



Standard Test Methods for Ethylcellulose¹

This standard is issued under the fixed designation D 914; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

- 1.1 These test methods cover the testing of ethylcellulose.
- 1.2 The test procedures appear in the following order:

	Sections
Moisture	4 to 6
Sulfated Ash	7 to 11
Chlorides (as Sodium Chloride)	12 to 16
Ethoxyl Content	20 to 24
Viscosity	25 to 39

1.3 *This standard may involve hazardous materials, operations, and equipment. 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.*

2. Referenced Documents

- 2.1 *ASTM Standards:*
 - D 362 Specification for Industrial Grade Toluene²
 - D 446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers³
 - D 841 Specification for Nitration Grade Toluene⁴
 - D 4794 Test Method for Determination of Ethoxyl or Hydroxyethoxyl Substitution in Cellulose Ether Products by Gas Chromatography⁵
 - E 1 Specification for ASTM Thermometers⁶

3. Purity of Reagents and Materials

3.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, all of the reagents used shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such

specifications are available.⁷ Where such specifications have not been established, reagents of the best grade available shall be used. References to water shall be understood to mean distilled water.

MOISTURE

4. Scope

4.1 This test method covers the determination of the volatile content of ethylcellulose.

5. Significance and Use

5.1 The results of this test are used for calculating the total solids in the sample and, by common usage, all materials volatile at this test temperature are designated as moisture.

5.2 Moisture analysis (along with sulfated ash) is used to calculate the amount of active polymer in the material and shall be considered when determining the amount of ethylcellulose in various functions.

6. Apparatus

- 6.1 *Oven*, gravity convection, capable of maintaining a temperature of $105 \pm 3^\circ\text{C}$.
- 6.2 *Weighing Bottles*.
- 6.3 *Analytical Balance*.

7. Procedure

7.1 Weigh accurately 2 to 5 g of the sample to the nearest 0.001 g into a tared dish (fitted with a lid) and dry for 2 h in an oven at 100 to 105°C. Remove the dish from the oven, cover with a lid, cool in a desiccator, and weigh.

8. Calculation

8.1 Calculate the percent moisture, M as follows:

$$M = (A/B) \times 100 \quad (1)$$

where:

A = mass loss on heating, g, and

¹ These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications, and are the direct responsibility of Subcommittee D01.36 on Cellulose and Cellulose Derivatives.

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² Discontinued 1989; see 1988 *Annual Book of ASTM Standards*, Vol 06.03.

³ *Annual Book of ASTM Standards*, Vol 05.01.

⁴ *Annual Book of ASTM Standards*, Vol 06.04.

⁵ *Annual Book of ASTM Standards*, Vol 06.03.

⁶ *Annual Book of ASTM Standards*, Vol 14.03.

⁷ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

B = sample used, g.

9. Precision and Bias

9.1 *Precision*—Statistical analysis of intralaboratory (repeatability) test results indicates a precision of $\pm 5\%$ at the 95 % confidence level.

9.2 *Bias*—No statement of bias can be made as no suitable reference material is available as a standard.

SULFATED ASH

10. Scope

10.1 This test method covers the determination of the residue on ignition of ethylcellulose after a specimen has been treated with sulfuric acid.

11. Significance and Use

11.1 This test method (along with moisture) is used to calculate the active polymer in the material. It shall be used when testing ethylcellulose in United States government regulated applications. Excessive ash may also affect solution clarity and film properties.

12. Apparatus

12.1 *Muffle Furnace*.

12.2 *Crucibles*, either porcelain, Coors No. 1, or platinum.

13. Reagent

13.1 *Sulfuric Acid* (sp gr 1.84)—Concentrated sulfuric acid (H_2SO_4).

14. Procedure

14.1 Ignite a crucible for 10 to 15 min at $800 \pm 25^\circ\text{C}$, cool in a desiccator, and weigh to the nearest 0.001 g.

14.2 Weigh about 5 g of sample to the nearest 0.001 g (previously dried for 3 h at 105°C) into the crucible. Burn off the bulk of the carbonaceous material directly over a flame. After cooling, add 1 mL of H_2SO_4 in such a way as to moisten the entire ash; then cautiously heat with the burner to dense white fumes. Ignite in a muffle furnace at $800 \pm 25^\circ\text{C}$ until all signs of carbon are gone. Cool in a desiccator and reweigh to the nearest 0.001 g.

15. Calculation

15.1 Calculate the percent ash (as sulfate), C , as follows:

$$C = (A/B) \times 100 \quad (2)$$

where:

A = ash, g, and

B = sample used, g.

16. Precision and Bias

16.1 *Precision*—Statistical analysis of interlaboratory (reproducibility) test results indicates a precision of $\pm 10\%$ at the 95 % confidence level.

16.2 *Bias*—No statement of bias can be made as no suitable reference material is available as a standard.

CHLORIDES (as Sodium Chloride)

17. Scope

17.1 This test method covers the determination of the

chloride content of ethylcellulose.

18. Significance and Use

18.1 Sodium chloride is a major by-product of the ethylcellulose manufacturing process. This test is a measure of the purity of ethylcellulose. Chlorides may also affect solution properties.

19. Apparatus

19.1 *Titration pH Meter*.

19.2 *Mercury-Mercurous Sulfate Reference Electrode*—The electrode uses a potassium sulfate electrolyte to avoid chloride contamination from a chloride electrolyte.

19.3 *Silver-Silver Chloride Electrode*—The electrode is coated with silver chloride periodically. Prepare the electrode by polishing with fine steel wool, briefly soaking it in 5 % potassium cyanide solution, and rinsing it with water. Coat the electrode with silver chloride by electrodeposition from 0.1 N potassium chloride solution using a 3-V dry cell and a platinum wire electrode. Connect the silver electrode to the positive pole of the battery and electrolyze for 20 s; then reverse the connections for 5 s. Repeat these operations twice, and finally, chloridize the silver electrode for 20 s at the positive terminal. Store the silver electrode in 0.1 N potassium chloride solution. Rinse the electrode with water and wipe it with a soft tissue before each titration.

19.4 *Salt Bridge for Reference Electrode*—Fig. 1 shows one configuration in use. Exact dimensions are not important. The salt bridge is used to keep the reference electrode from plugging with the ethylcellulose slurry.

19.5 *Air-Driven Stirrer*.

20. Reagents

20.1 *Ethanol* (95 volume %), undenatured or specially

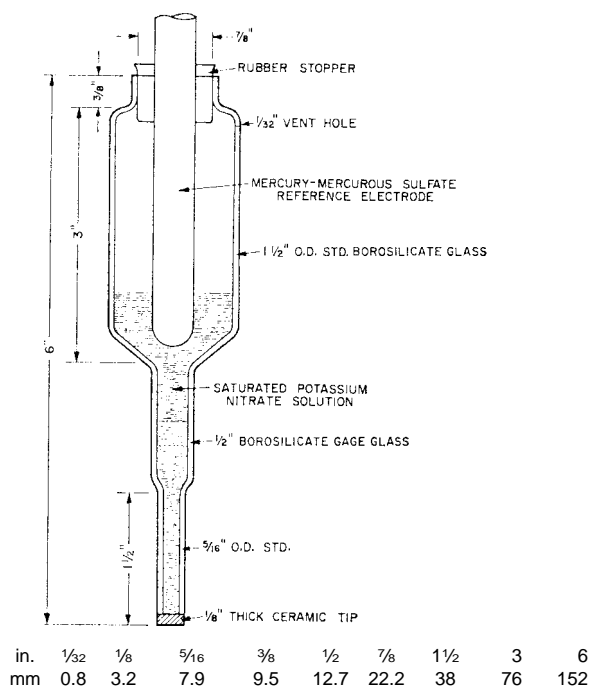


FIG. 1 Salt Bridge and Reference Electrode for Chloride Determination

denatured conforming to Formula 2B of the U.S. Bureau of Internal Revenue.

20.2 *Ethanol-Distilled Water Solvent Mixture* (80 + 20)—Mix 800 g of 2B ethanol with 200 g of water. Add 7.5 g of aerosol OT 100 % surface-active agent per 3000 g of ethanol-water mixture.

20.3 *Potassium Nitrate* (KNO_3) *Solution* (saturated) for salt bridge (Fig. 1).

20.4 *Silver Nitrate, Standard Solution* (0.02 N)—Dissolve 3.4 g of silver nitrate (AgNO_3) in water, dilute to 1 L with water in a volumetric flask, and mix. Weigh exactly 0.5845 g of dry, primary standard sodium chloride (NaCl), dissolve in 25 mL of water, and dilute to 1 L with water in a volumetric flask. Add 10 mL of H_2SO_4 (1 + 16) to each aliquot before titrating. Titrate aliquots of this solution potentiometrically with the AgNO_3 solution. Calculate the normality, N , of the AgNO_3 solution as follows:

$$N = (A/B) \times 0.01 \quad (3)$$

where:

A = 0.01 N NaCl solution added, mL, and

B = AgNO_3 solution required for the titration, mL.

20.5 *Sulfuric Acid* (1 + 16)—Add 1 volume of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) slowly with stirring into 16 volumes of water.

20.6 *Toluene*, meeting the requirements of Specification D 362.

20.7 *Toluene-Ethanol Solvent Mixture* (90 + 10)—Mix 900 g of toluene with 100 g of ethanol.

21. Procedure

21.1 Weigh accurately 10 g of sample to the nearest 0.001 g (previously dried for 2 h at 100 to 105°C) and transfer to a 600-mL beaker containing 200 mL of the toluene-ethanol solvent mixture. Stir with an air-driven stirrer until solution is complete.

21.2 Add 200 mL of the ethanol-water mixture and agitate for 5 min to form a uniform emulsion. Immerse the electrodes in the emulsion using an air-driven stirrer for mixing. Add 10 mL of H_2SO_4 (1 + 16) and agitate for 3 to 4 min to allow the system to reach equilibrium.

21.3 Titrate slowly with the 0.02 N AgNO_3 solution. Make intermittent additions of 0.1 mL. It is advisable to allow longer periods of time between additions of titrant as the end point is approached to avoid passing the equivalence point. Run a blank by the same procedure.

22. Calculation

22.1 Calculate parts per million of chlorides as NaCl , C , as follows:

$$C = [(VN \times 0.05845)/W] \times 1\,000\,000 \quad (4)$$

where:

V = AgNO_3 solution, mL,

N = normality of AgNO_3 solution,

W = sample used, g, and

0.05845 = milliequivalent mass of NaCl .

23. Precision and Bias

23.1 *Precision*—Statistical analysis of interlaboratory (re-

producibility) test results indicates a precision of $\pm 5\%$ at the 95 % confidence level.

23.2 *Bias*—No statement of bias can be made as no suitable reference material is available as a standard.

ETHOXYL CONTENT

24. Scope

24.1 This test method covers the determination of the ethoxyl content of ethylcellulose.

24.2 For an alternative method see Test Method D 4794.

25. Significance and Use

25.1 This test method determines the amount of substituent groups added to the cellulose backbone. The level can greatly affect solution properties, rheology, solubility parameters, and film properties.

26. Apparatus

26.1 *Distillation Apparatus*, as illustrated in Fig. 2, consisting of a boiling flask with a side arm for admission of carbon dioxide (CO_2) or nitrogen, an air condenser with a trap, and a receiver.

26.2 *Oil Bath*, equipped with a heating device, preferably electrical, so that the bath can be maintained at 145 to 150°C.

27. Reagents

27.1 *Bromine Solution*—Dissolve 5 mL of bromine in 145 mL of the potassium acetate ($\text{KC}_2\text{H}_3\text{O}_2$) solution. Prepare the

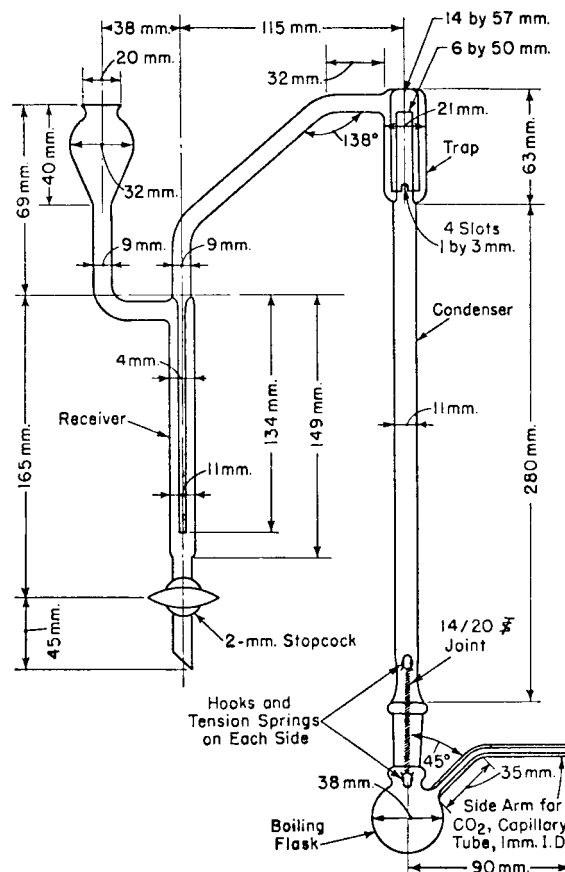


FIG. 2 Distillation Apparatus for Ethoxyl Determination

bromine solution fresh daily in a hood to remove bromine vapors.

27.2 Carbon Dioxide—Pass the CO_2 through a bubble counter and a dry trap, and then through a pressure regulator consisting of a glass tee whose vertical arm extends almost to the bottom of a 10-in. (254-mm) column of water. A screw clamp shall be attached to the thin-walled rubber tubing connecting the horizontal arm of the tee with the boiling flask. This arrangement permits regulation of the flow of gas and allows any excess gas to escape. Nitrogen may be used in place of CO_2 .

27.3 Formic Acid (90 %).

27.4 Gelatin Capsules—Gelatin capsules of a suitable size to hold from 50 to 60 mg of the dried sample will be required.

27.5 Hydriodic Acid (sp gr 1.70)⁸—Hydriodic acid (HI) forms with water a constant-boiling mixture (boiling point 126 to 127°C) that contains 57 % HI. The concentration of HI in the reagent used should be not less than 56.5 %. The blank determination, which is affected primarily by free iodine in the reagent, should require no more than 0.5 mL of 0.1 *N* sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3$) standard solution. If necessary, the acid may be purified by adding to it a small amount of red phosphorus and boiling for 20 to 30 min in a hood while passing a stream of CO_2 into the liquid. Distillation is then carried out behind a safety-glass shield in a hood, using an all-glass apparatus with a slow stream of CO_2 running through the receiver. Under some conditions, the poisonous gas phosphine (PH_3) is formed during distillation, and this may unite with molecular iodine to form phosphorus triiodide (PI_3) which may explode on contact with air. It is, therefore, advisable to keep the current of CO_2 going after the distillation is ended and until the apparatus has cooled; this will prevent air from being sucked into the apparatus. Put the purified HI in small, brown, glass-stoppered bottles, previously swept out with CO_2 , and seal the stoppers with molten paraffin. Store in a dark place. To minimize decomposition of HI due to contact with air, run CO_2 into the bottle while withdrawing portions of the acid for use.

27.6 Phosphorus Slurry (0.06 g/100 mL)—Add about 0.06 g of red phosphorus to 100 mL of water. Shake well before using.

27.7 Potassium Acetate Solution (100 g/L)—Dissolve 100 g of anhydrous potassium acetate ($\text{KC}_2\text{H}_3\text{O}_2$) crystal in 1 L of a solution containing 900 mL of glacial acetic acid and 100 mL of acetic anhydride.

27.8 Potassium Iodide (KI).

27.9 Sodium Acetate Solution (220 g/L)—Dissolve 220 g of anhydrous sodium acetate in water and dilute to 1 L.

27.10 Sodium Thiosulfate, Standard Solution (0.1 *N*)—Dissolve 25 g of sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in 200 mL of water and dilute to 1 L. Use freshly boiled and cooled water. It is preferable to allow the solution to stand for a few days before standardization. Standardize the solution against 0.1000 *N* potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$) solution prepared by dissolving exactly 4.9037 g of $\text{K}_2\text{Cr}_2\text{O}_7$ (National Institute

of Standards and Technology Standard Sample No. 136) in water and diluting to 1 L in a volumetric flask. By means of a buret, measure accurately 35 to 45 mL of the $\text{K}_2\text{Cr}_2\text{O}_7$ solution into a 250-mL Erlenmeyer flask. Add 2 g of KI and 50 mL of sulfuric acid (H_2SO_4 , 1 + 9) and allow to stand for about 5 min. Titrate the liberated iodine with the $\text{Na}_2\text{S}_2\text{O}_3$ solution, using starch indicator solution near the end point. At the end point, the blue color of the starch indicator will be destroyed, leaving the pale green color of the chromate ion. The normality of the $\text{Na}_2\text{S}_2\text{O}_3$ solution should be checked at least once a week. Calculate the normality, *N*, of the $\text{Na}_2\text{S}_2\text{O}_3$ solution as follows:

$$N = (A/B) \times 0.1 \quad (5)$$

where:

A = 0.1000 *N* $\text{K}_2\text{Cr}_2\text{O}_7$ solution added, mL, and

B = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for the titration, mL.

As an alternative procedure, the $\text{Na}_2\text{S}_2\text{O}_3$ solution may be standardized against 0.1 *N* iodine that has been standardized in turn against arsenic trioxide (As_2O_3) (National Institute of Standards and Technology Standard Sample No. 83) or potassium iodate (KIO_3).

27.11 Starch Indicator Solution.

27.12 Sulfuric Acid (1 + 9)—slowly add with stirring 1 volume of concentrated H_2SO_4 (sp gr 1.84) to 9 volumes of water.

28. Procedure

28.1 Dry the sample at 105°C for at least 30 min. Through the condenser, add to the trap in the distillation apparatus (Fig. 2) enough of the phosphorus slurry to make the trap about half full. Add 19 to 20 mL of the bromine solution to the receiver. Accurately weigh from 50 to 60 mg of the dry sample into a gelatin capsule and drop it into the boiling flask. (The weighing should be done as rapidly as possible without sacrificing accuracy because dry ethyl-cellulose picks up moisture rapidly.)

28.2 Add a few small glass beads or chips of clay plate and then 6 mL of HI. Attach the boiling flask at once to the condenser, using a few drops of HI to moisten the ground-glass joint, and then connect the side arm of the flask to the source of CO_2 . Pass a current of CO_2 into the apparatus at the rate of about 2 bubbles per second. Immerse the flask in the oil bath, maintained at 150°C, and heat for 40 min.

28.3 Add 10 mL of sodium acetate solution to a 500-mL Erlenmeyer flask and wash into it the contents of the receiver; dilute to 125 mL with water. Add formic acid dropwise, with swirling, until the brown color of bromine is discharged, and then add about 6 drops more. A total of 12 to 15 drops is usually required. After about 3 min, add 3 g of KI and 15 mL of H_2SO_4 (1 + 9) and titrate immediately with 0.1 *N* $\text{Na}_2\text{S}_2\text{O}_3$ solution to a light straw color. Add a little starch solution and continue the titration to the disappearance of the blue color.

28.4 Blank—Make a blank determination, using the same amounts of reagents and the same procedures as for the specimen. (Usually, about 0.1 mL of 0.1 *N* $\text{Na}_2\text{S}_2\text{O}_3$ solution is required.)

29. Calculation

29.1 Calculate the percent ethoxyl, *E*, as follows:

⁸ Hydriodic acid suitable for ethoxyl determination may also be prepared by the method of Samsel, E. P., and McHard, J. A., *Industrial and Engineering Chemistry, Analytical Edition*, Vol 14, 1942, p. 750.

$$E = [(A - B)N \times 0.00751]/W \times 100 \quad (6)$$

where:

A = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for titration of the sample, mL,

B = $\text{Na}_2\text{S}_2\text{O}_3$ solution required for titration of the blank, mL,

N = normality of the $\text{Na}_2\text{S}_2\text{O}_3$ solution, and

W = sample used, g.

30. Precision and Bias

30.1 *Precision*—Statistical analysis of intralaboratory (repeatability) test results indicates a precision of ± 1 % absolute at the 95 % confidence level.

30.2 *Bias*—No statement of bias can be made as no suitable reference material is available as a standard.

VISCOSITY

31. Scope

31.1 Ethylcellulose viscosity is arbitrarily measured on a 5 % solution in a specific solvent system. The viscosity normally ranges from 7 to 200 cP at 25°C.

31.2 Two test methods for measuring ethylcellulose viscosity are given below. One test method specifies use of the Hercules Horizontal Capillary Viscometer and a solvent system of 80 + 20 toluene-ethanol while the other specifies the Dow Modified Ubbelohde viscometer and solvent systems of 80 + 20 toluene-ethanol for product with an ethoxyl content above 47 % and 60 + 40 toluene-ethanol for ethoxyl content below 47 %.

31.3 The two test methods given below do not give the same resultant viscosity. Therefore, they should be used only in a relative sense and not interchangeably.

31.4 Neither test method is intended to be a referee method.

32. Significance and Use

32.1 This test method determines the relative ability of the polymer to thicken organic solvents and is therefore related to the concentration required in various formulations to achieve the desired finished product viscosity. It can also affect film properties such as tensile strength, flexibility, elongation, and brittleness.

HERCULES HORIZONTAL CAPILLARY VISCOSITY

33. Apparatus

33.1 *Hercules Horizontal Capillary Viscometer*⁹ (Fig. 3)—The tube is surrounded by a glass tube that acts as a water jacket and is connected with it by rubber stoppers at both ends which have proper holes to provide for water inlet and outlet and for insertion of a thermometer. For convenient use, it is desirable to cut the openings in the stopper at the end next to the reservoir bulb. One of the thermostated water lines should extend inside the jacket, nearly to the opposite end, to provide good circulation. The tube and its jacket are attached to a wood

or metal baseboard large enough to support its entire length. The board is in turn hinged at one end to a larger board and is provided with a movable-support arm to hold it at a 45° angle with the base. The bottom board should have adjustable legs so that it can be leveled. The board that supports the capillary tube is equipped with a spirit level. Care must be taken in mounting the tube to see that the capillary is exactly parallel to the board holding the spirit level. The water running through the jacket should come from a constant-temperature bath maintained at $25.0 \pm 0.1^\circ\text{C}$.

33.2 *Shaker, Bottle.*

33.3 *Timer*, calculated in 0.1-s units.

34. Reagents

34.1 *Standard Calibrating Liquid*, 100 cP and 25 C.

34.2 *Ethanol* (95 volume %)—Undenatured or specially denatured conforming to Formula 2B of the U.S. Bureau of Internal Revenue.

34.3 *Mixed Solvent* (80 + 20 toluene ethanol by mass)—Prepare by weighing proportional amounts of toluene and ethanol described in 34.1 and 34.2. Mix thoroughly.

34.4 *Toluene*, meeting the requirements of Specification D 362.

35. Calibration

35.1 Make a mark on the capillary tube about 10 cm from the point where the capillary is attached to the reservoir. Mount the tube in a water jacket, adjust the temperature, elevate the board holding the tube to the 45° position, and fill the reservoir to the etched mark with the standard liquid. Lower the board holding the tube to the baseboard and level the instrument while holding one finger over the end of the capillary tube. Remove the finger when ready and the liquid will start to flow through the capillary. Start the timer when the liquid reaches the etched mark. Determine the place where the second mark is to be placed by noting the distance the liquid has traveled when the elapsed time, in seconds, is equal to the viscosity of the oil in centipoises. It is advisable to use a movable mark, such as a small rubber band, until the exact position of the mark has been rechecked quite carefully; then an etched line can be made.

36. Procedure

36.1 Dry a portion of the sample at 100 to 105°C for 1 h. Weigh 5 g of the sample to the nearest 0.01 g into an 8-oz (250-mL) widemouth bottle. Add 95.00 g of the mixed solvent, cover the neck of the bottle with a sheet of cellophane, and screw on the cap.

36.2 Agitate on a bottle shaker until the solution is complete by visual inspection. After the solution is complete, place the bottle of the solution in a constant-temperature bath maintained at $25 \pm 0.1^\circ\text{C}$ for 1 h.

36.3 Remove the bottle of solution from the bath and check to be sure it is free of air bubbles. Fill the viscometer reservoir to the etched mark, while vertical, with the solution to be tested. Place a finger over the end of the capillary, release the brace, lower the tube, and level.

36.4 Release the finger and determine the time for the liquid to flow from the first mark to the second.

⁹ Detailed drawings of the complete installation can be obtained from Hercules Incorporated, Wilmington, DE as Drawings No. 2173U and 2174U.

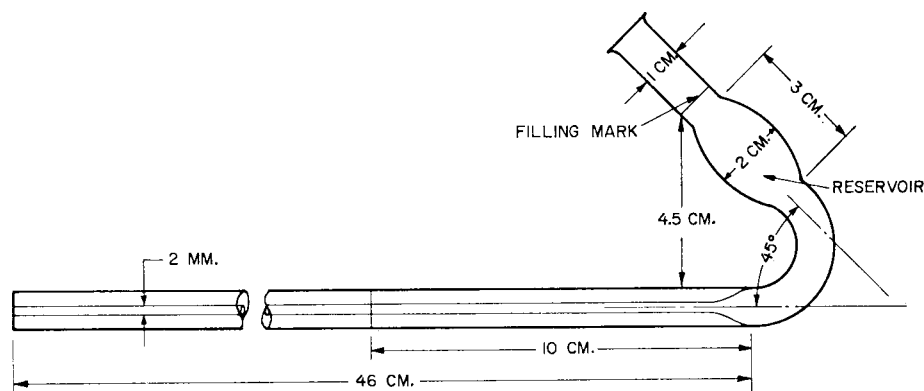


FIG. 3 Hercules Horizontal Capillary Viscometer

37. Calculation

37.1 Calculate the viscosity as follows:

$$N = td/D \quad (7)$$

where:

- N = viscosity, cP,
- t = time of flow for the sample, s,
- d = density of the sample solution at 25°C (0.859), and
- D = density of the oil used for calibration of the viscometer.

38. Report

38.1 Report the viscosity in centipoises, the solution concentration, and the test method (Hercules Horizontal Capillary).

39. Precision and Bias

39.1 *Precision*—Statistical analysis of intralaboratory (repeatability) test results indicates a precision of $\pm 5\%$ at the 95 % confidence level.

39.2 *Bias*—No statement of bias can be made as no suitable reference material is available as a standard.

DOW MODIFIED UBBELOHDE VISCOSITY

40. Apparatus

40.1 *Constant-Temperature Bath*, capable of maintaining a temperature of $25 \pm 0.1^\circ\text{C}$.

40.2 *Shaker, Bottle*.

40.3 *Timer*, calibrated in 0.1-s units.

40.4 *Viscometers*, modified Ubbelohde (Fig. 4).

40.5 *Thermometer*—ASTM Kinematic Viscosity Thermometer, having a range from 74.5 to 79.5 F and conforming to the requirements for Thermometer 45F as prescribed in Specification E 1.

41. Reagents

41.1 *Ethanol* (95 volume %), undenatured or specially denatured conforming to Formula 2B of the U.S. Bureau of Internal Revenue.

41.2 *Toluene*, meeting the requirements of Specification D 841.

41.3 *Standard Calibrating Liquids* (see 42.2).

41.4 *Mixed Solvents* (80 + 20 and 60 + 40 toluene-ethanol by mass)—Mix proportional amounts of toluene and ethyl

alcohol described in 41.1 and 41.2. Mix thoroughly.

42. Calibration

42.1 Select viscosity-calibrating liquids that will yield a minimum efflux time of 200 s in the viscometers to be standardized at $25 \pm 0.1^\circ\text{C}$. See Specification D 446.

42.2 Calibrate one viscometer of each capillary size to be used in the viscosity test work using the calibrating liquid that is indicated in Table 1.

42.3 Charge the Ubbelohde viscometer selected with the appropriate viscometer calibrating liquid by pouring the liquid into the large tube to a level in line with the bottom of the vent-tube entrance. Charge the viscometer in such a manner so that the U-tube at the bottom fills completely without trapping air.

42.4 Place the viscometer in a constant-temperature water bath at $25 \pm 0.1^\circ\text{C}$, immersing the viscometer to cover the functioning areas. Allow the liquid to reach the required temperature. Determine the temperature by placing the thermometer into the liquid. Remove the thermometer. Place a suction bulb over the end of the calibrated flow tube and your finger over the end of the vent tube; then apply suction on the calibrated tube until the liquid level is drawn half way up into the upper bulb of the calibrated tube. Release the suction and finger from the viscometer.

42.5 Time the flow in seconds between the upper and lower calibration lines to the nearest 0.10 s with a timer. If the time in seconds is less than 200, select a viscometer with a smaller capillary and repeat the operation.

42.6 Without recharging the viscometer, make check determinations by repeating the procedures five times. Determine the efflux time by averaging the five values. The range of the individual values should not exceed 0.5 s.

42.7 Calculate the standard viscometer constant, F , for ethylcellulose solutions as follows:

$$F = (V \times d_e) / (d_0 / t) \quad (8)$$

where:

- V = absolute viscosity of the calibrating liquid at 25°C ,
- d_0 = density of the calibrating liquid at 25°C ,
- d_e = density of the sample solution at 25°C (use 0.861 for 80 + 20 and 0.845 for 60 + 40 toluene-ethanol), and
- t = flow time, s.

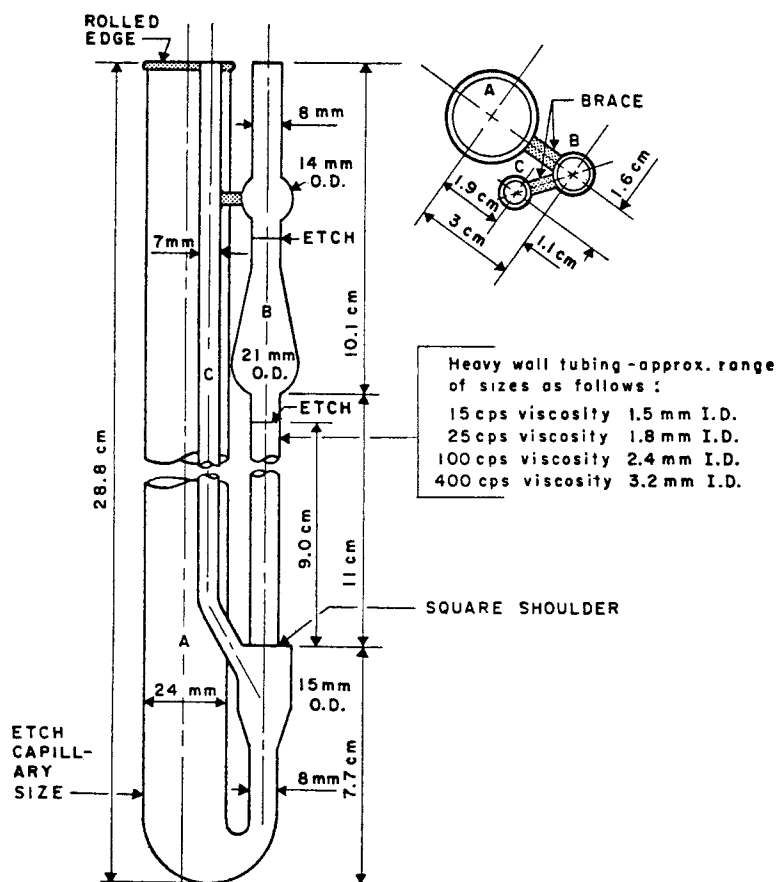


FIG. 4 Dow Modified Ubbelohde Viscometer

TABLE 1 Solutions for Viscosity Determination

Dow Viscometer Designation	Capillary Bore Diameter, mm	Approximate Viscosity, cP at 25 C	Approximate D 446 Design Ubbelohde Viscometers	Cannon Standard Viscometer Calibrating Liquid
P	1.10	...	2	S-60
Q	1.49	10	2C	S-200
R	1.77	20	2B	S-200
S	2.51	80	3C	S-600
T	3.15	150	3B	S-600
U	4.40	...	4C	S-8000

43. Procedure

43.1 Weigh 57 ± 0.02 g of the solvent solution into an 8-oz (250-mL) jar. Weigh 3.0 ± 0.01 g of dry sample and add to the solvent in the jar. Close the jar with a lined screw cap and shake to wet and disperse the sample. Continue to shake for about 15 min on the shaker, or until the sample is completely dissolved.

43.2 Select a Ubbelohde viscometer based on the approximate viscosity range (see 42.2). Add sample solution to the large tube to a level in line with the bottom of the vent tube entrance.

43.3 Place the viscometer in the $25 \pm 0.1^\circ\text{C}$ constant-temperature bath. (Immerse the viscometer to cover the functioning areas.)

43.4 Place a thermometer in the viscometer (see 42.4). After the liquid has reached the required temperature, remove the

thermometer. Charge the viscometer by placing a suction bulb over the end of the calibrated flow tube and then, with a finger over the opening of the vent tube, apply suction on the calibrated tube until the liquid level is drawn half-way up into the upper half of the calibrated tube. Release suction and finger from the tubes.

43.5 Time the flow between the upper and lower calibration lines to the nearest 0.1 s.

43.6 Repeat the above procedure and average the two efflux times. If the time range exceeds 1 s, repeat the operation a third time and average the two values that deviate the least.

44. Calculation

44.1 Calculate viscosity, V_1 in centipoises as follows:

$$V_1 = F \times T \quad (9)$$

where:

F = viscometer constant (see 42.7); and

T = efflux time, s, from 43.6.

45. Report

45.1 Report viscosity in centipoises, solution concentration, and the test method (Dow Modified Ubbelohde).

46. Precision and Bias

46.1 *Precision*—Statistical analysis of intralaboratory (repeatability) test results indicates a precision of $\pm 5\%$ at the 95 % confidence level.

46.2 *Bias*—No statement of bias can be made as no suitable reference material is available as a standard.

47. Keywords

47.1 chlorides; ethoxyl content; ethylcellulose; moisture; sulfated ash; viscosity

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