



Designation: D789 – 19

Standard Test Method for Determination of Relative Viscosity of Concentrated Polyamide (PA) Solutions¹

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, but 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)

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.

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:

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 $0.58 \text{ mm} \pm 2\%$. 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 $25 \pm 0.1^\circ\text{C}$.

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 $\text{Na}_2\text{CrO}_7 \cdot 2\text{H}_2\text{O}$, 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 $1.029 \pm 0.0011 \text{ g/mL}$ 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 $1.1985 \pm 0.001 \text{ g/mL}$ at 25°C ; viscosity $1.56^\circ \pm 0.02 \text{ cP}$ 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).

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.

9.2.3 Calibration of ASTM Ubbelohde (suspended level)-type viscometer (note that a kinetic energy correction factor is required on all flow times less than 200 seconds, refer to 7.2 of Specification D446)—Size 1 type used to determine absolute viscosity of formic acid. 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.

9.2.3.1 Add to the viscometer 10-18 mL of viscosity oil standard from a volumetric pipet. Use S-3 for Size 1 viscometer and N-100 for Size 3 viscometers. Immerse the viscometer in the constant temperature bath at $25 \pm 0.02^\circ\text{C}$ and allow it to remain at least 20 minutes. Block off the air arm (not the capillary) and apply air pressure to the large diameter (filling) tube by means of a rubber bulb so that oil passes into the capillary until oil is above the upper timing mark. Un-block the air arm and simultaneously allow the oil to flow down. This ensures that the viscometer is wet. Again, force oil above the upper timing mark, and observe the time (to 0.2 seconds) required for the liquid to fall from the upper timing mark to the lower timing mark. Repeat until three successive values agree within 0.5 %, and record the average for the viscosity oil standard at 25°C as t_3 (S-3) or t_{100} (N-100). Remove the viscometer from the bath, clean and dry the inside surfaces thoroughly.

9.2.3.2 Repeat the above procedure, using 10-18 mL of 90 % formic acid in a Size 1 tube. Record the average efflux time as t_f . Calculate the absolute viscosity of the 90 % formic acid as follows:

$$\eta_f = f_t \times d_f \times t_f \quad (1)$$

where:

- η_f = absolute viscosity of formic acid, $\text{kPa} \times \text{s} (10^6 \text{ cP})$
- f_t = Size 1 viscometer tube factor, $\text{mm}^2/\text{s}(\text{cSt})/\text{s} = \eta_3/t_3$
- d_f = density of formic acid at 25°C , $\text{g/mL} = 1.1985$
- t_f = average efflux time for 90 % formic acid at 25°C , s
- η_3 = kinematic viscosity of Oil S-3 $\text{mm}^2/\text{s} (\text{cSt})$
- η_{100} = kinematic viscosity of Oil N-100, $\text{mm}^2/\text{s} (\text{cSt})$
- t_3 = average efflux time for oil S-3 at 25°C , s
- t_{100} = average efflux time for oil N-100 at 25°C , s

9.2.4 Calibration of Pipet Viscometer—(Note that a kinetic energy correction factor may be required on all flow times of less than 200 seconds, refer to 7.2 of Specification D446.) Use Oil S-20. Assemble the pipet viscometer so that the lowest mark on the pipet aligns with the 50-mL mark on the reservoir to the pipet. Place the assembly in the water bath adjusted to a temperature of $25 \pm 0.1^\circ\text{C}$. After at least 20 min, apply air pressure to the reservoir or vacuum to the capillary, by means of a rubber bulb, to drive the oil up into the pipet above the upper timing mark. Place a finger over the top of the pipet, and release the pressure by opening the system to air. Remove the finger and allow pipet to drain. Repeat at least three times to wet the pipet thoroughly, and then record the time (to 0.2 s) for the liquid level to fall from the upper timing level to the lower. Determine the efflux time, t_{20} , repeating until three successive values agree within 0.5 %, and record the average. Repeat the procedure with Oil S-60 to obtain t_{60} . Calculate the viscometer tube factor as follows:

$$\text{tube factor} = (f_{20} + f_{60})/2 \quad (2)$$

where:

- f_{20} = kinematic viscosity of S-20 oil, $\text{mm}^2/\text{s} (\text{cSt})/t_{20}$,
- f_{60} = kinematic viscosity of S-60 oil, $\text{mm}^2/\text{s} (\text{cSt})/t_{60}$,
- t_{20} = average efflux time of S-20 oil, s, and
- t_{60} = average efflux time of S-60 oil, s.

This value shall be used in calculating the relative viscosity of a polymer solution, as shown in 9.2.8.

9.2.5 Calibration of Ostwald (Cannon-Fenske Routine) Viscometer—(Note that a kinetic energy correction factor may be required on all flow times of less than 200 seconds, refer to 7.2 of Specification D446.) Add to the viscometer 10 mL of Oil S-3 at approximately 25°C from a volumetric pipet. Immerse the viscometer in the constant-temperature bath at $25 \pm 0.1^\circ\text{C}$ and allow it to remain at least 20 min. Apply air pressure to the large diameter leg by means of a rubber bulb until oil is above the upper timing mark. Allow the oil to flow down. Repeat several times to ensure thorough wetting of the viscometer. Again, force oil above the upper timing mark, and observe the time (to 0.2 s) required for the liquid to fall from the upper timing mark to the lower timing mark. Repeat until three successive values agree within 0.5 %, and record the average for Oil S-3 at 25°C as t_3 . Remove the viscometer from the bath, clean and dry the inside surfaces thoroughly, and repeat the above procedure, using 10 mL of 90 % formic acid. Record the average efflux time as t_f . Calculate the absolute viscosity of the 90 % formic acid as follows:

$$\eta_f = f_t \cdot d_f \cdot t_f \quad (3)$$

where:

- η_f = absolute viscosity of formic acid, $\text{kPa} \cdot \text{s} (10^6 \text{ cP})$,
- f_t = viscometer tube factor, $\text{mm}^2/\text{s} (\text{cSt})/\text{s} = \eta_3/t_3$,
- η_3 = kinematic viscosity of Oil S-3, $\text{mm}^2/\text{s} (\text{cSt})$,
- t_3 = average efflux time for Oil S-3 at 25°C , s,
- d_f = density of 90 % formic acid at 25°C , g/mL , = 1.1985, and
- t_f = average efflux time for 90 % formic acid at 25°C , s.

9.2.6 Preparation of Solutions:

9.2.6.1 Preparation of Polyamide Polymer-Formic Acid Solutions—Weigh 11.00 g of polyamide polymer into a clean, dry, 250-mL, container (see Note 2). Add, by means of the calibrated 100-mL automatic pipet, 100 mL of 90 % formic acid at $25 \pm 1^\circ\text{C}$. If other volumes are required, maintain the same weight to volume ratio as stated. Slowly shake the container while adding the acid to prevent the polymer from forming a gelatinous mass. Set the container in an oven at 50°C for 15 min, if needed, to obtain complete solutions. Then seal the container, and place the container and contents on a shaking machine. Agitate until the solution is complete (see Note 3).

NOTE 2—It is best if the polymer contains less than 0.28 % moisture. If it contains more than 0.28 %, the polymer can be dried. Normally, drying at 70°C in a vacuum for 4 to 6 h or 90°C for 20 min is adequate.

NOTE 3—Heating can be continued for a maximum of 2 h while shaking at a temperature not exceeding 50°C .

9.2.6.2 The procedure for the preparation of *n*-alkoxy-alkyl polyamide 6:6 and polyamide 6:12 polymers in *m*-cresol is the same as for the preparation of formic acid solutions, except that the quantity of polyamide polymer shall be 9.44 g instead of