Designation: ~~D789 – 15~~ D789 – 18

Standard Test ~~Methods~~Method for Determination of ~~Solution Viscosities~~Relative Viscosity of Concentrated Polyamide (PA) Solutions[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~~This test ~~methods cover~~method covers the determination of ~~solution viscosities as they apply to~~ 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~~ safety, health, and ~~health~~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.~~
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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)
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~~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.

[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**

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 ~~Viscosity for groups 03, 04, and 05 (PA11, PA12, and PA6,12) in Classification System~~ Solvents used to prepare dilute solutions of various polyamides are designated in ASTM 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:~~, ISO 16396, and ISO 307. These include:

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

~~where:~~

~~t_s = average efflux time for sample solution,~~
~~t_c = average efflux time for solvent, and~~
~~C = concentration in g/100 mL~~

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[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.

[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.

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~~ calibrated, ~~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~~ calibrated, ~~9.2.5 and~~ manufactured from low-expansion borosilicate glass.

9.2.1.5 *Pipet Viscometer,*[4,5] ~~calibrated by an ISO 17025-accredited laboratory or in accordance with the procedure set out in~~ calibrated, ~~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,*~~*Suitable Containers,* ~~250-mL,~~ 250-mL or larger, made of inert material such as heat-resistant glass.

9.2.1.9 ~~*Shaking Machine.*~~ *Shaking Machine*.

9.2.1.10 ~~*Rubber Bulbs.*~~ *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,*[5,6] 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 ~~gg/mL~~/~~mL~~ at 25°C; viscosity 1.56° ± 0.02 cP at 25°C.

9.2.2.5 *Standard Viscosity Oils*[7]—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.~~ 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.~~respectively, and the certified viscosity reference standards shall be traceable to master viscometer procedures described in Practice D2162.

9.2.2.6 ~~*Stopcock Lubricant.*~~[5,9] *Stopcock Lubricant.*[5,8]

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~~ 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. ~~Size 3 type used to determine polyamide polymer-formic acid solutions.~~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 ± 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 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:

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

[4] The sole source of supply of the ~~Brookfield viscometer~~ Drawing No. 66-1644 known to the committee at this time is ~~Brookfield Engineering Laboratories, Inc., 240 Cushing St., Stoughton, MA 02072.~~Scientific Glass Apparatus Co., 51 Ackerman St., Bloomfield, NJ 07003.

~~[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.~~

[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.

where:

~~η_f~~ = ~~absolute viscosity of formic acid, kPa × s(E+6cP)~~
η_f = absolute viscosity of formic acid, kPa × s(10^6 cP)
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~~
d_f = density of formic acid at 25°C, 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 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:

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

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:

$$\eta_f = f_t \cdot d_f \cdot t_f \tag{3}$$

where:

~~η_f~~ = ~~absolute viscosity of formic acid, kPa · s (E+6cP),~~
η_f = absolute viscosity of formic acid, kPa · s (10^6 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 Polyamide Polymer-Formic Acid Solutions*—Weigh 11.00 g of polyamide polymer into a clean, dry, 250-mL, ~~ground-glass stoppered Erlenmeyer flask~~ container (see Note 2). Add, by means of the calibrated 100-mL automatic pipet, 100 mL of 90 % formic acid at 25 ± 1°C. If other volumes are required, maintain the same weight to volume ratio as stated. Slowly shake the ~~flask~~container while adding the acid to prevent the polymer from forming a gelatinous mass. Set the ~~flask~~container 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,~~ seal the container, and place the ~~flask~~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 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:

$$\eta_r = (\eta_p/\eta_f) = (f_t \cdot d_p \cdot t_p)/\eta_f \quad (4)$$

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)~~
η_f = absolute viscosity of formic acid, kPa × s(10^6 cP)
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,~~centimeter, is calculated by the following formulas:

$$d_p = \frac{m_p - m_o}{V} \quad (5)$$

and

$$V = \frac{m_w - m_o}{d_w} \quad (6)$$

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*~~*Rotational 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.*~~ *Rotational Viscometer,* (see E1953) with a a torque constant of 60 to 75 µN-m linear to within ±1 % capable of rotational speeds of 6, 12, 30, and 60 r/min stable within ±1 %

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:*

$$\eta_f = f_t \cdot d_f \cdot t_f \tag{7}$$

where:

~~η_f = viscosity of formic acid, kPa · s (E+6cP),~~
η_f = viscosity of formic acid, kPa · s (10^6 cP),
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.

$$f_t = \eta_d / t_d \tag{8}$$

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~~rotational viscometer and adjust to the immersion mark. (See 9.3.8 for calibration of the ~~Brookfield~~rotational 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~~ Initiate spindle rotation and adjust the spindle rotaitional speed until the torque indicator stabilizes at a fixed ~~position on the dial. (This requires~~ mid-scale reading. This may require about 30 s for 50 ~~RV; it may require~~ RV materials and 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.)~~ 200 RV materials.

9.3.5.7 ~~Read the position of the pointer on the dial, estimating~~ Record the indicated torque (or viscosity) to the nearest ~~0.1 scale division.~~0.1 % (full scale). Take one reading if the RV is reported to the nearest whole number. Take four readings if the RV is