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Designation: D 789 – 04

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

This standard is issued under the fixed designation D 789; 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 polyamide (PA).

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

1.3 *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.*

NOTE 1—There is no similar or equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 883 Terminology Relating to Plastics

D 4000 Classification System for Specifying Plastic Materials

3. Terminology

3.1 Definitions—The definitions used in this test method is in accordance with Terminology D 883.

4. Significance and Use

4.1 This test method is intended for use as a control and acceptance test. It is 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 Since some materials require special treatment, refer to the ASTM test methods applicable to the material being tested. Classification System D 4000 lists materials that would be applicable to the test contained in this test method.

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 at the end of this test method 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 nylon polymer by either the pipet viscometer method (9.2) or the Brookfield viscometer method[3] (9.3). The pipet viscometer method is the referee method.

9.2 *Pipet Viscosity*:

9.2.1 *Apparatus*:

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

9.2.1.2 *Precision Thermometer,* calibrated, for use in the water bath.

9.2.1.3 *Pipet Viscometer,* calibrated, 25-mL.[4]

9.2.1.4 *Ostwald Viscometer*, calibrated.

9.2.1.5 *Pycnometer*, calibrated, 50-mL.

[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] A Brookfield viscometer is available from Brookfield Engineering Laboratories, Inc., 240 Cushing St., Stoughton, MA 02072.

[4] Drawing No. 66-1644, available from the Scientific Glass Apparatus Co., 51 Ackerman St., Bloomfield, NJ 07003.

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

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

9.2.1.7 *Erlenmeyer Flasks*, 250-mL, heat-resistant glass.

9.2.1.8 *Shaking Machine*.

9.2.1.9 *Rubber Bulbs*.

9.2.1.10 *Timer*, accurate to 0.2 s.

9.2.1.11 With the exception of the pipet and Ostwald 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] 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*—Use Cannon Instrument Company[6] standard viscosity oils S-3, S-20, K-50, S-60, and S-200. The approximate kinematic viscosities at 25°C are 4.0, 35, 90, 120, and 480 cSt, respectively.

9.2.2.6 *Stopcock Lubricant.*[7]

9.2.3 *Analytical Balance*:

Capable of weighing 0.1 mg (four decimal place balance).

9.2.4 *Calibration of Pipet Viscometer*—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 ± 0.1°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:

$$\text{tube factor} = (F_{20} + F_{60})/2 \quad (1)$$

where:

F_{20} = kinematic viscosity of S-20 oil, mm^2/s (cST)/t_{20},
F_{60} = kinematic viscosity of S-60 oil, mm^2/s (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 Viscometer*—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 ± 0.1°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:

$$\eta_f = f_t \cdot d_f \cdot t_f \quad (2)$$

where:

η_f = absolute viscosity of formic acid, kPa · s (cP),
f_t = viscometer tube factor, mm^2/s (cSt)/s = η_3/t_3,
η_3 = kinematic viscosity of Oil S-3, mm^2/s (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, = 1.1975, 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 Nylon Polymer-Formic Acid Solutions*—Weigh 11.00 g of nylon polymer into a clean, dry, 250-mL, ground-glass stoppered Erlenmeyer flask (see Note 2). Add, by means of the calibrated 100-mL automatic pipet, 100 mL of 90 % formic acid at 25 ± 1°C. Slowly shake the flask while adding the acid to prevent the polymer from forming a gelatinous mass. Set the flask in an oven at 50°C for 15 min, if needed, to obtain complete solutions. Then put stopcock lubricant on the glass stopper, insert it tightly into the flask, and place the flask and contents on a shaking machine. Agitate until the solution is complete (see Note 3).

9.2.6.2 The procedure for the preparation of *n*-alkoxy-alkyl nylon 6:6 and nylon 6:12 polymers in *m*-cresol is the same as for the preparation of formic acid solutions, except that the quantity of nylon polymer shall be 9.44 g instead of 11.00 g, and the *m*-cresol shall be specified as the solvent instead of formic acid.

NOTE 2—The polymer should contain less than 0.28 % moisture. If it contains more than 0.28 %, the polymer should 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 may be continued for a maximum of 2 h while shaking at a temperature not exceeding 50°C.

9.2.7 *Procedure*—Fill the reservoir bottle of a dry, clean pipet viscometer to the 50-mL mark with the nylon polymer-formic acid solution. Insert the pipet and reservoir top and fasten securely. Determine the efflux time, t_p, as described in 9.2.5.

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

[6] Suitable standard viscosity oils are available from Cannon Instrument Co., P.O. Box 16, State College, PA 16801.

[7] "Cello-Grease," available from the Fisher Scientific Co., 717 Forbes St., Pittsburgh, PA 15219, has been found satisfactory for this purpose.

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