Designation: D4603 – 03 (Reapproved 2011)ε1

Standard Test Method for Determining Inherent Viscosity of Poly(Ethylene Terephthalate) (PET) by Glass Capillary Viscometer[1]

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 (ε) indicates an editorial change since the last revision or reapproval.

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

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

NOTE 1—This test method and ISO 1628-5 are similar but not technically equivalent. 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.

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

D5225 Test Method for Measuring Solution Viscosity of Polymers with a Differential Viscometer

E1 Specification for ASTM Liquid-in-Glass Thermometers

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

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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.[3]

2.3 *NIST Standard:*

C 602 Testing of Glass Volumetric Apparatus[4]

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.

[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] 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

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 E1. 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.[5] 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).[6]

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.

9.3 If the sample is difficult to dissolve, reduce the sample size by grinding it to a 20-mesh screen size in a Wiley Mill or other size reduction technique. Avoid overheating the sample during the grinding operation. It is possible to maintain a low temperature by grinding in the presence of dry ice or liquid nitrogen. Grind a 15 to 20- g sample, representative of the entire lot being tested. It is likely that drying is necessary after the dry ice grinding step.

10. Procedure

10.1 Accurately weigh between 0.2475 and 0.2525 g (accurate to ±0.0002 g) of sample into a clean, dry 50-mL volumetric flask. If the sample contains more than 0.5 % inert material, weigh the amount of sample necessary to give the specified amount of PET.

10.2 Place a TFE-fluorocarbon plastic-coated stirring bar into the flask and add approximately 25 mL of solvent. Prepare one flask without any sample present. Cap the flasks.

10.3 Place the flasks in steel beakers and place on a magnetic hot plate which has been preheated to 110°C (230°F)

[5] *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.

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