



Designation: D 2363 – 79 (Reapproved 2000)^{ε1}

Standard Test Methods for Hydroxypropyl Methylcellulose¹

This standard is issued under the fixed designation D 2363; 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.

ε¹ NOTE—Editorial changes were made throughout in November 2000.

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 SI 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 33.5.1.

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

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 Cellulosics.

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² Annual Book of ASTM Standards, Vol 05.01.

³ Annual Book of ASTM Standards, Vol 15.05.

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.

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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

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 (HNO_3).

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 32 mm (1¼ in.) high, with 6.4-mm (¼-in.) sidewall, and 44-mm (1¾-in.) 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 dessicator 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 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 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, wide-mouth 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 = H₂SO₄ required for titration of the sample, mL,
 B = normality of the H₂SO₄, 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*—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 mL and mix.