



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 Plastics²

D 1898 Practice for Sampling of Plastics²

D 4000 Classification System for Specifying Plastic Materials³

3. Terminology

3.1 Definitions—The definitions used in these test methods are in accordance with Terminology D 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 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 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 method⁴ (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 $25 \pm 0.1^\circ\text{C}$.

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

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

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.

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

- 9.2.1.3 *Pipet Viscometer*, calibrated, 25-mL.⁵
 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 $\text{Na}_2\text{CrO}_7 \cdot 2\text{H}_2\text{O}$, 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* ($90 \pm 0.2\%$)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 $1.56^\circ \pm 0.02$ cP at 25°C.

9.2.2.5 *Standard Viscosity Oils*—Use Cannon Instrument Company⁸ 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 $25 \pm 0.1^\circ\text{C}$. 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:

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

⁵ Drawing No. 66-1644, available from the Scientific Glass Apparatus Co., 51 Ackerman St., Bloomfield, NJ 07003.

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 $90.0 \pm 0.2\%$, 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.

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 $25 \pm 0.1^\circ\text{C}$ 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:

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

where:

- η_f = absolute viscosity of formic acid, $\text{kPa} \cdot \text{s}$ (cP),
 f_i = 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 $25 \pm 1^\circ\text{C}$. 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.

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:

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

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 pycnometer 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 pycnometer, 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 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 water bath, maintained at $25 \pm 0.1^\circ\text{C}$. Allow 20 to 30 min for temperature equilibrium to be reached.

9.2.8.3 Remove the pycnometer 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 pycnometer 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 pycnometer and nylon 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 Water Bath*, set to operate at $25 \pm 0.1^\circ\text{C}$.

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 $25.0 \pm 0.5^\circ\text{C}$) 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 $25 \pm 0.1^\circ\text{C}$. 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:*

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

where:

η_f = viscosity of formic acid, $\text{kPa} \cdot \text{s}$ (cP),

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

d_f = density of formic acid at $25 \pm 0.1^\circ\text{C}$, $\text{g}/\text{cm}^3 = 1.1975$, 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 .

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

where:

f_i = 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.)

Allowing the cap to remain loose, heat the mixture to $50 \pm 5^\circ\text{C}$, using any convenient method.

9.3.4.2 Tighten the cap thoroughly, and place the sample in the shaker. Agitate until all the nylon is in solution. Then place the bottle in a constant-temperature bath maintained at $25 \pm 0.1^\circ\text{C}$ for not less than 1 h.

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

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

RV	BV _s	Spindle Number for Indicated Speed, r/min		
		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. The same spindle and speed should be used for similar viscosity level polymers.

9.3.4.5 Immerse the spindle and guard of the calibrated Brookfield viscometer and adjust to the immersion mark. (See 9.3.7 for calibration of the Brookfield viscometer.) (The temperature of the spindle and guard should be maintained at $25 \pm 0.1^\circ\text{C}$ by keeping them immersed in a bottle of water in the bath between uses and wiping them dry before using.)

9.3.4.6 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.)

9.3.4.7 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.)

9.3.5 *Calculation of Brookfield Viscosity of Nylon-Formic Acid Solution:*

$$BV_s = (IR, -0.4) \times F \text{ for } 60 \text{ r/min} \quad (8)$$

$$BV_s = (IR) \times F \text{ for } 30 \text{ r/min or } 12 \text{ r/min} \quad (9)$$

where:

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

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

9.3.6 *Calculation of Relative Viscosity of the Nylon-Formic Acid Solution:*

$$RV = (\eta_r/\eta_f) = [(BV_s \cdot C_f)/\eta_f] \quad (10)$$

where:

- R_V = relative viscosity of the nylon-formic acid solution,
- η_f = absolute viscosity of formic acid, 9.3.3.2,
- η_s = absolute viscosity of nylon-formic acid solutions,
- BV_s = Brookfield viscosity of the solution, kPa · s (cP) (9.3.5), and
- C_f = calibration factor (the instruments should be calibrated periodically against standard viscosity oils; see 9.3.7).

9.3.7 *Calibration of Brookfield Viscometer:*

9.3.7.1 Select the standard viscosity oil closest or within 10 % of the absolute viscosity level of the nylon-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 nylon-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 nylon-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.)

9.3.7.2 Determine the instrument reading of the oil following 9.3.4.1. The calibration factor for the instrument is:

$$C_f = TV_o/BV_o \quad (11)$$

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 9.3.5).

9.3.8 *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 a 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.

10. Moisture Content

10.1 The procedure described in 10.2-10.8 is expected to be sufficient to meet most equipment and test operation needs, without sacrificing the quality of measurement intent. In cases

of dispute, in which test results may differ significantly, the following additional conditions are to be specified:

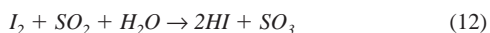
10.1.1 *Reagents*—Fresh containers filled to optimum volumes.

10.1.2 *Sample Weight*—1 g (min), weighed to ± 1 mg.

10.1.3 *Oven Temperature*— $200 \pm 1^\circ\text{C}$.

10.1.4 *Sensitivity* (instruments with automatic calculation)—0.1.

10.2 *Principle*—Samples are heated to vaporize water which is transported by nitrogen carrier gas to the titration cell. The moisture collected is then determined by Karl Fischer Reagent titration based on the reduction of iodine by sulfur dioxide in the presence of water to form sulfur trioxide and hydriodic acid.



10.2.1 End-point detection for calculation of moisture content is made by visual (colorimetric) observation or by instrumented electrochemical methods, which is preferred. Potentiometric or amperometric-detection modes have been found satisfactory in determinations of moisture content. However, for precise measurement of samples with low moisture, combining coulometry with the Karl Fischer Reagent titration is recommended. Unlike the usual Karl Fischer Reagents that include iodine, the coulometric technique electrolytically generates iodine with 10.71 C of generating current corresponding to 1 mg of water in accordance with Faraday's Law.



NOTE 5—Some compounds such as aldehydes and ketones interfere in KFR determination.

10.3 *Apparatus:*

10.3.1 *Heating Unit*, consisting of an oven capable of heating the sample to 300°C , a heating tube, a temperature control unit, and a carrier gas flowmeter desiccating tube;

10.3.2 *Sample Pan* (“Boat”)—Single-use aluminum foil may be used to hand-fashion a suitable boat of sufficient size to contain sample and fit into the heating tube;

10.3.3 *Karl Fischer Titration Unit*, consisting of reagent reservoir, reagent-delivery system, electrode(s), magnetic stirrer, and stirrer bar;

10.3.4 *Precision Balance*, 0.1-mg accuracy; and

10.3.5 *Ordinary Laboratory Equipment*.

10.4 *Reagents:*

10.4.1 *Karl Fischer Reagent*, minimum water equivalent of 5.0 mg of water/mL;

NOTE 6—Hydranal Karl Fischer reagents are recommended for volumetric and coulometric titrations. These reagents do not contain pyridine, are nontoxic, very stable, and have no offensive odor.

10.4.2 *Karl Fischer Diluent Reagent*;

10.4.3 *Nitrogen Gas*, containing less than $5 \mu\text{g/g}$ water as purge gas;

10.4.4 *Silica Gel Granules*, approximately 2 mm, desiccant for drying tube of titration tube assembly;

10.4.5 *Phosphorus Pentoxide or Sicapent*¹⁰ for drying carrier gas;

10.4.6 *Grease*, for sealing ground-glass joints;

10.4.7 *Deionized Water*, or water of equivalent purity; and

10.4.8 *Anhydrous Methanol*.

10.5 *Safety:*

10.5.1 Avoid unnecessary inhalation or contact with reagents.

10.5.2 Wear gloves when handling hot materials.

10.5.3 Observe normal laboratory rules.

10.5.4 If large volume of pyridine is used, work should be conducted under a hood.

10.6 *Standardization of Karl Fischer Reagent:*

10.6.1 Dilute Karl Fischer Reagent with diluent to the desired level. A water equivalency of 1.0 to $1.8 \mu\text{L/g}$ has been found satisfactory for most applications.

10.6.2 Add sufficient methanol to cover electrodes in titration cell.

10.6.3 Titrate to the end point with (diluted) Karl Fischer reagent and record the volume used in millimetres.

10.6.4 Fill a 10- μL syringe with water and record the weight. Inject 5 μL of water into the titration reservoir and reweigh the syringe. Record the difference in weight as *WO*.

10.6.5 Titrate the added water to the Karl Fischer end point and record the volume of Karl Fischer reagent used in millimetres. Subtract the volume recorded in 10.6.3 from the volume of Karl Fischer reagent used to titrate the added water and record as *VKF*.

10.6.6 Calculate the Karl Fischer Equivalent number (*KFE*) as follows:

$$KFE = WO/VKF = \text{mg water/mL of Karl Fischer reagent} \quad (14)$$

10.7 *Sample Preparation:*

10.7.1 Samples for moisture determination should be retained in glass vials with plastic screw-top caps having aluminum foil-backed paperboard liners. The cap must be taped in place after tight closure using two turns of vinyl electric tape.

10.7.2 Vials with foil-lined caps, 107 by 28 mm in diameter, will hold 20 to 30 g of polymer (depending on size and shape of pieces).

10.7.3 Transfer the samples quickly to avoid undue exposure to the atmosphere.

10.8 *Test Procedures:*

10.8.1 *Determination of Blank:*

10.8.1.1 Place the dry, clean sample pan into the heating tube, omitting only the sample.

10.8.1.2 Turn on the heating and titration unit. (Set heating unit to the temperature to be used in running sample.) Adjust carrier-gas-flow to $300 \text{ cm}^3/\text{min}$.

10.8.1.3 Record titration valve as *B*.

10.8.2 *Testing of Sample:*

10.8.2.1 Weigh sample (weight depends on expected water content) into the sample pan (“boat”) and place in the heating tube.

10.8.2.2 Turn on heating unit (see 10.9 for selection of temperature). Adjust carrier-gas-flow to about $300 \text{ cm}^3/\text{min}$. Titration must be started as soon as the sample is placed in the heating tube and not delayed until the selected temperature is reached.

¹⁰ A trademarked product of Merck. Available from E. M. Science Products.

10.8.2.3 Calculate the sample water content as follows:

$$\text{Percent water} = (V - B \times KFE/WS) \times 100 \quad (15)$$

where:

- V = volume of KFR used, mL,
- B = volume of KFR used for blank, mL,
- KFE = Karl Fischer equivalent, and
- WS = weight of sample, mg.

NOTE 7—Some equipment has automatic calculation capability.

10.9 Selection of Optimum Heating Temperature:

10.9.1 Select optimum heating temperature for material to be tested by carrying out tests in several different temperatures to make a curve as shown in Fig. 1.

10.9.1.1 In the range from 1 to 2, the water in the sample is not vaporized sufficiently so that the water content indicated increases in proportion to the temperature.

10.9.1.2 Between 2 and 3, the water content measured appears nearly constant and is considered the optimum heating temperature range for determining moisture content.

10.9.1.3 Water content appears to increase between 3 and 4. This is probably caused by the generation of water due to thermal decomposition of the sample.

10.9.1.4 Measurement of time is also a consideration in selection of the optimum heating temperature.

11. Precision and Bias

11.1 A task group is being formed to establish a precision and bias statement for the moisture test method. See Table 1 for information on repeatability of this test method.

TABLE 1 Repeatability for Relative Viscosity

Material	Average	Sr	SR	r	R
Nylon 6	140.9	2.11	5.86	5.90	16.41
Nylon 66	45.4	1.72	2.16	4.80	6.04

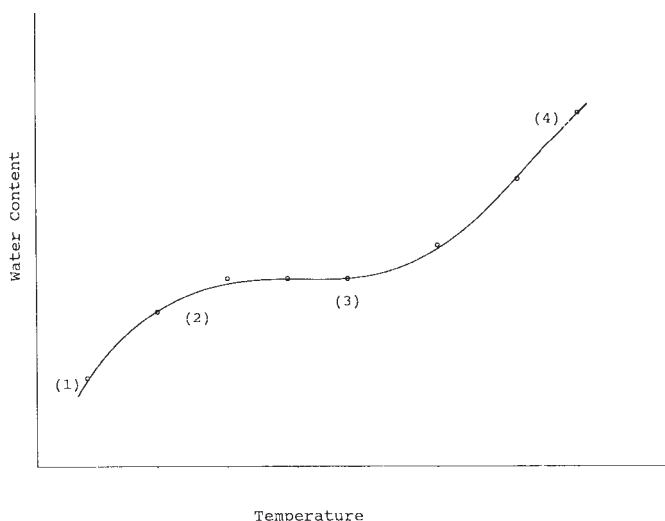


FIG. 1 Optimum Temperature Selection

12. Keywords

12.1 Brookfield viscosity; coulometry; density of nylon polymer-formic acid solution; Karl Fischer; moisture content; optimum heating temperature; pipet viscosity; relative viscosity

SUMMARY OF CHANGES

This section identifies the location of selected changes to these test methods. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of these test methods. This section may also include descriptions of the changes or reasons for the changes, or both.

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(1) Addition of Table 1, Repeatability for Relative Viscosity.

(2) Changed the specification for formic acid density in 9.2.2.4.

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