Designation: D789 – 19

Standard Test Method for Determination of Relative Viscosity of Concentrated Polyamide (PA) Solutions[1]

This standard is issued under the fixed designation D789; 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.

1. Scope*

1.1 This test method covers the determination of relative viscosity as it applies to concentrated solutions of polyamide (PA).

1.2 This test method does not address measures of viscosity derived from measurements with dilute solutions.

1.3 The values stated in SI units are to be regarded as standard. The values given in brackets 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.*

NOTE 1—This standard and ISO 307 address the same subject, buy the technical content is different.

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]

D446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers

D883 Terminology Relating to Plastics

D2162 Practice for Basic Calibration of Master Viscometers and Viscosity Oil Standards

D6779 Classification System for and Basis of Specification for Polyamide Molding and Extrusion Materials (PA)

E1953 Practice for Description of Thermal Analysis and Rheology Apparatus

E2975 Test Method for Calibration or Calibration Verification of Concentric Cylinder Rotational Viscometers

2.2 *ISO Standards:*[3]

ISO 307 Determination of Viscosity Number of Polyamides in Dilute Solutions

ISO 16396-1 Plastics—Polyamide (PA) moulding and extrusion materials—Part 1: Designation system, marking of products and basis for specifications

ISO 17025 General Requirements for the Competence of Testing and Calibration Laboratories

3. Terminology

3.1 *Definitions*—The definitions used in these test methods are in accordance with Terminology D883.

4. Significance and Use

4.1 These test methods are intended for use as control and acceptance tests. They are also applicable in the partial evaluation of materials for specific end uses and as a means for detecting changes in materials due to specific deteriorating causes.

4.2 The steps involved in running this method are:

4.2.1 Calibration of the viscometers,

4.2.2 Preparation of solutions,

4.2.3 Determination of efflux time,

4.2.4 Calculation of relative viscosity (which requires the following),

4.2.4.1 Determining the density of the polymer/formic acid solution, and

4.2.4.2 Determining the absolute viscosity of the formic acid used.

4.3 Solvents used to prepare concentrated solutions for use in this test method are formic acid (9.2.6.1) and m-cresol (9.2.6.2).

4.4 Solvents used to prepare dilute solutions of various polyamides are designated in ASTM D6779, ISO 16396, and ISO 307. These include:

[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 (Section D20.15.09).

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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.

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

4.4.1 *Formic Acid*—PA 6, PA 46, PA 66, PA 69, PA 610, PA MXD6 and corresponding copolyamides

4.4.2 *Sulfuric Acid*—PA 6, PA 46, PA 66, PA 69, PA 610, PA 612, PA MXD6 and corresponding copolyamides

4.4.3 *m-cresol*—PA 612, PA1010, PA1012, PA 11, PA 12, PA1212, PA 11/12 copolymers, PA 6T/66, PA 6I/66, PA 6I/6T, PA 6T/6I/66, PA 6T/6I, PA 6I/6T/66

4.4.4 *Phenol/1,1,2,2-tetrachloroethane (where legal)*—PA 6T/66, PA 6I/66, PA 6I/6T, PA 6T/6I/66, PA 6T/6I, PA 6I/6T/66, PA 6T/66, PA 6I/66, PA 6I/6T, PA 6T/6I/66, PA 6T/6I, PA 10T PA 6I/6T/66, PPA and copolyamides

5. Test Specimen

5.1 Test specimens for the various tests shall conform to the requirements prescribed herein.

6. Number of Tests

6.1 One determination shall be considered sufficient for testing each molding powder batch or resin lot. Table 1 gives repeatability and reproducibility statistics for relative viscosity testing.

7. Sampling

7.1 The material shall be sampled statistically or the sample shall come from a process that is in statistical control.

7.2 Samples in many forms, such as molded powder, molded shapes, or re-grind are permitted. It is recommended that molded specimens be cut into smaller parts prior to testing.

8. Conditioning

8.1 *Test Conditions*—Do not remove samples from sealed, airtight containers until ready for testing.

TEST METHOD

9. Relative Viscosity

9.1 *General*—Determine the relative viscosity of the polyamide polymer by ASTM Ubbelohde (Suspended-Level)-type viscometer. The ASTM Ubbelohde-type viscometer is the reference and referee method. Ostwald-type viscometers, pipet viscometer, and rotational viscometer are acceptable as an alternative method.

9.2 *ASTM Ubbelohde (Suspended Level)-type Viscometer*—To determine the viscosity of formic acid use an ASTM Ubbelohde viscometer Size 1 with an inside diameter of 0.58 mm ± 2 %. For use to determine the viscosity of the polyamide solutions use the appropriate ASTM Ubbelohde viscometer as defined in Specification D446, Fig. A2.1 for the polyamide viscosity range.

9.2.1 *Apparatus:*

9.2.1.1 *Constant-Temperature Liquid Bath,* set to operate at 25 ± 0.1°C.

9.2.1.2 *Precision Thermometer,* calibrated, for use in the liquid bath (ASTMS45C (non-mercury), and ASTM 45C (mercury-filled)). (**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.)

9.2.1.3 *Ubbelohde (Suspended Level)-type Viscometer),* calibrated, manufactured from low-expansion borosilicate glass.

9.2.1.4 *Ostwald-type Viscometer,* calibrated, manufactured from low-expansion borosilicate glass.

9.2.1.5 *Pipet Viscometer*[4,5], calibrated, manufactured from low expansion borosilicate glass.

9.2.1.6 *Pycnometer,* calibrated, 50-mL.

9.2.1.7 *Automatic Pipet,* calibrated, 100-mL.

9.2.1.8 *Suitable Containers,* 250-mL or larger, made of inert material such as heat-resistant glass.

9.2.1.9 *Shaking Machine.*

9.2.1.10 *Rubber Bulbs.*

9.2.1.11 *Timer,* accurate to 0.2 s.

9.2.1.12 With the exception of the pipet, Ostwald, and Ubbelohde viscometers, apparatus capable of equivalent accuracy may be substituted.

9.2.2 *Reagents and Materials:*

9.2.2.1 *Acetone,* commercial grade.

9.2.2.2 *Chromic Acid Cleaning Solution*—Dissolve sodium dichromate $Na_2CrO_7 \cdot 2H_2O$, technical grade, in concentrated sulfuric acid (H_2SO_4, sp gr 1.84).

9.2.2.3 *m-Cresol*[5,6], having a viscosity of 12.83 cP at 25°C and a density of 1.029 ± 0.0011 g/mL at 25°C.

9.2.2.4 *Formic Acid* (90 ± 0.2 %)—Clear, water-white. ACS-grade formic acid with the following additional requirements: Methyl formate content 0.2 % maximum; density 1.1985 ± 0.001 g/mL at 25°C; viscosity 1.56° ± 0.02 cP at 25°C.

9.2.2.5 *Standard Viscosity Oils*[7]—Use viscosity oils, S-3, S-20, K-50, S-60, and S-200, which have been certified by a laboratory that has been shown to meet the requirements of ISO 17025 by independent assessment. The approximate kinematic viscosities at 25°C are 4.0, 35, 90, 120, and 480 cSt, respectively, and the certified viscosity reference standards shall be traceable to master viscometer procedures described in Practice D2162.

9.2.2.6 *Stopcock Lubricant.*[5,8]

9.2.2.7 *Analytical Balance*—Capable of weighing 0.1 mg (four decimal place balance).

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[4] The sole source of supply of the Drawing No. 66-1644 known to the committee at this time is Scientific Glass Apparatus Co., 51 Ackerman St., Bloomfield, NJ 07003.

[5] If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[6] The compound *m*-cresol is used with *n*-alkoxyalkyl polyamide 6:6 resin because formic acid tends to crosslink this polyamide. It is used with polyamide 6:10 resin because of this polyamide's insolubility in formic acid. The sole source of supply of what is known as No. 5072 is Matheson, Coleman, and Bell Co., East Rutherford, NJ 07073.

[7] Suitable standard viscosity oils are available from a number of companies.

[8] The sole source of supply of "Cello-Grease" known to the committee at this time is Fisher Scientific Co., 717 Forbes St., Pittsburgh, PA 15219.