Designation: D 789 – 98

Standard Test Methods for Determination of Relative Viscosity and Moisture Content 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 These test methods cover the determination of relative viscosity and moisture content as they apply 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. For a specific statement on safety,* see 10.4.

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

2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics[2]

D 1898 Practice for Sampling of Plastics[2]

D 4000 Classification System for Specifying Plastic Materials[3]

3. Terminology

3.1 Definitions—The definitions used in these test methods are in accordance with Terminology D 883D 883.

4. Significance and Use

4.1 These test methods are intended for use as control and acceptance tests. They may be used also 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, reference should also be made to the ASTM test methods applicable to the material being tested. Classification System D 4000D 4000 lists materials that would be applicable to the tests contained in these test methods.

5. Test Specimen

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

6. Number of Tests

6.1 Duplicate determinations, using two separate samples, shall be considered sufficient for testing each molding powder batch or resin lot.

7. Sampling

7.1 Unless otherwise agreed upon between the purchaser and the seller, sample the materials in accordance with the sampling procedure prescribed in Practice D 1898D 1898. Adequate statistical sampling shall be considered an acceptable alternative. A batch or lot of resin shall be considered as a unit of manufacture as prepared for shipment and may consist of a blend of two or more "production runs" of material.

8. Conditioning

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

TEST METHODS

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[4] (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.[5]

[1] These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and are the direct responsibility of Subcommittee D20.15 on Thermoplastic Materials (Section D20.15.09).

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[2] *Annual Book of ASTM Standards,* Vol 08.01.

[3] *Annual Book of ASTM Standards,* Vol 08.02.

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

[5] 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.**

9.2.1.4 *Ostwald Viscometer*, calibrated.
9.2.1.5 *Pycnometer*, calibrated, 50-mL.
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,*[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 %)*[7]—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[8] 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.*[9]
9.2.3 *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 9.2.7.

9.2.4 *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.

9.2.5 *Preparation of Solutions*:
9.2.5.1 *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).
9.2.5.2 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.

[6] 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.
[7] Available in drums from Union Carbide Corp., Chemical and Plastics Div., Moorestown, NJ 08057. Available in smaller quantities from Polyscience, Incorporated, Paul Valley Industrial Park, Warrington, PA 18976. Matheson, Coleman, and Bell 97 to 100 % formic acid, diluted with water and checked by titration to 90.0 ± 0.2 %, has also been found satisfactory.
[8] Suitable standard viscosity oils are available from Cannon Instrument Co., P.O. Box 16, State College, PA 16801.
[9] "Cello-Grease," available from the Fisher Scientific Co., 717 Forbes St., Pittsburgh, PA 15219, has been found satisfactory for this purpose.

9.2.6 *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 9.2.4.

9.2.7 *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:

$$\eta_r = (\eta_p/\eta_f) = (f_t \cdot d_p \cdot t_p)/\eta_f \quad (3)$$

where:

d_p = density of formic acid-polymer solution at 25°C (see 9.2.8), and

t_p = average efflux time for formic acid-polymer solution, s.

Calculate the relative viscosity of *n*-alkoxyalkyl nylon 6:6 and nylon 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.

9.2.8 *Density of Nylon Polymer-Formic Acid Solution*:

9.2.8.1 Prepare the nylon polymer-formic acid solution as described in 9.2.5.1.

NOTE 4—Calibration of the pyknometer used to determine density is made by repeating the procedure specified in 9.2.8.2 and 9.2.8.3, using distilled water in place of the nylon polymer-formic acid solution.

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

9.2.8.3 Remove the pyknometer from the water 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 pyknometer thoroughly, and weigh immediately (±0.1 mg).

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

$$d_p = \frac{m_p - m_o}{V} \quad (4)$$

and

$$V = \frac{m_w - m_o}{d_w} \quad (5)$$

where:

m_p = mass of pyknometer and nylon polymer-formic acid solution, g,

m_o = mass of empty pyknometer, g,

V = volume of water at 25°C, cm^3,

m_w = mass of pyknometer 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 Water Bath*, set to operate at 25 ± 0.1°C.

9.3.1.2 *Precision Thermometer*, calibrated, for use in water 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 *Determination of Absolute Viscosity of Formic Acid*:

9.3.3.1 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 may be calibrated as described in 9.3.3.3. Suspend the viscometer from the lid of the constant-temperature water 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.

9.3.3.2 *Calculation of Absolute Viscosity for Formic Acid*:

$$\eta_f = f_t \cdot d_f \cdot t_f \quad (6)$$

where:

η_f = viscosity of formic acid, kPa · s (cP),

f_t = tube factor, mm^2/s (cSt)/s (9.3.3.3),

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.

9.3.3.3 *Calibration of Viscometer, Cannon-Fenske, Size 75*—Determine the efflux time of the standard Cannon viscosity Oil S-3, following the procedures of 9.3.3.1. Record the efflux time as *t*.

$$f_t = \eta_d/t_d \quad (7)$$

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.

9.3.4 *Determination of Relative Viscosity of Nylon-Formic Acid Solutions*:

9.3.4.1 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 nylon polymer and add to the formic acid in the 8-oz bottle. (Use care to avoid splashing formic acid out of the bottle.)