



Standard Test Methods for Methylcellulose¹

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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 ₂ CO 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

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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.

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 (H₂SO₄).

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 H₂SO₄ 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:

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 ($\text{Fe}_2\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}$) 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 thiocyanate (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 (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 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.