



Designation: D4603 – 18

Standard Test Method for Determining Inherent Viscosity of Poly(Ethylene Terephthalate) (PET) by Glass Capillary Viscometer¹

This standard is issued under the fixed designation D4603; 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 reappraisal. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope*

1.1 This test method is for the determination of the inherent viscosity of poly(ethylene terephthalate) (PET) soluble at 0.50 % concentration in a 60/40 phenol/1,1,2,2-tetrachloroethane solution by means of a glass capillary viscometer. Highly crystalline forms of PET that are not soluble in this solvent mixture will require a different procedure.

1.2 The inherent viscosity values obtained by this test method are comparable with those obtained using differential viscometry described in Test Method [D5225](#).

1.3 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.4 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use. Specific hazards statements are given in Section 8.

1.4.1 *Warning—Mercury has been designated by many regulatory agencies as a hazardous material that can cause serious medical issues. Mercury, or its vapor, has been demonstrated to be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury containing products. See the applicable product Safety Data Sheet (SDS) for additional information. Users should be aware that selling mercury and/or mercury containing products into your state or country may be prohibited by law*

NOTE 1—This standard and ISO 1628-5 address the same subject matter, but differ in technical content. This ISO standard gives an option of solvents for PET. Solvent specified in this ASTM test method is one of the options in the ISO method. ISO also uses Type 1C Ubbelohde viscometer, rather than the 1B, and reports viscosity number, rather than inherent viscosity.

¹ This test method is under the jurisdiction of ASTM Committee [D20](#) on Plastics and is the direct responsibility of Subcommittee [D20.70](#) on Analytical Methods (D20.70.05).

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1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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](#)

[D1972 Practice for Generic Marking of Plastic Products \(Withdrawn 2014\)](#)³

[D5225 Test Method for Measuring Solution Viscosity of Polymers with a Differential Viscometer](#)

[E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids](#)

[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

[E2935 Practice for Conducting Equivalence Testing in Laboratory Applications](#)

[IEEE/ASTM SI-10 Practice for Use of the International System of Units \(SI\)](#)

2.2 *ISO Standard:*

[ISO 1628-5 Determination of the Viscosity of Polymers in Dilute Solution Using Capillary Viscometers—Part 5: Thermoplastic Polyester \(TP\) homopolymers and Copolymers.](#)⁴

2.3 *NIST Standard:*

[C 602 Testing of Glass Volumetric Apparatus](#)⁵

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 The last approved version of this historical standard is referenced on www.astm.org.

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

⁵ 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

3. Terminology

3.1 Units, symbols, and abbreviations used in this test method are those recommended in Practice [IEEE/ASTM SI-10](#).

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *inherent viscosity* (also known as the logarithmic viscosity number)—defined by the equation given in [11.1](#).

3.2.2 *PET*—as outlined in Practice [D1972](#). The PET acronym may be used to avoid trademark infringement and to comply with various state or federal laws.

4. Summary of Test Method

4.1 The inherent viscosity is determined by measuring the flow time of a solution of known polymer concentration and the flow time of the pure solvent in a capillary viscometer at a fixed temperature. The inherent viscosity value is calculated from the flow time values.

5. Significance and Use

5.1 The inherent viscosity is relatable to the composition and molecular weight of a polyester resin. It must be controlled so that the processability and end properties of the resin remain in a desired range.

6. Apparatus

6.1 *Cannon Ubbelohde Type 1B Viscometer*, as described in Specifications and Operating Instructions [D446](#).

6.2 *Viscometer Holder*.

6.3 *Electric Timer*, readable to 0.1 s, as described in Test Method [D445](#).

6.4 *Constant Temperature Bath*, control label at 30°C (86°F) ± 0.01°C (0.02°F).

6.5 *Kinematic Viscosity Thermometer ASTM 118* (for use at 30°C), conforming to Specification [E2251](#). Thermometric devices such as liquid-in-glass thermometers, resistance temperature detectors, thermistors and thermocouples with equal or better accuracies within the temperature range involved, may be used.

6.6 *Temperature Controllable Magnetic Stirring Hot Plate*.

6.7 *TFE-Fluorocarbon Plastic-Coated Stirring Bars and a Magnetic Bar Retriever*.

6.8 *Volumetric Flasks and Stoppers*, 50-mL capacity, conforming to the standards of accuracy in NIST Circular No. C 602.

6.9 *Analytical Balance*, with readout to 0.0001 g.

6.10 *Borosilicate Funnels*.

6.11 *Stainless Steel Filter Screening*, 325-mesh or finer.

6.12 *Aspirator*.

6.13 *Wiley Mill Grinder*, with 20-mesh stainless steel screen.

6.14 *Drying Oven*, for equipment.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available.⁶ Other grades are permitted to be used, providing it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Phenol/1,1,2,2-tetrachloroethane Solution*, 60/40 weight % mixture (0.5 % moisture maximum, protected in such a manner as to maintain this maximum level). Protect PET against the degradation which has been found to occasionally occur during the heating stage in [10.3](#) through the addition of a hydrogen chloride scavenger to the solvent, such as 0.2 weight % *n*-octyl-bis-(isoethyl mercaptoacetate).⁷

7.3 *Reagent Grade Methylene Chloride and Acetone*, rinsing solvents.

7.4 *Chromic Acid*, cleaning solution.

8. Hazards

8.1 The solvent used in this procedure is a mixture of 60 weight % phenol and 40 weight % 1,1,2,2-tetrachloroethane. Both compounds and the mixture are toxic and require care in handling. Make reference to the material safety data sheets available from the suppliers of these compounds for dealing with the hazards they present. In addition to using a hood for adequate ventilation in handling these materials, protection against skin contact is essential.

8.2 Obtain the material safety data sheets for methylene chloride, acetone, and the chromic acid solution used for cleaning the testing equipment from their suppliers. Consult the material safety data sheets before using the materials.

9. Conditioning

9.1 If the sample of PET contains 0.5 % or more of inert material such as titanium dioxide or glass fiber, determine the amount of inert material accurately by a procedure suitable for the type of inert material present.

9.2 If the sample is suspected of being wet (in excess of the moisture level derived from exposure to ambient humidity conditions), dry the sample in an oven for a suggested minimum of 2 h at 65°C (149°F) ± 5°C (9°F) or until a constant weight of ± 0.1 % is reached. Moisture picked up from ambient humidity will weigh 0.30 % and is negligible in this procedure. (See [Note 2](#).)

NOTE 2—Use a suitable technical method to determine moisture content.

⁶ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

⁷ Available from M&T Chemicals, Inc., P. O. Box 1104, Rahway, NJ 07065.