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Standard Test Method for Dilute Solution Viscosity of Vinyl Chloride Polymers[1]

This standard is issued under the fixed designation D 1243; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—Reference to ISO 1628-2 was corrected editorially in March 2000.

1. Scope

1.1 This test method covers the determination of the dilute solution viscosity of vinyl chloride polymers in cyclohexanone. The viscosity is expressed in terms of inherent viscosity (logarithmic viscosity number). The test method is limited to those materials that give clear, uniform solutions at the test dilution.

NOTE 1—Other expressions for viscosity may be used as described in the Appendix, but any change from the test method as specified shall be stated in the report.

1.2 The values stated in SI units are to be regarded as the standard.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

NOTE 2—Although this test method and ISO 1628-2-1998 differ in approach or detail, data obtained by either are technically equivalent.

2. Referenced Documents

2.1 *ASTM Standards:*

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)[2]

D 883 Terminology Relating to Plastics[2]

D 1600 Terminology for Abbreviated Terms Relating to Plastics[3]

D 1755 Specification for Poly(Vinyl Chloride) Resins[3]

D 2857 Test Method for Dilute Solution Viscosity of Polymers[4]

E 77 Method for Verification and Calibration of Liquid-in-Glass Thermometers[5]

2.2 *ISO Standard:*

ISO 1628-2-1998 Determination of Viscosity Number and Limiting Viscosity Number—Part 2: Poly(Vinyl Chloride) Resins[6]

2.3 *National Institute of Standards and Technology Circular:*[7]

C-434 Testing of Glass Volumetric Apparatus

3. Terminology

3.1 *Definitions:* Definitions are in accordance with Terminology D 883 and Terminology D 1600, unless otherwise indicated.

4. Summary of Test Method

4.1 A sample of resin is dissolved in cyclohexanone to make a solution of specified concentration. Inherent viscosity (logarithmic viscosity number) is calculated from the measured flow times of the solvent and of the polymer solution.

NOTE 3—For additional information, refer to Test Method D 445 and Test Method D 2857 for Dilute Solution Viscosity of Polymers.[3]

5. Significance and Use

5.1 Dilute solution viscosity values for vinyl chloride polymers are related to the average molecular size of that portion of the polymer that dissolves in the solvent.

6. Apparatus

6.1 *Transfer Pipets*.

6.2 *Volumetric Flasks*, 100-mL, glass-stoppered, in accordance with National Institute of Standards and Technology Circular C-434.

6.3 *Viscometer*, Ubbelohde Series U-1 or Cannon-Ubbelohde No. 75.

[1] This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials (Section D20.15.07).

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This edition contains changes in Sections 1 and 2 to include an ISO equivalency statement.

[2] *Annual Book of ASTM Standards*, Vols 05.01 and 10.03.

[3] *Annual Book of ASTM Standards*, Vol 08.01.

[4] *Annual Book of ASTM Standards*, Vol 08.02.

[5] *Annual Book of ASTM Standards*, Vol 14.03.

[6] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

[7] Available from National Institute of Standards and Technology, U. S. Dept. of Commerce, Washington, DC 20234.

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6.4 *Water Bath*, at 30± 0.5°C, controlled to within ± 0.01°C.

6.5 *Timer*, as specified in Test Method D 445, graduated in divisions of 0.1 s or less.

6.6 *Filter Funnel*, fritted-glass.[8]

6.7 *Thermometer*, standard, in accordance with Method E 77.

7. Materials

7.1 *Solvent*—Cyclohexanone, analytical reagent grade or laboratory-distilled technical grade, boiling between 155 and 156°C at 760 mm Hg has been found acceptable if stored in a closed container.

8. Procedure

8.1 Dissolve duplicates of resin as follows: Weigh 0.2± 0.002 g of the sample (moisture content below 0.1 %) and transfer it to a 100-mL glass-stoppered volumetric flask. Take care to transfer all of the weighed resin into the flask. As an alternative method, the resin (0.2± 0.002 g) may be weighed directly into a tared, 100-mL glass-stoppered volumetric flask.

8.2 Add 50 to 70 mL of cyclohexanone to the flask, taking care to wet the resin so that lumps do not form.

8.3 Heat the flask at 85 ± 10°C until the resin is dissolved. Occasional shaking will reduce the time required for solution. Heating should not exceed 12 h and should preferably be less to minimize degradation. If any gel-like particles can be seen, prepare a new solution.

8.4 Cool the solution to the test temperature by immersing flask in the 30°C bath for a minimum time of 30 min and adjust to a solution volume of 100 mL. Filter through a fritted-glass filter directly into the viscometer.

8.5 Measure at 30°C the flow time of the prepared solution (8.4) and of the pure solvent (aged at 85 ± 10°C) in the viscometer. Allow 10 min for the viscometer to come to temperature equilibrium after placing it in the water bath. The flow time of the solution or the solvent should be within 0.1 % on repeat runs on the same filling.

NOTE 4—Keep the Ubbelohde viscometer clean when not in use. Acetone may be used as a cleaning flush. The viscometer may be stored filled with pure solvent or it may be stored dry.

[8] Filters may be obtained from Corning Glass, No. 36060 "Coarse" type.

9. Calculation

9.1 Calculate the relative and inherent viscosity (viscosity ratio and logarithmic viscosity number) as follows:

$$\eta_{rel} = t/t_o$$

$$\eta_{inh} = (\ln \eta_{rel})/C$$

where:

η_{rel}	=	relative viscosity (viscosity ratio),
t	=	efflux time of the solution,
t_o	=	efflux time of the pure solvent,
C	=	weight of sample used (8.1) per 100 mL of solution,
η_{inh}	=	inherent viscosity (logarithmic viscosity number), and
$ln\ \eta_{rel}$	=	natural logarithm of relative viscosity (viscosity ratio).

10. Report

10.1 Report the average inherent viscosity of two analyses to the nearest 0.01.

11. Precision and Bias [9]

11.1 An interlaboratory test program utilizing this test method was carried out in 1973 involving seven laboratories, each performing pairs of determinations on one polymer.

NOTE 5—See Specification D 1755, Table 3, for inherent viscosity value of ASTM PVC Reference Standard No. 1.

11.2 *Precision*—The following values of precision have been calculated from the interlaboratory test program at a 95 % confidence level:

Within-laboratory precision (within one pair of values)	1.4 % of mean
Between-laboratories precision (between averages of pairs)	2.2 % of mean

11.3 *Bias*—No justifiable statement of bias can be made for this test method, since the true value of the property cannot be established by an accepted referee method.

12. Keywords

12.1 dilute solution viscosity; inherent viscosity; intrinsic viscosity; relative viscosity; specific viscosity; test method; vinyl chloride polymers

[9] Supporting data are available from ASTM Headquarters. Request RR: D20-1112.

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