



Standard Test Methods for Hydroxypropyl Methylcellulose¹

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^{ε1} NOTE—Editorial changes were made throughout in October 1995.

1. Scope

1.1 These test methods cover the testing of hydroxypropyl methylcellulose.

1.2 The test procedures appear in the following order:

	Sections
Moisture	4 to 6
Ash (as Sulfate)	7 to 10
Chlorides (as NaCl)	11 to 14
Alkalinity (as Na ₂ CO ₃)	15 to 18
Iron	19 to 24
Heavy Metals	25 to 29
Methoxyl Content	30 to 35
Hydroxypropoxyl Content	36 to 41
Viscosity	42 to 46
pH	47
Solids	48 to 51
Density	52 to 56

1.3 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses 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 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

E 70 Test Method for pH of Aqueous Solutions With the Glass Electrode³

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.⁴ 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. Scope

4.1 This test method covers the determination of the volatile content of hydroxypropyl methylcellulose and, by common usage, designated moisture.

5. Procedure

5.1 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 for 2 h in an oven at 100 to 105°C with lid removed. Remove the dish from the oven, cover with a lid, cool in a desiccator, and weigh.

6. Calculation

6.1 Calculate the percent of moisture as follows:

$$\text{Moisture, \%} = (A/B) \times 100 \quad (1)$$

where:

A = mass loss on heating, and

B = sample used, g.

ASH—AS SULFATE

7. Scope

7.1 This test method covers the determination of the amount of residue left from igniting a sample of hydroxypropyl methylcellulose after being moistened with sulfuric acid.

8. Reagents

8.1 *Nitric Acid (sp gr 1.42)*—Concentrated nitric acid (HNO₃).

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 Cellulosics.

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² *Annual Book of ASTM Standards*, Vol 05.01.

³ *Annual Book of ASTM Standards*, Vol 15.05.

⁴ *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.

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

9. Procedure

9.1 Weigh to the nearest 0.01 g about 2 g of the sample (previously dried for ½ h at 105°C) into a tared Coors No. 1, high-form, porcelain crucible. Add 5 drops of H_2SO_4 around the inside surface of the crucible. Place the crucible inside of a loosely fitting aluminum ring (approximately 1¼ in. (32 mm) high, with ¼-in. (6.4-mm) sidewall, and 1¾-in. (44-mm) inside diameter, cut from a piece of aluminum pipe) on a hot plate. Loosely cover with a crucible cover. Carefully char the hydroxypropyl methylcellulose until all the volatiles are removed.

9.2 Cool the crucible, add 1 ml of H_2SO_4 and 2 ml of HNO_3 so that it completely wets the charred residue. Cautiously heat to dense white fumes on a hot plate. Place the uncovered crucible in a muffle furnace at 600°C and ignite until all the carbon is gone (for about 1 h). Transfer to a desiccator until cool, then weigh. (Save the residue for the Heavy Metals determination.)

10. Calculation

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

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

where:

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

CHLORIDES—AS SODIUM CHLORIDE

11. Scope

11.1 This test method covers the determination of the total percent of chloride (bromide included if present) calculated as sodium chloride (NaCl) in hydroxypropyl methylcellulose. The sample is dispersed and the chloride titrated volumetrically with 0.100 *N* silver nitrate solution.

12. Reagents

12.1 *Ferric Alum Indicator Solution*—Add 100 g of ferric ammonium sulfate $\text{FeNH}_4(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}$ to 250 mL of water. Heat to boiling and add NH_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 in a glass bottle.

12.2 *Nitric Acid (sp gr 1.42)*—Concentrated nitric acid (HNO_3).

12.3 *Potassium Thiocyanate Standard Solution (0.1 N)*—Dissolve 10 g of potassium thiocyanate (KCNS) in 1 L of water. By means of a pipet, measure 25 mL of 0.100 *N* silver nitrate (AgNO_3) solution into a 400-mL beaker. Add 100 mL of water, 10 mL of HNO_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, *N*, of the KCNS solution 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.

12.4 *Silver Nitrate-Standard Solution (0.100 N)*—Grind silver nitrate (AgNO_3) crystals fine enough to pass through a No. 20 (850- μm) sieve and then dry for 2 h at 110°C. Prepare a 0.100 *N* solution by dissolving 16.989 g of dry AgNO_3 in chloride-free water and diluting to 1 L in a volumetric flask.

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 to a 500-mL, wide-mouth Erlenmeyer flask. Add 250 mL of hot water and swirl for a few minutes; then cool to dissolve.

13.2 Add 5 mL of 0.100 *N* AgNO_3 solution and 5 mL of ferric alum indicator solution, and back-titrate with 0.1 *N* KCNS solution to the first appearance of a faint pink color.

14. Calculation

14.1 Calculate the percent of chlorides as NaCl as follows:

$$\text{Chlorides, \%} = [(AB - CD) \times 0.0585] / E \times 100 \quad (4)$$

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

15. Scope

15.1 This test method covers the determination of the total alkalinity of hydroxypropyl methylcellulose expressed as sodium carbonate (Na_2CO_3).

16. Reagents

16.1 *Methyl Purple Indicator Solution*.

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

17. Procedure

17.1 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, widemouth Erlenmeyer flask. Add 250 mL of hot water and swirl for a few minutes; then cool to dissolve.

17.2 Add 4 drops of methyl purple indicator solution and titrate to the first faint pink color with 0.01 *N* H_2SO_4 .

18. Calculation

18.1 Calculate the percent alkalinity as Na_2CO_3 , *S*, as follows:

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

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

19. Scope

19.1 This test method covers the determination of total iron

content in samples of hydroxypropyl methylcellulose. The iron is converted to ferric sulfate which reacts with the indicator to form a pink color that can be quantitatively measured.

20. Apparatus

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

20.2 *Kjeldahl Flasks*—Calibrated to contain 50 mL, and made of heat- and chemical-resistant glass.

21. Reagents

21.1 *Ammonium Hydroxide (sp gr 0.90)*—Concentrated ammonium hydroxide (NH₄OH).

21.2 *Buffer Solution*—Dissolve 20 g of sodium bicarbonate (NaHCO₃) and 10 g of sodium carbonate (Na₂CO₃) in water and dilute to 1 L.

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

21.4 *Hydrochloric Acid (sp gr 1.19)*—Concentrated hydrochloric acid (HCl).

21.5 *Hydrogen Peroxide (30 %)*—Concentrated hydrogen peroxide (H₂O₂).

21.6 *Iron Standard Solution (0.0001 g Fe/ml)*—Dissolve 0.01 g of iron powder containing not less than 99.9 % iron in 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.

21.7 *Phenolphthalein Indicator Solution (1 g/100 mL)*—Dissolve 1 g of phenolphthalein in 100 mL of ethanol (95 %).

21.8 *Sulfuric Acid (sp gr 1.84)*—Concentrated sulfuric acid (H₂SO₄).

21.9 *Sulfuric Acid (1+4)*—Carefully mix 1 volume of H₂SO₄ (sp gr 1.84) with 4 volumes of water, adding the H₂SO₄ gradually while mixing.

22. Preparation of Calibration Curve

22.1 Following the procedure given in Section 23, and using varied amounts of the standard iron solution prepared in accordance with 21.6, prepare a calibration curve showing iron content in parts per million and the corresponding photometer readings.

23. Procedure

23.1 Weigh to the nearest 0.01 g about 2 g of the sample (previously dried for ½ h at 100 to 105°C). Transfer by means of a funnel to a Kjeldahl flask. Place the flask at a 20° angle in the furnace at 500°C and heat until some charring of the sample has taken place. (Care must be taken not to char too much.) Remove and allow to cool.

23.2 Add 3 mL of H₂SO₄ to the flask. Place on the digestion rack and digest. Cool and add H₂O₂ 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 NH₄OH to a red end point. Wash the neck of the flask. The solution should be clear and not greater than 20 mL in volume.

23.3 Add 2 mL of the color-forming solution described in 21.3, and mix. Adjust pH to 7.0 and then dilute to mark with buffer. Transfer a small portion to an absorption cell and determine the photometer reading at 480 nm.

23.4 *Blank*—Make a blank determination, using the same amount of reagents and the same procedure as for the sample.

24. Calculation

24.1 Read the iron content, in parts per million, directly from the calibration curve (Section 22). Subtract the parts per million of iron due to iron in the blank.

HEAVY METALS

25. Scope

25.1 This test method covers the determination of whether or not the heavy metals content of hydroxypropyl methylcellulose is below a given level based on a lead standard.

26. Summary of Test Method

26.1 The ash residue from the sulfated ash test is digested with dilute hydrochloric acid. A standard containing a known amount of lead is prepared, and the heavy metals content is determined qualitatively by comparing the sample to the standard.

27. Apparatus

27.1 *Nessler Tubes*, 50-mL.

27.2 *Volumetric Flasks*, 50-mL.

28. Reagents

28.1 *Acetic Acid*—Glacial acetic acid.

28.2 *Ammonium Hydroxide (sp gr 0.90)*—Concentrated ammonium hydroxide (NH₄OH).

28.3 *Ammonium Hydroxide (2+3)*—Dilute 400 mL of NH₄OH (sp gr 0.90) with sufficient water to make 1000 mL.

28.4 *Buffer Solution*—Dissolve 60 mL of acetic acid in about 500 mL of water, add 10 mL of NH₄OH, and dilute to 1 L.

28.5 *Hydrochloric Acid (1+2)*—Dilute 1 volume of concentrated hydrochloric acid (HCl, sp gr 1.19) with 2 volumes of water.

28.6 *Hydrogen Sulfide TS*—Saturate a convenient volume of water with hydrogen sulfide (H₂S) in a narrow-neck, glass-stoppered, amber bottle. This solution must be made fresh.

28.7 *Hydroxylamine Hydrochloride Solution (200 g/L)*—Dissolve 20 g of hydroxylamine hydrochloride (NH₂OH·HCl) in 100 mL of water.

28.8 *Lead Nitrate Stock Solution*—Dissolve 159.8 mg of lead nitrate (Pb(NO₃)₂) in 100 mL of water containing 1 mL of HNO₃ (sp gr 1.42). Dilute with water to 1000.0 mL and mix. This solution should be prepared and stored in glass containers that are free from lead salts.

28.9 *Lead Standard Solution (1 mL = 1 µg Pb)*—Dilute 10 mL of the lead nitrate stock solution, accurately measured, with water to 100.0 mL. Each millilitre of the solution so prepared contains 10 µg of lead.

29. Procedure

29.1 Pipet into a 50-mL Nessler tube 0.15 mL of HCl (1+2),

⁵ A suitable prepared solution of this reagent, known as "Tiferron" or "Tiron," is supplied by the LaMotte Chemical Products Co., Baltimore, MD.

METHOXYL CONTENT

2 mL of the buffer solution, and a volume of the standard lead solution containing the quantity of lead equivalent to the specified heavy metals limit. Add water to make 40 mL and label as Solution A. Make sure that the final pH of these solutions is between 3 and 4. This can be tested by pH indicator paper or pH meter. For visual comparison make sure that the optimum lead content is between 20 and 40 µg.

29.2 To the crucible containing the sulfated ash residue add 4 mL of HCl (1+2). Cover and carefully digest on a steam bath for 10 min. Uncover and slowly evaporate to dryness. Moisten the residue with 0.15 mL of HCl (1+2), 0.5 mL of hydroxylamine hydrochloride solution, and 10 mL of hot water. Carefully digest for 2 min. Add 2 mL of the buffer solution. Filter if necessary. Thoroughly wash the crucible and filter with water into a 50-mL Nessler tube that matches the one used for Solution A. If necessary, adjust the pH to 3 to 4 with NH₄OH (2+3) or HCl (1+2) using pH indicator paper. Dilute to 40 mL and label this Solution B.

29.3 Add 10 mL of H₂S solution to each tube: Solution A and Solution B. Mix and view downward over a white surface. The color of Solution B shall be no darker than that of Solution A. Make the comparison within 10 min.

30. Scope

30.1 This test method covers the determination of the methoxyl content of hydroxypropyl methylcellulose. Total alkoxy is first determined and the methoxyl content found by subtracting the hydroxypropyl content from the total alkoxy figure.

31. Summary of Test Method

31.1 The hydroxypropyl methylcellulose is heated with a strong solution of hydriodic acid to form an alkyl iodide, which by means of a carbon dioxide stream is swept through a condenser and finally absorbed in a mixture of bromine, acetic acid, and sodium acetate. The alkyl iodide absorbed in the aforementioned mixture is oxidized to an alkyl iodate. The excess bromine is reduced with formic acid, and the iodate is determined iodometrically using potassium iodide and standard thiosulfate.

32. Apparatus

32.1 *Distillation Apparatus*, as illustrated in Fig. 1, consisting of a boiling flask with a side arm for admission of carbon

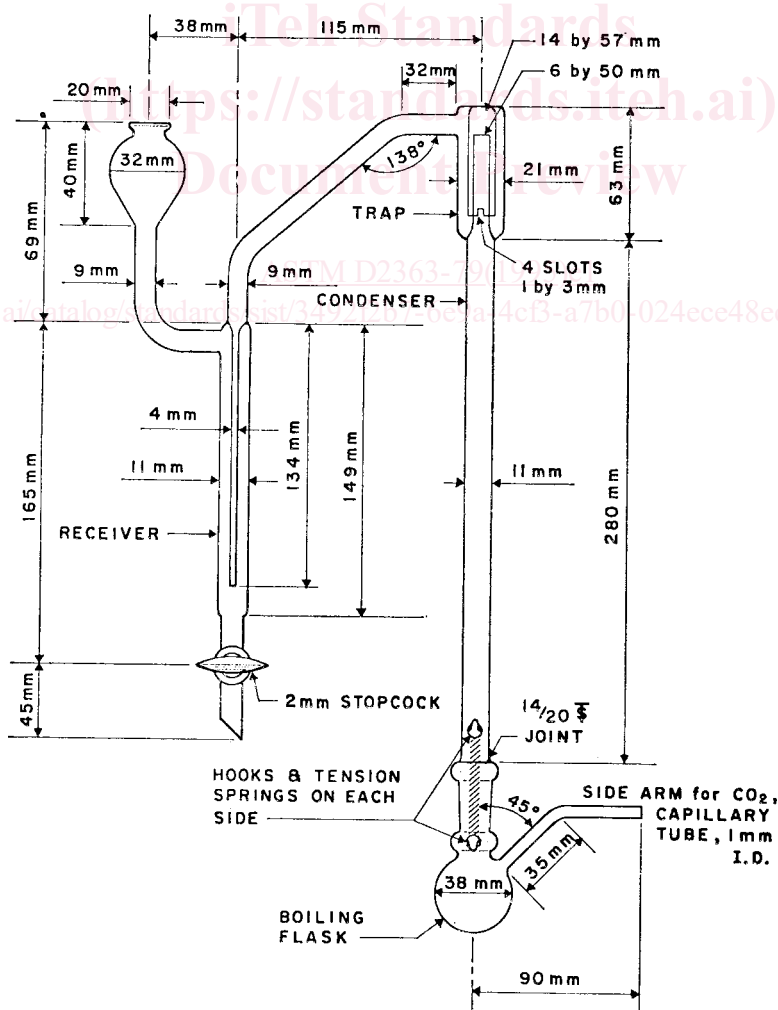


FIG. 1 Distillation Apparatus for Methoxyl Determination

dioxide or nitrogen, an air condenser with a trap, and a receiver.

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

33. Reagents

33.1 *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.

33.2 *Carbon Dioxide*—This may be obtained by the interaction of marble and hydrochloric acid (HCl, 1+1) in a Kipp generator or preferably from a cylinder of the gas equipped with a suitable needle valve. The carbon dioxide (CO₂) shall be passed 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 CO₂.

33.3 *Formic Acid (90 %)*.

33.4 *Gelatin Capsules*—Gelatin capsules of a suitable size to hold 50 to 60 mg of the dried sample.

33.5 *Hydriodic Acid (57 %, sp gr 1.70)*—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 not more than 0.5 mL of 0.1 *N* sodium thiosulfate (Na₂S₂O₃) solution.⁶ 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 CO₂ 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 CO₂ running through the receiver. (**Warning**—See Note 1) Put the purified HI in small, brown, glass-stoppered bottles, previously swept out with CO₂, and seal the stoppers with molten paraffin. Store in a dark place. To minimize decomposition of HI due to contact with air, run CO₂ into the bottle while withdrawing portions of the acid for use.

NOTE 1—**Warning:** Under some conditions the poisonous gas phosphine (PH₃) is formed during distillation, and this may unite with molecular iodine to form phosphorus triiodide (PI₃), which may explode on contact with air. It is, therefore, advisable to keep the current of CO₂ going after the distillation is ended and until the apparatus has cooled.

33.6 *Phosphorus Slurry*—Add about 0.06 g of red phosphorus to 100 mL of water. Shake well before using.

33.7 *Potassium Acetate Solution (100 g/L)*—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.

33.8 *Potassium Iodide (KI)*.

⁶ Hydriodic acid suitable for methoxyl determination may be prepared by the method of Samsel, E. P., and McHard, J. A., "Determination of Alkoxy Groups in Cellulose Ethers," *Industrial and Engineering Chemistry*, Analytical Edition, IECHA, Vol 14, 1942, p. 750. Hydriodic acid available from Merck and Co. has been found satisfactory for this purpose.

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

33.10 *Sodium Thiosulfate Standard Solution (0.1 N)*—Dissolve 25 g of sodium thiosulfate (Na₂S₂O₃·5H₂O) 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 (K₂Cr₂O₇) prepared by dissolving exactly 4.9037 g of K₂Cr₂O₇ (National Institute of Standards and Technology Standard Sample No. 136) in water and diluting to a 1 L in a volumetric flask. By means of a buret, measure accurately 35 to 45 mL of the K₂Cr₂O₇ solution into a 250-mL Erlenmeyer flask. Add 2 g of KI and 50 mL of H₂SO₄ (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 Na₂S₂O₃ 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 Na₂S₂O₃ solution should be checked at least once a week. Calculate the normality of the Na₂S₂O₃ solution, *N*, as follows:

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

where:

A = 0.1000 *N* K₂Cr₂O₇ solution added, mL, and

B = Na₂S₂O₃ solution required for the titration, mL.

As an alternative procedure, the Na₂S₂O₃ solution may be standardized against arsenic trioxide (As₂O₃) (National Institute of Standards and Technology standard sample No. 83) or potassium iodate (KIO₃).

33.11 *Starch Indicator Solution*.

33.12 *Sulfuric Acid (1+9)*—Carefully mix 1 volume of concentrated H₂SO₄ (sp gr 1.84) with 9 volumes of water, adding the H₂SO₄ gradually while mixing.

34. Procedure

34.1 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 of the phosphorus slurry to make the trap about half full (Note 2). Add 19 to 20 mL of the 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. (Do the weighing as rapidly as possible without sacrificing accuracy, since dry hydroxypropyl methylcellulose picks up moisture rapidly.)

NOTE 2—Water may be used in the trap to scrub out entrained vapors of iodine quite successfully. If the test method is to be used as a routine control test, this may be advisable for safety purposes. If so, check the accuracy of the apparatus with the water trap against samples that have been run using the phosphorus slurry trap.

34.2 Add a few small glass beads or chips of clay plate and then 6 mL of the HI. Moisten the ground-glass joint with 2 drops of the HI, then fasten to the distillation assembly. Connect the source of CO₂ to the side arm of the flask. Pass a current of CO₂ 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 40 min.

34.3 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 H₂SO₄ (1 + 9) and titrate immediately with 0.1 N Na₂S₂O₃ solution to a light straw color. Add a little starch indicator solution and continue the titration to the disappearance of the blue color.

34.4 *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 Na₂S₂O₃ solution is required.)

35. Calculation

35.1 Calculate the percent of methoxyl as follows:

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

where:

M = Methoxyl, total (methyl + hydroxypropyl groups calculated as methoxyl),

A = Na₂S₂O₃ solution required for titration of the sample, mL,

B = Na₂S₂O₃ solution required for titration of the blank, mL,

C = normality of the Na₂S₂O₃ solution, and

D = sample used, g.

35.2 In 35.1 the percent OCH₃ represents the total of methyl and hydroxypropyl groups calculated as methoxyl. To obtain the corrected methoxyl content, the total alkoxy must be corrected for the percent OC₃H₆OH obtained in Section 41. The percent OC₃H₆OH shall be first corrected by a factor of 0.93 (an average obtained by running Morgan determinations on a large number of samples) for the propylene produced from the reaction of HI with the hydroxypropyl group as follows:

$$A = B - (C \times 0.93 \times 31/75) \quad (8)$$

where:

A = corrected OCH₃, %,

B = total OCH₃, % , and

C = OC₃H₆OH, % .

HYDROXYPROPOXYL CONTENT⁷

36. Scope

36.1 This test method covers the determination of the hydroxypropoxyl content of hydroxypropyl methylcellulose. The figure obtained from this analysis is used in determining the corrected percent methoxyl content.

37. Summary of Test Method

37.1 The hydroxypropoxyl group of hydroxypropyl meth-

ylcellulose is oxidized by hot chromic acid to acetic acid and this in turn is titrated with 0.02 N sodium hydroxide solution. Procedures are also given for (1) eliminating the error resulting from oxidation of the cellulose backbone, which yields an apparent hydroxypropyl value, and (2) preparing a synthetic hydroxypropyl methylcellulose standard using methylcellulose and propylene glycol.

38. Apparatus

38.1 *Chromic Acid Oxidation Apparatus*, as illustrated in Fig. 2.⁸

38.2 *Oil Bath*, equipped with an electrical heating device so the bath can be maintained at 155°C.

38.3 *pH Meter*, expanded scale, capable of giving reproducible results within ± 0.1 pH units and equipped with glass and calomel electrodes.

39. Reagents

39.1 *Chromium Trioxide Solution (30 %)*—Dissolve 60 g of chromic trioxide (CrO₃) in 140 mL of organic-free water.

39.2 *Nitrogen*.

39.3 *Potassium Iodide (KI)*.

39.4 *Sodium Bicarbonate (NaHCO₃)*.

39.5 *Sodium Hydroxide, Standard Solution (0.02 N), carbon dioxide-free*—Standardize against primary standard potassium hydrogen phthalate (KHC₈H₄O₄) using a pH meter to an end point of pH 7.0 ± 0.1.

39.6 *Sodium Thiosulfate Standard Solution (0.1 N)*—Dissolve 24.8 g of sodium thiosulfate (Na₂S₂O₃) and 0.2 g sodium bicarbonate (NaHCO₃) in freshly boiled water. Dilute to 1 L with water. Standardize against potassium iodate (KIO₃) using starch indicator to determine the end point.

39.7 *Sodium Thiosulfate Standard Solution (0.02 N)*—Dilute 200 ml of 0.1 N sodium thiosulfate standard solution to 1 L with water. Prepare fresh solutions daily as needed.

39.8 *Sulfuric Acid (1 + 165)*—Carefully add, while stirring 10 mL of concentrated H₂SO₄ (sp gr 1.84) to 165 mL of distilled water.

39.9 *Methylcellulose*, free of foreign material such as other substituted celluloses or glycols that will break down to acetic acid.

39.10 *Propylene Glycol*.

39.11 Water, organic-free, obtained by distillation or by ion-exchange treatment and to pass the following test: To 100 mL of water add 10 mL of dilute H₂SO₄ (1+16.5), heat to boiling, and add 0.1 mL of potassium permanganate (KMnO₄) solution (approximately 0.1 N). The water must retain a pink coloration after boiling for 10 min.

40. Procedure

40.1 Weigh to the nearest 0.0001 g about 100 mg of the sample (previously dried at 105°C for ½ h) and transfer to flask, *D*, and add 10 mL of 30 % CrO₃ solution. Fill the steam

⁷ References for the hydroxypropoxyl determination are as follows: Dow Method No. MC-15, "The Determination of the Hydroxypropyl Group in the Presence of an Alkyl Group in Hydroxypropyl Methylcellulose."

Lemieux, R. U., and Purves, C. B., "Quantitative Estimation as Acetic Acid of Acetyl, Ethylidene, Ethoxy, and Hydroxyethyl Groups," *Canadian Journal of Research*, Vol B-25, 1947, p. 485.

Morgan, P. W., "Determination of Ethers and Esters of Ethylene Glycol," *Industrial and Engineering Chemistry, Analytical Edition*, IECHEA, Vol 18, 1946, p. 500.

⁸ The following parts, manufactured by Ace Glass, Inc., Vineland, NJ 08360 (properly modified), have been found satisfactory for this test method: Steam generator, trap vacuum, No. 8753; Reaction flask, 25-mL, No. 9293 P; Vigneaux column, 100-mL, No. 9224; Adapter bleeder tube, No. 9222; Condenser, micro, 100-mL, No. 9195.