Designation: D789 – 15

Standard Test Methods for Determination of Solution Viscosities of Polyamide (PA)[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 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:*[2]

D446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers

D883 Terminology Relating to Plastics

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

2.2 *ISO Standards:*[3]

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 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 Viscosity for groups 03, 04, and 05 (PA11, PA12, and PA6,12) in Classification System D6779 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.

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.

[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**

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[4,5] 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 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,*[5,6]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.

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 $Na_2CrO_7 \cdot 2H_2O$, technical grade, in concentrated sulfuric acid (H_2SO_4, sp gr 1.84).

9.2.2.3 *m-Cresol,*[5,7]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*[8]—Use certified viscosity oils, which have been calibrated by a laboratory-accredited to ISO 17025. 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.*[5,9]

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 D446)—Size 1 type used to determine absolute viscosity of formic acid. Size 3 type used to determine polyamide 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 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

[4] 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.

[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 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.

[7] 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.

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

[9] 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.

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:

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

where:

η_f = absolute viscosity of formic acid, kPa × s(E+6cP)
f_t = Size 1 viscometer tube factor, $mm^2/s(cSt)/s = \eta_3/t_3$
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 mm^2/s (cSt)
η_{100} = kinematic viscosity of Oil N-100, mm^2/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 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 ± 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, 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 (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 ± 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:

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

where:

η_f = absolute viscosity of formic acid, kPa · s (E+6cP),
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 Polyamide Polymer-Formic Acid Solutions*—Weigh 11.00 g of polyamide 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).

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 11.00 g, and the *m*-cresol shall be specified as the solvent instead of formic acid.

9.2.7 *Procedure*—Pipet or pour 10 mL of the polyamide 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:

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

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(E+6cP)
f_t = viscometer tube factor, mm^2/s (cSt)/s = η_3/t_3

Calculate the relative viscosity of *n*-alkoxyalkyl polyamide 6:6 and polyamide 6:12 resins using *m*-cresol as the comparison base, not formic acid. Substitution of proper constants in the calculation formulas will then be necessary.

9.2.9 *Density of Polyamide Polymer-Formic Acid Solution:*

9.2.9.1 Prepare the polyamide polymer-formic acid solution as described in 9.2.6.1.

NOTE 4—Calibration of the pycnometer used to determine density is made by repeating the procedure specified in 9.2.9.2 and 9.2.9.3, using distilled water in place of the polyamide polymer-formic acid solution.

9.2.9.2 Weigh (to ±0.1 mg) a clean, dry, calibrated 50-mL pycnometer, and fill it with the polyamide polymer-formic acid solution at a temperature slightly below (1 to 2°C) the test temperature. Stopper or cap the pycnometer, leaving the overflow orifice open. Take care to prevent the formation of bubbles in the pycnometer. Immerse the filled pycnometer (the neck of the pycnometer shall be above the water line) into a constant-temperature liquid bath, maintained at 25 ± 0.1°C. Allow 20 to 30 min for temperature equilibrium to be reached.

9.2.9.3 Remove the pycnometer from the liquid bath, and wipe away any overflow with paper towels or other absorbent material, taking care not to remove any subsequent overflow that may be caused in this step. Dry the pycnometer thoroughly, and weigh immediately (±0.1 mg).

9.2.9.4 The density of the polyamide polymer-formic acid solution, in grams per cubic centimetre, is calculated by the following formulas:

$$d_p = \frac{m_p - m_o}{V} \quad (6)$$

and

$$V = \frac{m_w - m_o}{d_w} \quad (7)$$

where:

m_p = mass of pycnometer and polyamide polymer-formic acid solution, g,
m_o = mass of empty pycnometer, g,
V = volume of water at 25°C, cm^3,
m_w = mass of pycnometer and water, g, and
d_w = density of water at 25°C (0.9970), g/cm^3.

9.3 *Brookfield Viscometer:*

9.3.1 *Apparatus:*

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

9.3.1.2 *Precision Thermometer,* calibrated, for use in liquid bath.

9.3.1.3 *Brookfield Synchro-Lectric Viscometer, Model LVF.*

9.3.1.4 *Viscometer,* Cannon-Fenske type, Size 75, uncalibrated.

9.3.1.5 *Automatic Pipet,* 200-mL.

9.3.1.6 *Shaking Machine,* reciprocating type.

9.3.1.7 *Stopwatch,* having divisions of at least 0.1 s or 0.01 min and accuracy of at least 0.05 %.

9.3.1.8 *Bottles,* 8-oz, round, wide-mouth with caps containing polyethylene liners.

9.3.1.9 With the exception of the Brookfield and Cannon-Fenske viscometers, apparatus capable of equivalent accuracy may be substituted.

9.3.2 *Reagents and Materials*—Same as described in 9.2.2.

9.3.3 *Analytical Balance*—Same as described in 9.2.2.7.

9.3.4 *Determination of Absolute Viscosity of Formic Acid:*

9.3.4.1 Add 10.0 mL (pipet) of 90 ± 0.2 % formic acid (at 25.0 ± 0.5°C) to a Size 75 Cannon-Fenske viscometer. The viscometer is calibrated as described in 9.3.4.3. Suspend the viscometer from the lid of the constant-temperature liquid bath in a vertical position so that the upper bulb is well immersed in the bath at 25 ± 0.1°C. Allow 20 to 30 min for temperature equilibrium to be reached. Apply suction (bulb or vacuum) to the small leg of the viscometer and draw the liquid above the upper timing mark. Allow to drain. Repeat twice to ensure complete wetting of the tube. Observe and record the time required for the meniscus of liquid to fall from the upper timing mark to the lower timing mark. Repeat until three successive readings agree within 0.5 %. Average the results; record the efflux time as t_f.

9.3.4.2 *Calculation of Absolute Viscosity for Formic Acid:*

$$\eta_f = f_t \cdot d_f \cdot t_f \quad (8)$$

where:

η_f = viscosity of formic acid, kPa · s (E+6cP),
f_t = tube factor, mm^2/s (cSt)/s (9.3.4.3),
d_f = density of formic acid at 25 ± 0.1°C, g/cm^3 = 1.1975, and
t_f = efflux time of formic acid, s.

9.3.4.3 *Calibration of Viscometer, Cannon-Fenske, Size 75*—Determine the efflux time of the standard viscosity Oil S-3, following the procedures of 9.3.4.1. Record the efflux time as *t*.

$$f_t = \eta_d / t_d \quad (9)$$

where:

f_t = tube factor, mm^2/s (cSt)/s,
η_d = viscosity of S-3 oil, mm^2/s (cSt), and
t_d = efflux time of S-3 oil, s.

9.3.5 *Determination of Relative Viscosity of Polyamide-Formic Acid Solutions:*

9.3.5.1 Using an automatic pipet, add 200 mL of 90 ± 0.2 % formic acid to an 8-oz screw-cap bottle with a metal cap, containing a polyethylene liner. Weigh 22 ± 0.01 g of polyamide polymer and add to the formic acid in the 8-oz bottle. (Use care to avoid splashing formic acid out of the bottle.) Allowing the cap to remain loose, heat the mixture to 50 ± 5°C, using any convenient method.

9.3.5.2 Tighten the cap thoroughly, and place the sample in the shaker. Agitate until all the polyamide is in solution. Then place the bottle in a constant-temperature liquid bath maintained at 25 ± 0.1°C for not less than 1 h.

9.3.5.3 Some polyamides that dissolve slowly can be subject to time-temperature effects. To avoid possible degradation, it is recommended that materials having relative viscosities above 200 not be heated. The supplier's recommended procedures for dissolving should be followed in such cases.

9.3.5.4 Select the spindle and speed according to the expected viscosity of the solution by using the following table:

		Spindle Number for Indicated Speed, r/min		
RV	BVs	60	30	12
5–61	9–100	1		
61–122	100–200		1	
122–305	200–500	2		1

Where a choice of two spindles is given, it is more convenient to use the smaller-numbered spindle and change the speed than to change spindles. Use the same spindle and speed for similar viscosity level polymers.

9.3.5.5 Immerse the spindle and guard of the calibrated Brookfield viscometer and adjust to the immersion mark. (See 9.3.8 for calibration of the Brookfield viscometer.) (The temperature of the spindle and guard shall be maintained at 25 ± 0.1°C by keeping them immersed in a bottle of water in the bath between uses and wiping them dry before using.)

9.3.5.6 Observe the spindle to see if air bubbles are clinging to it. Remove adhering air bubbles by removing and replacing spindle, or with a wire (avoid scratching spindle). Level the instrument. (Tilt the No. 1 spindle while immersing it to prevent trapping air on the bottom of the spindle.) Depress the clutch and turn on the motor. (Depressing the clutch first prevents unnecessary wear.) Adjust the proper spindle speed. (Set the speed regulator when the instrument is in motion, not when stopped.) Release the clutch and allow the spindle to rotate until the pointer stabilizes at a fixed position on the dial. (This requires about 30 s for 50 RV; it may require several minutes for 200-RV materials.) Depress the clutch, and when the pointer comes into view, stop the motor. (If the pointer goes to the full-scale limit, reduce the speed stepwise until the pointer stays on scale. If the pointer goes to full-scale limit at the lowest speed, change to the next higher-numbered spindle.)

9.3.5.7 Read the position of the pointer on the dial, estimating to the nearest 0.1 scale division. Take one reading if the RV is reported to the nearest whole number. Take four readings if the RV is reported to the nearest 0.1 unit, and report the average. (If additional readings are required, start the motor with the clutch still depressed, holding the original reading, and then release the clutch. This will speed up readings by reducing oscillation of the pointer.)

9.3.6 *Calculation of Brookfield Viscosity of Polyamide-Formic Acid Solution:*

$$BV_s = (IR, -0.4) \times F \text{ for } 60 \text{ r/min} \quad (10)$$

$$BV_s = (IR) \times F \text{ for } 30 \text{ r/min or } 12 \text{ r/min} \quad (11)$$

where:

IR = instrument reading,
BV_s = Brookfield viscosity of the solution, cP, and
F = spindle factor from the following table:

	Speed, r/min		
Spindle	12	30	60
1	5	2	1
2	25	10	5
3	100	40	20
4	500	200	100

9.3.7 *Calculation of Relative Viscosity of the Polyamide-Formic Acid Solution:*

$$RV = (\eta_s/\eta_f) = [(BV_s \cdot C_f)/\eta_f] \quad (12)$$

where:

R_V = relative viscosity of the polyamide-formic acid solution,
η_f = absolute viscosity of formic acid, 9.3.4.2,
η_s = absolute viscosity of polyamide-formic acid solutions,
BV_s = Brookfield viscosity of the solution, kPa · s (cP) (9.3.6), and
C_f = calibration factor (the instruments should be calibrated periodically against standard viscosity oils; see 9.3.8).

9.3.8 *Calibration of Brookfield Viscometer:*

9.3.8.1 Select the standard viscosity oil closest or within 10 % of the absolute viscosity level of the polyamide-formic acid solution to be tested. Make sure the height of the oil in the 8-oz bottle is the same as that of the polyamide-formic acid solutions. (The Brookfield viscometer is factory-adjusted to read viscosities within 1 % of the absolute, provided that the measurements are made in a container that is at least 76 mm (3 in.) in diameter and the spindle is properly centered in the container. The calibration must be performed in the same size container used for testing the polyamide-formic acid solution. Using the standard 8-oz bottle, the calibration factor (C_f) will be about 0.96 for an instrument in good repair.)

9.3.8.2 Determine the instrument reading of the oil following 9.3.5.1. The calibration factor for the instrument is:

$$C_f = TV_o/BV_o \quad (13)$$

where:

C_f = calibration factor,
TV_o = true viscosity of the oil, kPa · s (cP), and
BV_o = Brookfield viscosity of the oil (instrument reading corrected for speed and spindle factors, kPa · s (cP); see 9.3.6).

9.3.9 *Precision*—Relative viscosities by the Brookfield viscometer are comparable to those obtained by the pipet viscometer, both instruments being calibrated against viscosity oil standards. In a laboratory test, a sample measured by the Brookfield method had an $\bar{X}$ of 49.8 with an *Sd* of 0.53 covering 192 determinations made by 24 operators. The same sample measured by the pipet viscometer had an $\bar{X}$ of 50.4 with a *Sd* of 0.46 in a test of 20 determinations made by five operators. Table 1 at the end of this test method gives repeatability and reproducibility statistics for relative viscosity testing.

10. Precision [10]

10.1 Precision, characterized by repeatability, S_r, r, and reproducibility, S_R, R, has been determined for these materials to be:

11. Keywords

11.1 Brookfield viscosity; density of polyamide polymer-formic acid solution; pipet viscosity; relative viscosity

[10] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D20-1220.

TABLE 1 Repeatability and Reproducibility for Relative Viscosity

Material	Average	S_r	S_R	r	R
Polyamide 6	140.9	2.11	5.86	5.90	16.41
Polyamide 66	45.4	1.72	2.16	4.80	6.04

APPENDIX

(Nonmandatory Information)

X1. PROCEDURE FOR DETERMINATION OF RELATIVE VISCOSITY IN 90 % FORMIC ACID USING AN AUTOMATED TESTING DEVICE

X1.1 Scope

X1.1.1 This procedure describes the method for determining the relative viscosity of a solution of polyamide polymer in 90 % formic acid using an automated testing device.

X1.2 Principle

X1.2.1 The relative viscosity, RV, measured by the technique described in this method is a ratio of a concentrated solution of polyamide in formic acid to the polyamide solvent grade formic acid. The viscosity in centistokes (cSt) of an 8.4 % (w/w) solution of polyamide in formic acid is related to the viscosity of the solvent. The viscosity of the polymer/formic acid solution is determined by measuring the time of efflux of the solution in a calibrated viscometer.

X1.3 Equipment Required

X1.3.1 Constant temperature liquid bath regulated at 25.0 ± 0.1°C,

X1.3.2 ASTM Ubbelohde-calibrated viscometers,

X1.3.3 Bottles, glass or polypropylene, with screw type lids (polypropylene with or without PTFE liner) and pipets,

X1.3.4 Automated polymer viscometer system,

X1.3.5 Shaker,

X1.3.6 10 mesh screen and grinder, if samples require grinding,

X1.3.7 Desiccators with Drierite desiccant,

X1.3.8 Analytical balance, and

X1.3.9 Calibrated thermometer.

X1.4 Reagents

X1.4.1 90% ± 0.2 % formic acid ACS grade for testing, viscosity grade for cleaning.

X1.4.2 Acetone, reagent grade.

X1.5 Procedure

X1.5.1 *Sample Preparation*

NOTE X1.1—A control should be the first sample tested each day.

X1.5.1.1 Place the ground polymer in a 43-mm aluminum pan and place the pan in the vacuum oven for 20 minutes. The oven should be set at 93 ± 4°C.

X1.5.1.2 Store the dried sample in the desiccator until ready for use.

X1.5.2 *Solution Preparation*

X1.5.2.1 Place a glass bottle on the analytical balance. Wait until the weight stabilizes, then carefully add the required amount of polymer to the bottle.

X1.5.2.2 Using a pipet slowly add formic acid to the polymer until the weight reaches the weight required for an 8.4 % solution. Remove the bottle from the balance and place it on a shaking device.

NOTE X1.2—Sample preparation may also be done by automated systems.

X1.5.3 *Specimen Testing*

X1.5.3.1 Once the polyamide has dissolved, remove the sample from the shaker.

X1.5.3.2 Make sure the liquid bath is at the proper level and at 25 ± 0.1°C.

X1.5.3.3 Add the polyamide/polymer solution to the designated viscometer.

X1.5.3.4 Initiate the test as required by the automated equipment in use.

X1.5.3.5 The automated device will draw, release and time the sample drop using photoelectric devices. Obtain two consecutive flow times for each sample within 0.2 % of the mean.

X1.5.3.6 When the test is complete the computer will use the average flow time to calculate (see 9.2.8) and display the relative viscosity value. Care should be taken to test samples promptly as experience shows that samples may degrade as much as 2 % overnight.

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D789 - 07$^{\varepsilon 1}$) that may impact the use of this standard. (April 1, 2015)

(1) Corrected ISO equivalency notations.
(2) Replaced "nylon" with "polyamide" throughout standard.
(3) Removed old 4.2 due to unnecessary references back to Classification D4000.
(4) Updated Mercury statements based upon current ASTM caveat.
(5) Moved placement of footnotes to better align with the item being covered.