Designation: D1243 – 14

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

This standard is issued under the fixed designation D1243; 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.

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—This standard and ISO 1628-2 address the same subject matter, but differ in technical content.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers

D883 Terminology Relating to Plastics

D1600 Terminology for Abbreviated Terms Relating to Plastics

D1755 Specification for Poly(Vinyl Chloride) Resins

D2857 Practice for Dilute Solution Viscosity of Polymers

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

2.2 *ISO Standard:*

ISO 1628-2 Determination of Viscosity Number and Limiting Viscosity Number—Part 2: Poly(Vinyl Chloride) Resins[3]

2.3 *National Institute of Standards and Technology Circular:*[4]

C-434 Testing of Glass Volumetric Apparatus

3. Terminology

3.1 *Definitions*—Definitions are in accordance with Terminology D883 and Terminology D1600, 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 D445 and Test Method D2857 for Dilute Solution Viscosity of Polymers.

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,* ASTM Ubbelohde Size 1 or Cannon-Ubbelohde No. 75.

NOTE 4—ASTM Ubbelohde Size 1 is a commonly used name for a specific viscometer type, which is neither supplied nor endorsed by ASTM International.

NOTE 5—Operating instructions can be found in Specification D446.

[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.15.07).

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

[4] Available from National Institute of Standards and Technology (NIST), 100 Bureau Dr., Stop 1070, Gaithersburg, MD 20899-1070, http://www.nist.gov.

*A Summary of Changes section appears at the end of this standard

6.4 *Water Bath,* set at 30.0 ± 0.5°C, controlled to within ± 0.01°C.

6.4.1 The temperature of the bath medium shall not vary by more than ±0.02°C of the selected temperature in accordance with Test Method D445.

6.5 *Timer,* as specified in Test Method D445, graduated in divisions of 0.1 s or less.

6.6 *Filter Funnel,* fritted-glass.[5]

6.7 *Thermometer,* standard, in accordance with Method E2251.

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, weigh the resin (0.2 ± 0.002 g) 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. Take care that heating time does not exceed 12 h, preferably 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 30.0 ± 0.5°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. If the flow time of the solution or the solvent differs by more than 0.1 % on repeat runs on the same filling, the result is suspect.

NOTE 6—Keep the Ubbelohde viscometer clean when not in use. Acetone may be used to flush the pure solvent (Cyclohexanone) and enable subsequent drying. The viscometer may be stored filled with pure solvent or it may be stored dry.

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[6]

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.

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 analyses)	1.4 % of mean
Between-laboratories precision (between averages of analyses)	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

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

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