



Designation: D 1601 – 99

Standard Test Method for Dilute Solution Viscosity of Ethylene Polymers¹

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1. Scope *

1.1 This test method covers the determination of the dilute solution viscosity of ethylene polymers at 135°C. It is applicable to a reasonably wide spectrum of ethylene polymers having densities from 0.910 to 0.970 g/cm³. Directions are given for the determination of relative viscosity (viscosity ratio), inherent viscosity (logarithmic viscosity number), and intrinsic viscosity (limiting viscosity number) (see Appendix X4).

1.2 The values as 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 1—This test method is technically equivalent to ISO 1628-3.

NOTE 2—Appendix X1-Appendix X3 contain material from the previous version of Test Method D 1601 and are included for information only.

2. Referenced Documents

2.1 ASTM Standards:

D 86 Method for Distillation of Petroleum Products at Atmospheric Pressure²

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids²

D 883 Terminology Relating to Plastics³

D 1600 Terminology for Abbreviated Terms Relating to Plastics³

D 4020 Specification for Ultra-High-Molecular Weight Polyethylene Molding and Extrusion Materials⁴

E 1 Specification for ASTM Thermometers⁵

IEEE/ASTM SI-10 Standard for Use of the International System of Units (SI): The Modern Metric System⁶

¹ This test method is under the jurisdiction of ASTM Committee D-20 on Plastics and is the direct responsibility of Subcommittee D20.70 on Analytical Methods (Section D20.70.05).

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² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 08.01.

⁴ Annual Book of ASTM Standards, Vol 08.03.

⁵ Annual Book of ASTM Standards, Vol 14.03.

⁶ Annual Book of ASTM Standards, Vol 14.04.

2.2 ISO Standards:⁷

ISO 1628-3 Plastics—Determination of Viscosity Number and Limiting Viscosity Number—Part 3: Polyethylenes and Polypropylenes

3. Terminology

3.1 Units, symbols, and abbreviations used in this test method are those recommended in IEEE/ASTM SI 10 and Terminologies D 883 and D 1600 (see also Appendix X4).

4. Significance and Use

4.1 The knowledge of dilute solution viscosity serves as an additional tool in characterizing ethylene polymers. Viscosity data alone may be of limited value in predicting the processing behavior of the polymer. However, when used in conjunction with other flow and physical property values, the solution viscosity of ethylene polymers may contribute to their characterizations.

4.2 Satisfactory correlation between solution viscosity and certain other properties is possible from polymers of a single manufacturing process. The solution viscosity test is not sensitive to some molecular configurational patterns that may occur among polymers from different manufacturing processes. Hence, its correlation with other properties of polymers produced by different processes, by even one manufacturer, may be limited.

4.3 The viscosity of polymer solutions may be drastically affected by the presence of known or unknown additives in the sample. The use of solution viscosity may be of questionable value where ethylene polymers are known or suspected to contain colorants, carbon black, low molecular weight hydrocarbons, fillers, or other additives.

4.4 The measurement of dilute solution viscosity of ethylene polymers presents problems not ordinarily encountered in viscosimetry. Ethylene polymers are not soluble at room temperature in any known solvent. Some of the higher density materials are insoluble below 100°C. Extreme care must be exercised in transferring the solution to the viscometer for the test if the correct solution concentration is to be maintained. This test has no significance unless the sample is completely soluble.

⁷ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

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

4.5 The solution viscosity is a function of the root-mean-square size of the polymer molecules in solution. It is known that the solvent selected and the temperature of the determination have an effect on the root-mean-square size of the particles. Hence, where a viscometer, solvent, or temperature other than specified is used, data may not be comparable to that obtained by this procedure.

5. Apparatus

5.1 *Volumetric Flasks*, 100-mL, grade EXAX or better.⁸

5.2 *Transfer Pipets*, Grade EXAX or better.⁸

5.3 *Constant Temperature Bath*, capable of maintaining $135 \pm 0.1^\circ\text{C}$.

5.4 *Viscometer*, Ubbelohde No. 1, calibrated for kinetic energy correction constant.

NOTE 3—Other types of viscometers may be used provided they can be shown to agree with the type specified.

5.5 *Oven*, maintained at $140 \pm 5^\circ\text{C}$.

5.6 *Timer*, as specified in 4.5 of Test Method D 445.

5.7 *Thermometer*—An ASTM High Softening Point Thermometer having a range from 30 to 200°C , and conforming to the requirements for Thermometer 16C in Specification E 1.

6. Reagents and Materials

6.1 *Solvent—Decahydronaphthalene*, practical grade, purified and redistilled, as follows:

6.1.1 The solvent shall be purified by percolation through 100 to 200 mesh commercial grade silica gel. This treatment removes naphthalene, tetrahydronaphthalene, and oxy compounds, particularly peroxides.

6.1.2 The redistilled product shall conform to the following requirements when tested in accordance with Method D 86:

Standard Distillation	ASTM Method D 86
Initial boiling point	190°C min
10 mL	191°C min
20 mL	192°C min
80 mL	194°C max
90 mL	195°C max
Dry point	196°C max

NOTE 4—While use of other solvents, such as tetrahydronaphthalene or xylene, may sometimes be advantageous, they will generally yield different values for solution viscosities.

6.1.3 Immediately after redistillation of the decahydronaphthalene, add 0.2 % Tetrakis [methylene 3-(3',5'-di-tert-butyl-4'-hydroxyphenyl) propionate] methane to inhibit oxidation during the viscosity determination.

6.2 *Heat Transfer Medium*—Any liquid heat transfer medium that will not appreciably affect the accuracy of the test may be used. Care should be exercised in using fluids that discolor or smoke with prolonged heating.

NOTE 5—Silicone heat-transfer fluids are adequate for this use⁹.

7. Procedure

7.1 *Decahydronaphthalene Preparation*—Add 0.2 % weight/volume stabilizer (Irganox 1010 or equivalent), to the decahydronaphthalene, stir to dissolve, filter through fast filter paper, and store in a clean amber bottle.

7.2 *Cleaning the Viscometer*—Clean the viscometer thoroughly as follows: fill with cleaning solution, let stand overnight, empty and rinse several times with distilled water, then rinse with acetone and purge with dry nitrogen.

7.3 *Solution Preparation*—Weigh 20 to 30 mg of the UHMWPE (see Note 6) into the Erlenmeyer flask. Add the decahydronaphthalene at room temperature, using a burette to measure in milliliters a volume equal to 4.5 times the UHMWPE weight in milligrams (for example, 26 mg of UHMWPE and 117 ml of decahydronaphthalene). Heat the flask, with stirring, to $150^\circ\text{C} \pm 2^\circ\text{C}$. Continue stirring at $150^\circ\text{C} \pm 2^\circ\text{C}$ for 1 h, with the flask lightly stoppered. Examine the flask to see if undissolved UHMWPE remains. If so, heat an additional 15 min and reexamine. Heat for an additional 15 min, if necessary, but do not heat longer than 2 h total.

7.4 Viscosity Measurement:

7.4.1 Place the clean viscometer into the constant-temperature bath. Fill the viscometer with decahydronaphthalene, and allow it and the solvent to equilibrate at $135 \pm 0.1^\circ\text{C}$. Measure the flow time of the solvent, t_0 , three times, and average the results. The viscometer may have to be cleaned if replicate measurements of flow time differ by more than 0.3 s (see 7.4.3). Remove the decahydronaphthalene from the viscometer with vacuum and thoroughly rinse the viscometer with xylene at 110 to 120°C or decahydronaphthalene at 135°C . Remove the xylene or decahydronaphthalene with vacuum and aspirate dry air or nitrogen to dry the viscometer (2 or 3 min). It is essential that the viscometer be completely dry.

7.4.2 Transfer the hot (150°C) polymer solution to the viscometer with sufficient quantity to fill to the mark (see Note 7), allow to equilibrate at $135^\circ\text{C} \pm 0.1^\circ\text{C}$, and measure the flow time of the solution, t_s , in triplicate, and average the results.

7.4.3 Between uses, clean the viscometer by rinsing with hot xylene or decahydronaphthalene, drying with air or nitrogen, and storing filled with hot decahydronaphthalene. If solvent flow time changes by more than 0.3 s, or if difficulty is encountered in obtaining reproducible flow times with any solution or solvent, clean the viscometer with cleaning solution as described in 7.2.

NOTE 6—If the UHMWPE is suspected to contain moisture or other volatiles, it shall first be dried in a vacuum oven at 60°C for 2 h.

NOTE 7—Filling of the viscometer is made easier by the use of a glass funnel warmed with a heating mantle. This helps to prevent the UHMWPE solution from precipitating.

⁸ Glassware used in this test method should be tested in accordance with the procedures described in the National Institute of Standards and Technology Circular No. C-434, "Testing of Glass Volumetric Apparatus," and should not exceed the limits of accuracy set forth in the circular.

⁹ The silicone fluids available from the Dow Corning Corp., Midland, MI, or from the Union Carbide Corp., Linde Silicones Div., New York, NY, have been found satisfactory for this purpose.