

Designation: $\frac{D4603 - 03}{E}$ (Reapproved 2011)^{£1} 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 reapproval. A superscript epsilon (\$\epsilon\$) indicates an editorial change since the last revision or reapproval.

ε¹ NOTE—Reapproved with editorial change to 6.5 in November 2011.

1. Scope-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 and health practices and determine the applicability of regulatory limitations prior to use. Specific hazards statements are given in Section 8.
- 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 test method-standard and ISO 1628-5 are similar but not technically equivalent, 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.
- 1.5 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

E1E2251 Specification for ASTM Liquid-in-Glass Thermometers Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

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

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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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.



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 Apparatus⁵

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 E1E2251. 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. 8a-3e1e-47e4-a6dd-de5e5a11899 [astm-d4603-18]
 - 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.

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.

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.