right)1{,}5\lg\dfrac{t}{t_0}\right]^{0{,}5}}{151{,}5} \times 1\ 000 \qquad (1)$$

where

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

t_0 is the efflux time of the solvent, in seconds.

Annex A gives the relation between the K-value and the viscosity number (reduced viscosity) for PVC resin.

8 Accuracy

The accuracy of the method for the determination of the K-value is ±2.

9 Test report

The test report shall include the following information:

a) a reference to this International Standard, i.e. ISO 13229:2010, and the referring standard;

b) complete identification of the pipe, fitting or compound tested;

c) the viscosity number;

d) the K-value;

e) any factor that could have affected the results, such as any incident or any operating details not specified in this International Standard;

f) the date of the test.

Annex A
(informative)

Relationship between K-value and viscosity number for PVC resin

For a PVC resin, the K-value according to Fikentscher[1] is calculated according to Equation (1).

For convenience, the K-values corresponding to a viscosity number from 60 ml/g to 178 ml/g for a solution in cyclohexanone containing 5 g resin/litre are given in Table A.1.

Table A.1 — Viscosity numbers and corresponding K-values

Viscosity number ml/g	K-value	Viscosity number ml/g	K-value	Viscosity number ml/g	K-value
60	49,6	100	63,5	140	73,8
62	50,5	102	64,1	142	74,3
64	51,3	104	64,7	144	74,7
66	52,1	106	65,2	146	75,1
68	52,8	108	65,8	148	75,6
70	53,6	110	66,3	150	76,0
72	54,3	112	66,9	152	76,5
74	55,1	114	67,4	154	76,9
76	55,8	116	67,9	156	77,3
78	56,5	118	68,5	158	77,7
80	57,2	120	69,0	160	78,1
82	57,9	122	69,5	162	78,5
84	58,5	124	70,0	164	78,9
86	59,2	126	70,5	166	79,3
88	59,8	128	71,0	168	79,7
90	60,5	130	71,5	170	80,1
92	61,1	132	71,9	172	80,5
94	61,7	134	72,4	174	80,9
96	62,3	136	72,9	176	81,3
98	62,9	138	73,3	178	81,7

Bibliography

[1] FIKENTSCHER, H. *Cellulosa chemie*, No. 13, 1932, pp. 58-64