Designation: D1243 – 15

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

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—Operating instructions can be found in Specification D446.

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

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[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).

Current edition approved Oct. 1, 2015. Published October 2015. Originally approved in 1952. Last previous edition approved in 2014 as D1243 - 14. DOI: 10.1520/D1243-15.

[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**

6.4.1 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,* funnel, Hirsh-Type; borosilicate glass; w/coarse fritted disc, pore size: 40–60 µm.

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, as described in 6.6, 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 5—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:

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

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

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[5]

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

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

APPENDIX

(Nonmandatory Information)

X1. OTHER EXPRESSIONS FOR VISCOSITY

X1.1 Definitions

X1.1.1 *relative viscosity*—ratio of the flow time of a specified solution of the polymer to the flow time of the pure solvent. The International Union of Pure and Applied Chemistry (IUPAC) term for relative viscosity is viscosity ratio.

X1.1.2 *specific viscosity*—relative viscosity minus one. Specific viscosity represents the increase in viscosity that may be attributed to the polymeric solute.

X1.1.3 *reduced viscosity*—ratio of the specific viscosity to the concentration. Reduced viscosity is a measure of the specific capacity of the polymer to increase the relative viscosity. The IUPAC term for reduced viscosity is viscosity number.

X1.1.4 *inherent viscosity*—ratio of the natural logarithm of the relative viscosity to the concentration. The IUPAC term for inherent viscosity is logarithmic viscosity number.

X1.1.5 *intrinsic viscosity*—limit of the reduced and inherent viscosities as the concentration of the polymeric solute approaches zero and represents the capacity of the polymer to increase viscosity. Interactions between solvent and polymer molecules have the affect of yielding different intrinsic viscosities for the same polymer in various solvents. The IUPAC term for intrinsic viscosity is limiting viscosity number.

X1.2 Determination of Intrinsic Viscosity (Limiting Viscosity Number)

X1.2.1 To determine the intrinsic viscosity (limiting viscosity number) of a polymer from dilute solution viscosity data, the reduced and inherent viscosities (viscosity and logarithmic viscosity number) of solutions of various concentrations of the polymer are determined at constant temperature and these values are then plotted against the respective concentrations. The two lines thus obtained converge to a point of zero concentration of the solute which represents the intrinsic viscosity (limiting viscosity number) of the polymer in that solvent at the temperature of the determination. Fig. X1.1 illustrates this convergence.

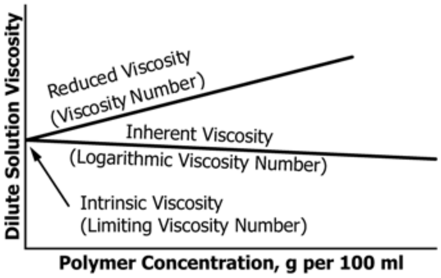

FIG. X1.1 Example of Plot to Determine Intrinsic Viscosity.

X1.2.2 At higher concentrations the viscosity curves may deviate from linearity; therefore, the greatest accuracy is obtained at less than 0.5 g/dl (0.005 g/mL) of solution. Since extrapolation of either reduced viscosity or inherent viscosity (viscosity or logarithmic viscosity number) curves to infinite dilution will give the same value for intrinsic viscosity (limiting viscosity number), a plot of either type of viscosity will permit the calculation of valid intrinsic viscosity (limiting viscosity number) data.

X1.3 Estimation of Intrinsic Viscosity (Limiting Viscosity Number)

X1.3.1 The mathematical method of Billmeyer (**1**)[6] permits a good approximation of intrinsic viscosity (limiting viscosity number). This method makes use of equations derived from the power series expansion of viscosity versus concentration. Neglecting the higher order terms, equations may be written which can be used to estimate intrinsic viscosity (limiting viscosity number). The following equation has been found suitable for poly(vinyl chloride) resins:

$$\eta_{iv} = \frac{1}{4}\left[(\eta_{rel} - 1)/C\right] + \frac{3}{4}\left[\ln(\eta_{rel})/C\right]$$

where C = concentration of polymer, g/100 mL.

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[6] The boldface numbers in parentheses refer to the list of references at the end of this test method.

REFERENCES

(1) Billmeyer, F. W., Jr., *Journal of Polymer Science*, Vol 4, 1949, p. 83.

(2) Cragg, L. H., and Fern, C. R. H., *Journal of Polymer Science*, Vol 10, 1953, p. 185.

(3) Huggins, M. L., *Journal of the American Chemical Society*, Vol 64, 1942, p. 2716.

(4) International Union of Pure and Applied Chemistry, *Journal of Polymer Science*, Vol 8, 1952, p. 269.

(5) Streeter, D. J., and Boyer, R. F., *Industrial and Engineering Chemistry*, Vol 43, 1951, p. 1790.

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D1243 - 14) that may impact the use of this standard. (October 1, 2015)

(1) Reworded 6.6, added pore size of 40 – 60µm.

(2) Removed old Footnote 5.

(3) Reworded 8.4, added reference to 6.6.

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