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Standard Test Methods for Determination of Solution Viscosities of Polyamide (PA)^{1, 2}

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1. Scope*

1.1 These test methods cover the determination of solution viscosities as they apply to polyamide (PA).

1.2 The values stated in SI units are to be regarded as standard. The values given in brackets 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—This standard and ISO 307 address the same subject, but the technical content is different.

2. Referenced Documents

2.1 *ASTM Standards:*³

D 446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers

D 883 Terminology Relating to Plastics

D 4000 Classification System for Specifying Plastic Materials

D 6779 Classification System for Polyamide Molding and Extrusion Materials (PA)

2.2 *ISO Standards:*⁴

ISO 307 Determination of Viscosity Number of Polyamides in Dilute Solutions

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 D 883.

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 There is no similar or equivalent ISO standard.

³ 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.

⁴ Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

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 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 tests contained in these test methods.

4.3 The steps involved in running this method are:

4.3.1 Calibration of the viscometers,

4.3.2 Preparation of solutions,

4.3.3 Determination of efflux time,

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

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

4.3.4.2 Determining the absolute viscosity of the formic acid used.

4.4 Viscosity for groups 03, 04, and 05 (PA11, PA12, and PA6,12) in Classification System D 6779 shall be measured using solvents other than formic acid. Relative viscosities for Groups 03 and 04 shall be measured using 0.5 g of polymer dissolved in 99.5 g of m-cresol at 25.0 ± 0.1°C in a Cannon-Fenske No. 200 viscometer. Inherent viscosity of Group 05 shall be measured using 0.5 g of polymer dissolved in 100 mL of m-cresol at 25.0 ± 0.1°C in a Cannon-Fenske No. 200 viscometer. The inherent viscosity is calculated as follows:

$$\text{Inherent viscosity} = \frac{\ln(t_s/t_c)}{C} \quad (1)$$

where:

t_s = average efflux time for sample solution,

t_c = average efflux time for solvent, and

C = concentration in g/100 mL

5. Test Specimen

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

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

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 nylon 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^{5, 6} 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 **D 446**, 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)).

9.2.1.3 *Ubbelohde (Suspended Level)-type Viscometer*, calibrated by an **ISO 17025**-accredited laboratory or in accordance with the procedure set out in 9.2.3 and manufactured from low-expansion borosilicate glass.

9.2.1.4 *Ostwald-type Viscometer*, calibrated by an **ISO 17025**-accredited laboratory or in accordance with the procedure set out in 9.2.5 and manufactured from low-expansion borosilicate glass.

9.2.1.5 *Pipet Viscometer*, calibrated by an **ISO 17025**-accredited laboratory or in accordance with the procedure set out in 9.2.4, 25 mL and manufactured from low expansion borosilicate glass.^{7, 6}

9.2.1.6 *Pycnometer*, calibrated, 50-mL.

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

9.2.1.8 *Erlenmeyer Flasks*, 250-mL, 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 Na₂CrO₇ · 2H₂O, technical grade, in concentrated sulfuric acid (H₂SO₄, sp gr 1.84).

9.2.2.3 *m-Cresol*,^{8, 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*—Use certified viscosity oils, which have been calibrated by a laboratory-accredited to **ISO 17025**⁹. 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*.^{10, 6}

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

9.2.3 Calibration of ASTM Ubbelohde (suspended level)-type viscometer (note that a kinetic energy correction factor may be required on all flow times less than 200 seconds, refer to 7.2 of Specification **D 446**)—Size 1 type used to determine absolute viscosity of formic acid. Size 3 type used to determine nylon polymer-formic acid solutions.

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 ± 0.02°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

⁷ 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.

⁸ 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. The sole source of supply of what is known as No. 5072 is Matheson, Coleman, and Bell Co., East Rutherford, NJ 07073.

⁹ Suitable standard viscosity oils are available from a number of companies.

¹⁰ 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.

⁵ The sole source of supply of the Brookfield viscometer known to the committee at this time is Brookfield Engineering Laboratories, Inc., 240 Cushing St., Stoughton, MA 02072.

⁶ 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.

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:

$$\eta_f = f_i \times d_f \times t_f \quad (2)$$

where:

- η_f = absolute viscosity of formic acid, kPa × s(cP)
- f_i = Size 1 viscometer tube factor, mm²/s(cSt)/s = η_3/t_3
- d_f = density of formic acid at 25°C, g/mL = 1.1975
- t_f = average efflux time for 90 % formic acid at 25°C, s
- η_3 = kinematic viscosity of Oil S-3 mm²/s (cSt)
- η_{100} = kinematic viscosity of Oil N-100, mm²/s (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 D 446.) 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:

$$\text{tube factor} = (f_{20} + f_{60})/2 \quad (3)$$

where:

- f_{20} = kinematic viscosity of S-20 oil, mm²/s (cST)/ t_{20} ,
- f_{60} = kinematic viscosity of S-60 oil, mm²/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 (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 D 446.) 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:

$$\eta_f = f_i \cdot d_f \cdot t_f \quad (4)$$

where:

- η_f = absolute viscosity of formic acid, kPa · s (cP),
- f_i = viscometer tube factor, mm²/s (cSt)/s = η_3/t_3 ,
- η_3 = kinematic viscosity of Oil S-3, mm²/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*—Pipet or pour 10 mL of the nylon polymer-formic acid solution into the viscometer. Determine the efflux time, t_p , as described in 9.2.3, 9.2.4, or 9.2.5.

9.2.8 *Calculation of Relative Viscosity*—The relative viscosity, η_r , is the ratio of the absolute viscosity of the polymer solution to that of the formic acid:

$$\eta_r = (\eta_p/\eta_f) = (f_i \cdot d_p \cdot t_p)/\eta_f \quad (5)$$

where:

- d_p = density of formic acid-polymer solution at 25°C (see 9.2.9), and
- t_p = average efflux time for formic acid-polymer solution, s.
- η_f = absolute viscosity of formic acid, kPa × s(cP)
- f_i = viscometer tube factor, mm²/s (cSt)/s = η_3/t_3

Calculate the relative viscosity of *n*-alkoxyalkyl nylon 6:6 and nylon 6:12 resins using *m*-cresol as the comparison base,