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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Methods for Methylcellulose[1]

This standard is issued under the fixed designation D 1347; 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 testing of methylcellulose.

1.2 The test methods appear in the following order:

	Sections
Moisture	4 and 5
Ash—as Sulfate	6-8
Chlorides—as Sodium Chloride	9-11
Alkalinity—as Na_2CO_3	12-14
Iron	15-19
Heavy Metals	20-22
Methoxyl Content	23-26
Viscosity:	
Water-Soluble Methylcellulose	27-29
Alkali-Soluble Methylcellulose	30 and 31
pH	32
Solids	33 and 34
Density	35-39

1.3 *This standard does not purport to address 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 hazard statement, see Note 1.

2. Referenced Documents

2.1 *ASTM Standards:*

D 96 Test Methods for Water and Sediment in Crude Oil by Centrifuge Method (Field Procedure)[2]

3. Purity of Reagents

3.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[3] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

3.2 Unless otherwise indicated, references to water shall be understood to mean distilled water.

MOISTURE

4. Procedure

4.1 Transfer 2 to 5 g of the sample, weighed to the nearest 0.01 g, to a tared dish (fitted with a lid) and dry it for 3 h in an oven at 105 ± 3°C. Remove the dish from the oven, cover it with a lid, cool in a desiccator, and weigh.

5. Calculation

5.1 Calculate the percent moisture, *M*, as follows:

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

where:

A = mass loss on heating, g, and
B = sample used, g.

ASH—AS SULFATE

6. Reagent

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

7. Procedure

7.1 Weigh, to the nearest 0.01 g, about 2 g of the sample (previously dried for ½ h at 105°C) and transfer it to a tared platinum crucible. Place it in a muffle furnace at 575 ± 25°C for approximately ½ h, to char the organic material.

7.2 Cool the crucible and add 1 mL of H_2SO_4 so that it completely wets the charred residue. Then cautiously heat it over a small flame to dense white fumes. Place the crucible in a muffle furnace at 575 ± 25°C and leave it there until all signs of carbon are gone (approximately 1 h). Transfer the specimen to a desiccator until cool, then weigh.

8. Calculation

8.1 Calculate the percent of ash, *C*, as follows:

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

where:

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[1] These test methods are under the jurisdiction of ASTM Committee D-1 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] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *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. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

A = ash, g, and
B = sample used, g.

CHLORIDES—AS SODIUM CHLORIDE

9. Reagents

9.1 *Ferric Alum Indicator Solution*—Add 100 g of ferric aluminum sulfate $(Fe_2SO_4)_3 \cdot (NH_4)_2SO_4 \cdot 24H_2O$ to 250 mL of water. Heat it to boiling and add HNO_3 (sp gr 1.42) slowly until the red color is removed. This will usually require about 6 to 15 mL of HNO_3. Filter the solution and store it in a glass bottle.

9.2 *Potassium Thiocyanate, Solution, Standard* (0.1 *N*)—Dissolve 10 g of potassium thioagonate (KCNS) in 1 L of water. By means of a pipet, measure 25 mL of 0.1000 *N* $AgNO_3$ solution into a 400-mL beaker. Add 100 mL of water, 10 mL of nitric acid (NHO_3, sp gr 1.42) and 5 mL of ferric alum indicator solution. Titrate with the KCNS solution, while stirring, until a faint persistent red color is produced. Calculate the normality of the KCNS solution, *N*, as follows:

$$N = (A/B) \times 0.1 \quad (3)$$

where:
A = 0.100 *N* $AgNO_3$ solution added, mL, and
B = KCNS solution required for the titration, mL.

9.3 *Silver Nitrate, Solution, Standard* (0.1 *N*)—Grind silver nitrate ($AgNO_3$) crystals fine enough to pass through a 850-µm (No. 20) sieve, and then dry for 2 h at 110°C. Prepare a 0.1000 *N* solution by dissolving 16.989 g of dry $AgNO_3$ in chloride-free water and diluting it to 1 L in a volumetric flask.

10. Procedure

10.1 Weigh, to the nearest 0.01 g, about 1.0 g of the sample (previously dried for ½ h at 100 to 105°C) and transfer to a 500-mL wide-mouth Erlenmeyer flask. Add 250 mL of hot water to the flask and swirl it for a few minutes, then cool to dissolve.

10.2 Add 5 mL of 0.1000 *N* $AgNO_3$ solution and 5 mL of ferric alum indicator solution, and then back-titrate with 0.1 *N* KCNS solution to the first appearance of a faint pink color.

11. Calculation

11.1 Calculate the percent of chlorides, *C*, as sodium chloride (NaCl) as follows:

$$C = ([(AB - CD) \times 0.0585]/E) \times 100 \quad (4)$$

where:
A = $AgNO_3$ solution added, mL,
B = normality of the $AgNO_3$ solution,
C = KCNS solution required to back-titrate the excess $AgNO_3$, mL,
D = normality of the KCNS solution, and
E = sample used, g.

ALKALINITY—AS SODIUM CARBONATE, ANHYDROUS

12. Reagents

12.1 *Methyl Purple Indicator Solution.*

12.2 *Sulfuric Acid, Standard* (0.01 *N*)—Prepare and standardize a 0.01 *N* solution of sulfuric acid (H_2SO_4).

13. Procedure

13.1 Weigh, to the nearest 0.01 g, about 1.0 g of the sample (previously dried for ½ h at 100 to 105°C) and transfer it to a 500-mL wide-mouth Erlenmeyer flask. Add 250 mL of hot water to the flask and swirl it for a few minutes, then cool to dissolve.

13.2 Add 4 drops of methyl purple indicator to the flask solution and titrate to a blue-gray end point with 0.01 *N* H_2SO_4.

14. Calculation

14.1 Calculate the percent of alkalinity, *D*, as anhydrous sodium carbonate (Na_2CO_3) as follows:

$$D = [(AB \times 0.053)/C] \times 100 \quad (5)$$

where:
A = H_2SO_4 required for titration of the sample, mL,
B = normality of the H_2SO_4, and
C = sample used, g.

IRON

15. Apparatus

15.1 *Photometer*—Any photoelectric filter photometer or spectrophotometer suitable for measurements at 430 nm.

15.2 *Kjeldahl Flasks*, calibrated to contain 30 mL, and made of heat- and chemical-resistant glass.

16. Reagents

16.1 *Ammonium Hydroxide* (sp gr 0.90)—Concentrated ammonium hydroxide (NH_4OH).

16.2 *Buffer Solution*—Dissolve 20 g of sodium bicarbonate ($NaHCO_3$) and 10 g of sodium carbonate (Na_2CO_3) in water and dilute to 1 L.

16.3 *Disodium-1,2-Dihydroxybenzene-3,5-Disulfonate Solution*[4]—Prepare an aqueous solution containing 25 g/L.

16.4 *Hydrogen Peroxide* (30 %)—Concentrated hydrogen peroxide (H_2O_2).

16.5 *Iron, Solution, Standard* (0.0001 g Fe/mL)—Dissolve 0.01 g of iron powder containing not less than 99.9 % Fe in hydrochloric acid (HCl, sp gr 1.19). Oxidize the solution with bromine water and expel the excess by boiling. Dilute to 1 L in a volumetric flask.

16.6 *Phenolphthalein Indicator Solution*.

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

16.8 *Sulfuric Acid* (1 + 4)—Carefully mix 1 volume of H_2SO_4 (sp gr 1.84) with 4 volumes of water, adding the H_2SO_4 gradually while mixing.

17. Preparation of Calibration Curve

17.1 Following the procedure given in Section 18, and using varied amounts of the standard iron solution prepared in accordance with 16.1, prepare a calibration curve showing iron content in parts per million and the corresponding photometer readings.

[4] A suitable prepared solution of this reagent, known as Tiron, is available from the La Motte Chemical Products Co., Chestertown, MD.

18. Procedure

18.1 Weigh approximately 2 g of the sample of methylcellulose to the nearest 0.01 g, and transfer by means of a funnel to a Kjeldahl flask. Place the flask at a 20° angle in a furnace at 600°C, or on a microdigestion rack equipped with electric heating elements, and heat until some charring of the methylcellulose has taken place. (Care must be taken not to char too much.) Remove and allow to cool.

18.2 Add 3 mL of concentrated H_2SO_4 to the flask. Place the flask on the digestion rack and digest. Cool, and add H_2O_2 dropwise until the solution is clear. Heat over a Meker burner to a volume of 2 mL. Cool, and wash the sides of the flask with water. Add 3 drops of phenolphthalein indicator solution. Add NH_4OH to a red end point. Wash the neck of the flask. The solution should be clear and not greater than 20 mL in volume.

18.3 Add 2 mL of the color-forming solution described in 16.3 and mix. Dilute to near the mark with buffer solution and mix thoroughly. Adjust the solution to a pH of 7 by adding NH_4OH or H_2SO_4 (1 + 4), and then dilute to the mark. Transfer a small portion to an absorption cell and determine the photometer reading at 430 nm.

19. Calculation

19.1 Read the iron content, in parts per million, directly from the calibration curve (Section 17).

HEAVY METALS

20. Apparatus

20.1 *Nessler Tubes*, 50-mL.

20.2 *Volumetric Flasks*, 50-mL.

21. Reagents

21.1 *Acetic Acid* (6 + 94)—Mix 6 volumes of glacial acetic acid with 94 volumes of water.

21.2 *Ammonium Hydroxide* (1 + 5)—Mix 1 volume of concentrated ammonium hydroxide (NH_4OH, sp gr 0.90) with 5 volumes of water.

21.3 *Hydrochloric Acid* (1 + 3)—Mix 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 3 volumes of water.

21.4 *Hydrogen Sulfide Solution* (Saturated)—Saturate cold water with hydrogen sulfide (H_2S).

21.5 *Lead, Solution, Standard* (1 mL = 0.1 mg Pb)—Dissolve 0.1598 g of lead nitrate ($Pb(NO_3)_2$) in 100 mL of water to which has been added 1 mL of concentrated nitric acid (HNO_3, sp gr 1.42). Dilute to 1000 mL with water.

21.6 *Lead, Solution, Standard* (1 mL = 0.01 mg Pb)—Dilute 10.0 mL of $Pb(NO_3)_2$ solution (1 mL = 0.1 mg Pb) to 100 mL with water. This solution must be freshly prepared. When 0.1 mL of this standard lead solution is employed to prepare the standard to be compared with a solution of 1 g of the substance being tested, the comparison solution thus prepared contains the equivalent of one part of lead per million parts of the substance tested.

21.7 *Phenolphthalein Indicator Solution*.

22. Procedure

22.1 Add 5 mL of HCl (1 + 3) to the residue in the platinum crucible that was used in the sulfate ash determination (Sections 6 and 7). Digest the residue by slowly boiling for a few minutes over a small flame. Transfer the contents of the crucible to a 50-mL volumetric flask, using about 25 mL of water to rinse the crucible. Neutralize with NH_4OH (1 + 5) to a phenolphthalein end point and dilute to 50 mL.

22.2 Transfer a 25-mL aliquot of the solution to a 50-mL Nessler tube, and add 2 mL of acetic acid (6 + 94) and 10 mL of a saturated solution of H_2S. Mix, allow to stand for 10 min, and compare with a standard lead solution to which H_2S has been added.

22.3 Report the lead content in parts per million.

METHOXYL CONTENT

23. Apparatus

23.1 *Distillation Apparatus* (Fig. 1), 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.

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

24. Reagents and Materials

24.1 *Bromine Solution*—Dissolve 5 mL of bromine in 145 mL of the potassium acetate solution. Prepare the bromine solution fresh daily in a hood to remove bromine vapors.

24.2 *Carbon Dioxide*—This may be obtained by the interaction of marble and HCl (1 + 1) in a Kipp generator or, preferably, from a cylinder of the gas equipped with a suitable needle valve. The CO_2 shall be passed through a bubble

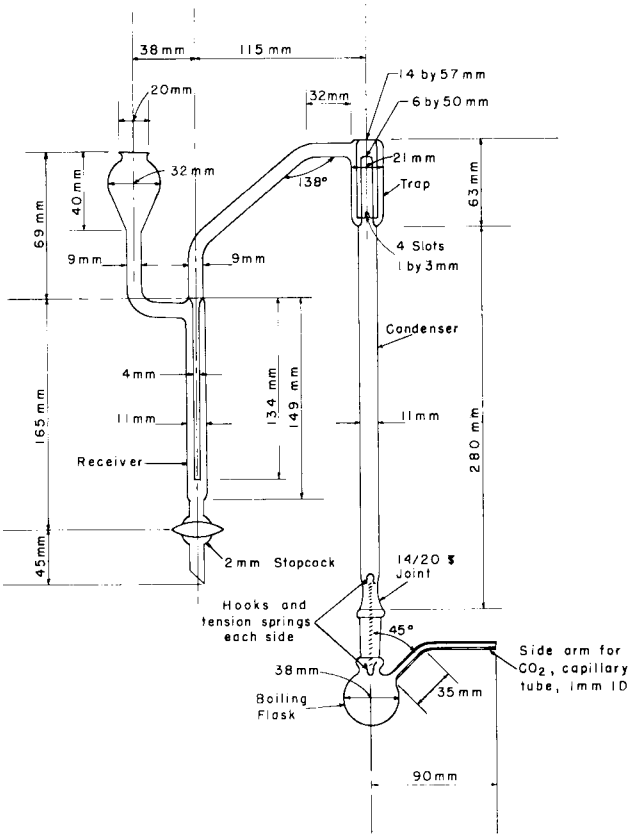

FIG. 1 Distillation Apparatus for Methoxyl Determination

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 254-mm column of water. A screw clamp is 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.

24.3 *Formic Acid* (HCOOH, 90 %).

24.4 *Gelatin Capsules*—Gelatin capsules of a suitable size to hold 50 to 60 mg of the dried specimen will be required.

24.5 *Hydriodic Acid* (57 %, sp gr 1.70)—Hydriodic acid (HI) forms with water a constant-boiling mixture (boiling point 126 to 127°C) which 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 not more than 0.5 mL of 0.1 *N* sodium thiosulfate ($Na_2S_2O_3$) solution.[5] 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 shall then be 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.

NOTE 1—**Warning:** 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.

24.6 *Potassium Acetate Solution*—Dissolve 100 g of anhydrous potassium acetate crystals in 1 L of a solution containing 900 mL of glacial acetic acid and 100 mL of acetic anhydride.

24.7 *Potassium Iodide* (KI).

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

24.9 *Sodium Thiosulfate Solution, Standard* (0.1 *N*)—Dissolve 25 g of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$) 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 ($K_2Cr_2O_7$) solution prepared by dissolving exactly 4.9037 g of $K_2Cr_2O_7$ (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 $K_2Cr_2O_7$ solution into a 250-mL Erlenmeyer flask. Add 2 g of KI and 50 mL of H_2SO_4 (1 + 9) and allow to stand for about 5 min. The flask should be stoppered during the standing period to avoid loss of iodine. Titrate the liberated iodine with the $Na_2S_2O_3$ 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 $Na_2S_2O_3$ solution should be checked at least once a week. Calculate the normality of the $Na_2S_2O_3$ solution, *N*, as follows:

$$N = (A/B) \times 0.1 \quad (6)$$

where:

A = 0.1000 N $K_2Cr_2O_7$ solution added, mL, and
B = $Na_2S_2O_3$ solution required for the titration, mL.

As an alternative procedure, the $Na_2S_2O_3$ solution may be standardized against 0.1 *N* iodine solution 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).

24.10 *Starch Indicator Solution.*

24.11 *Sulfuric Acid* (1 + 9)—Carefully mix 1 volume of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) with 9 volumes of water, adding the H_2SO_4 gradually while mixing.

25. Procedure

25.1 Dry the sample at 105°C for at least 30 min. Through the condenser, add to the trap in the distillation apparatus (Fig. 1) enough distilled water to make the trap about half full. Add 8 to 9 mL of bromine solution to the receiver. Weigh 50 to 60 mg of the dry sample, to the nearest 0.1 mg, into a gelatin capsule and drop it into the boiling flask. (The weighing should be done as rapidly as possible without sacrificing accuracy, since dry methylcellulose picks up moisture rapidly.)

25.2 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/s. Immerse the flask in the oil bath, maintained at 150°C, and heat for 50 min.

25.3 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 HCOOH 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* $Na_2S_2O_3$ solution to a light straw color. Add a little starch solution and continue the titration to the disappearance of the blue color.

25.4 *Blank*—Make a blank determination, using the same amounts of reagents and the same procedure as for the sample. (Usually, about 0.1 mL of 0.1 *N* $Na_2S_2O_3$ solution is required.)

26. Calculation

26.1 Calculate the percent of methoxyl, *M*, as follows:

$$M = [((A - B)C \times 0.00517)/D] \times 100 \quad (7)$$

where:

A = $Na_2S_2O_3$ solution required for titration of the sample, mL,

[5] Hydriodic acid suitable for methoxyl determination may be prepared by the method of Samsel, E. P., and McHard, J. A., *Industrial and Engineering Chemistry*, Analytical Edition, Vol 14, 1942, p. 750. Hydriodic acid available from Merck & Co., WBC 220, P.O. Box 2000, Rahway, NJ 07065 under the designation "For Methoxyl Determination" has been found satisfactory for this purpose.

B = $Na_2S_2O_3$ solution required for titration of the blank, mL,
C = normality of the $Na_2S_2O_3$ solution, and
D = sample used, g.

VISCOSITY OF WATER-SOLUBLE METHYLCELLULOSE

27. Apparatus

27.1 *Viscometer*, Fig. 2(a) or (b).

NOTE 2—If a viscometer has been repaired, it should be recalibrated before it is used again. Even minor repairs can cause significant changes in the *K* value.

27.2 *Mechanical Stirrer*.

28. Procedure

28.1 Determine the moisture content of a portion of the sample. Since cellulose and its water-soluble derivatives are hygroscopic, exposure of the sample to the atmosphere should be kept to a minimum. Changes in moisture content can introduce large errors into the accuracy of the determination and this step should never be omitted if precise results are desired.

28.2 Correcting for the moisture content, weigh out enough of the sample of undried methylcellulose to give 2.000 g of solids, calculated as follows:

$$\text{Weight of specimen, g} = [100/(100 - \text{moisture content, \%})] \times 2 \quad (8)$$

28.3 Place the sample in an 8-oz (250-cm^3) wide-mouth bottle. This weighing step is critical in obtaining good checks and should be done on a good balance sensitive to 1 mg. Weights to the nearest 0.01 g will be sufficiently accurate.

28.4 Add 98.0 g of hot water (85 to 90°C) to the 8-oz bottle containing the 2-g specimen of methylcellulose.

28.5 Agitate with a mechanical stirrer for 10 min, then place the bottle in an ice bath (0 to 5°C) until solution is complete. Equip the stirrer assembly with a one-hole stopper or bottle cap so that no water vapor is lost during agitation. For precision, it is preferable to deair the solution by some means such as centrifuging.

28.6 When solution is complete, as evidenced by the absence of partially swollen or undispersed particles, determine the viscosity in a methylcellulose viscometer at 20 ± 0.1°C. Observe two precautions at this point: (*1*) the solution shall be essentially free of air bubbles, and (*2*) the temperature of the material in the tube shall be checked to make certain that it is actually at the bath temperature.

28.7 The methylcellulose viscosity tube (Fig. 2) consists of three parts: (*1*) a large filling tube with a reservoir at its lower extremity, *A*; (*2*) the orifice tube, *B*; and (*3*) an air vent to the reservoir, *C*. When *B* is filled, close *C* to prevent the sucking of air bubbles into the orifice tube.

28.8 Before the specimen is allowed to flow through the orifice for the viscosity determination, open the vent, *C*, so that

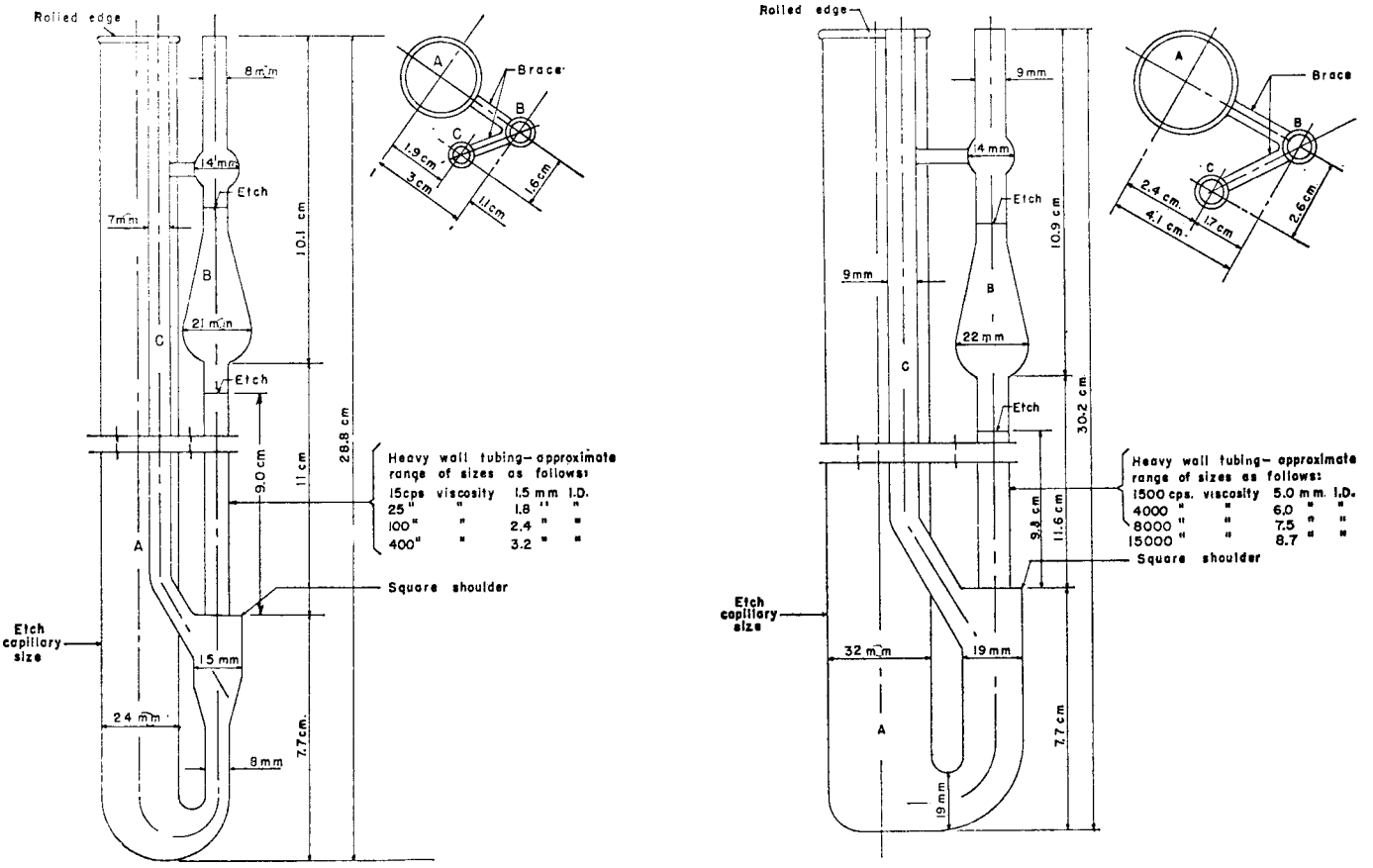

FIG. 2 Methylcellulose Viscometers

the column of solution in *B* will flow into the reservoir against atmospheric pressure. Failure to open *C* before running will cause false values in the viscosity results.

29. Calculation

29.1 Calculate the viscosity as follows:

$$V = Kdt \quad (9)$$

where:

V = viscosity, cP,
K = viscometer constant (Note 2),
d = density of the methylcellulose solution at 20/20°C (Note 3), and
t = time for the solution to pass from the upper to the lower mark of the viscometer, s.

NOTE 3—The viscometer constant is determined by passing a standard oil of known viscosity through the tube and determining the time of flow. The above equation can then be solved for *K*.

NOTE 4—For routine work, the density of solutions of methylcellulose may be assumed to be 1.00.

VISCOSITY OF ALKALI-SOLUBLE METHYLCELLULOSE

30. Reagents

30.1 *Sodium Hydroxide Solution* (40 g NaOH/L)—Dissolve 40 g of sodium hydroxide (NaOH) in carbon dioxide (CO_2)-free water and dilute to 1 L.

31. Procedure

31.1 Proceed as directed in Sections 28 and 29, except add 98.0 g of the NaOH solution to the sample, instead of adding hot water in accordance with 28.3.

pH

32. Procedure

32.1 Determine the pH of the viscosity solution from Section 28, using any suitable pH meter.

SOLIDS

33. Apparatus

33.1 *Oil Tubes*, graduated, 100-mL, tapered, conforming to the requirements prescribed in 3.2 and Fig. 1 of Test Methods D 96.

33.2 *Centrifuge*, capable of whirling filled centrifuge tubes at a speed that will produce a centrifugal force of 725 times gravity.

34. Procedure

34.1 Add 1.50 g of bone-dry methylcellulose to 148.5 g of 90°C water in a 2¼ by 6-in. (32 by 152-mm) bottle and agitate vigorously for about 15 min, or until the material has become finely divided. Place an ice bath around the bottle and agitate the mixture until the solution is effected. (This usually requires about 15 min.)

34.2 Place 100 mL of this 1 % solution in an oil tube, cool to 10°C, and centrifuge at 725 times gravity for 5 min. The solution temperature shall be below 20°C when finished. Read the volume percent of solids from the graduations on the tube.

DENSITY

35. Scope

35.1 This test method covers the determination of the bulk density of methylcellulose.

36. Summary of Test Method

36.1 A weighed amount of methylcellulose is transferred to a 250-mL graduated cylinder and the graduate vibrated to settle the powder.

37. Apparatus

37.1 *Vibrator*—A magnetic-type electric vibrator attached to the vertical support rod of a ring stand approximately 1 ft (0.3 m) above the base. A condenser clamp of sufficient size to hold a 250-mL graduated cylinder also shall be attached to the above rod. The base of the stand should be weighted.

38. Procedure

38.1 Place 50.0 g of methylcellulose in a 250-mL graduated cylinder and place the cylinder in the condenser clamp. Turn on the vibrator and allow the cylinder to vibrate for 3 min. Record the level (in millilitres) to which the specimen has compacted.

38.2 Alternatively, the specimen may be compacted manually. Tap it on a hard surface by dropping the cylinder repeatedly from a height of about 1 in. (25 mm), until the volume of the specimen remains constant. In order to prevent cylinder breakage, cover the tapping surface with a ⅛ to ¼-in. (3 to 6-mm) thick rubber sheet, or use a plastic graduated cylinder.

39. Calculation

39.1 Calculate the density, *D*, in grams per millilitre as follows:

$$D = 50/\text{observed reading, mL} \quad (10)$$

40. Keywords

40.1 ball drop; cellulose esters; viscosity