Designation: D 1347 – 72 (Reapproved 1995)

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:

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

[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.

Current edition approved Feb. 9, 1972. Published March 1972. Originally published as D 1347 – 54 T. Last previous edition D 1347 – 64.

[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.

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 $(Fe_2SO_4)_3 \cdot (NH_4)_2SO_4 \cdot 24H_2O$ 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

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

accordance with 16.1, prepare a calibration curve showing iron content in parts per million and the corresponding photometer readings.

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.

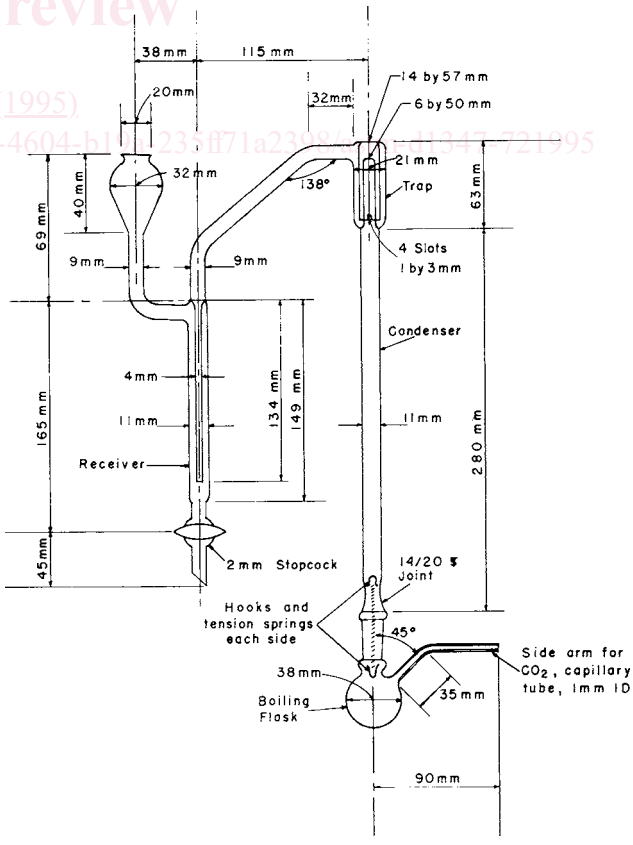

FIG. 1 Distillation Apparatus for Methoxyl Determination