Designation: D914 – 12 (Reapproved 2019)

Standard Test Methods for Ethylcellulose[1]

This standard is issued under the fixed designation D914; 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 ethylcellulose.

1.2 The test procedures appear in the following order:

	Sections
Moisture	4 to 6
Sulfated Ash	7 to 11
Chlorides (as Sodium Chloride)	12 to 16
Ethoxyl Content	20 to 24
Viscosity	25 to 39

1.3 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

1.4 **Warning—**Mercury has been designated by EPA and many state agencies as a hazardous material that can cause central nervous system, kidney and liver damage. Mercury, or its vapor, may be hazardous to health and corrosive to materials. Caution should be taken when handling mercury and mercury-containing products. See the applicable product Material Safety Data Sheet (MSDS) for details and EPA's website (http://www.epa.gov/mercury/faq.htm) for additional information. Users should be aware that selling mercury or mercury-containing products, or both, in your state may be prohibited by state law.

1.5 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D362 Specification for Industrial Grade Toluene (Withdrawn 1989)[3]

D446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers

D841 Specification for Nitration Grade Toluene

D4794 Test Method for Determination of Ethoxyl or Hydroxyethoxyl Substitution in Cellulose Ether Products by Gas Chromatography

E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

3. Purity of Reagents and Materials

3.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, all of the reagents used shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[4] Where such specifications have not been established, reagents of the best grade available shall be used. 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 ethylcellulose.

4.2 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the*

[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 Cellulose and Cellulose Derivatives.

Current edition approved Dec. 1, 2019. Published December 2019. Originally approved in 1947. Last previous edition approved in 2012 as D914 – 12. DOI: 10.1520/D0914-12R19.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

[4] *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.

Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

5. Significance and Use

5.1 The results of this test are used for calculating the total solids in the sample and, by common usage, all materials volatile at this test temperature are designated as moisture.

5.2 Moisture analysis (along with sulfated ash) is used to calculate the amount of active polymer in the material and shall be considered when determining the amount of ethylcellulose in various functions.

6. Apparatus

6.1 *Oven,* gravity convection, capable of maintaining a temperature of 105 ± 3°C.

6.2 *Weighing Bottles.*

6.3 *Analytical Balance.*

7. Procedure

7.1 Weigh accurately 2 to 5 g of the sample to the nearest 0.001 g into a tared dish (fitted with a lid) and dry for 2 h in an oven at 100 to 105°C. Remove the dish from the oven, cover with a lid, cool in a desiccator, and weigh.

8. Calculation

8.1 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.

9. Precision and Bias

9.1 *Precision*—Statistical analysis of intralaboratory (repeatability) test results indicates a precision of ±5 % at the 95 % confidence level.

9.2 *Bias*—No statement of bias can be made as no suitable reference material is available as a standard.

SULFATED ASH

10. Scope

10.1 This test method covers the determination of the residue on ignition of ethylcellulose after a specimen has been treated with sulfuric acid.

10.2 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

11. Significance and Use

11.1 This test method (along with moisture) is used to calculate the active polymer in the material. It shall be used when testing ethylcellulose in United States government regulated applications. Excessive ash may also affect solution clarity and film properties.

12. Apparatus

12.1 *Muffle Furnace.*

12.2 *Crucibles,* either porcelain, 30–mL high, form cracked, platinum.

13. Reagent

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

14. Procedure

14.1 Ignite a crucible for 10 to 15 min at 800 ± 25°C, cool in a desiccator, and weigh to the nearest 0.001 g.

14.2 Weigh about 5 g of sample to the nearest 0.001 g (previously dried for 3 h at 105°C) into the crucible. Burn off the bulk of the carbonaceous material directly over a flame. After cooling, add 1 mL of H_2SO_4 in such a way as to moisten the entire ash; then cautiously heat with the burner to dense white fumes. Ignite in a muffle furnace at 800 ± 25°C until all signs of carbon are gone. Cool in a desiccator and reweigh to the nearest 0.001 g.

15. Calculation

15.1 Calculate the percent ash (as sulfate), *C*, as follows:

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

where:

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

16. Precision and Bias

16.1 *Precision*—Statistical analysis of interlaboratory (reproducibility) test results indicates a precision of ±10 % at the 95 % confidence level.

16.2 *Bias*—No statement of bias can be made as no suitable reference material is available as a standard.

CHLORIDES (as Sodium Chloride)

17. Scope

17.1 This test method covers the determination of the chloride content of ethylcellulose.

17.2 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

18. Significance and Use

18.1 Sodium chloride is a major by-product of the ethylcellulose manufacturing process. This test is a measure of the purity of ethylcellulose. Chlorides may also affect solution properties.

19. Apparatus

19.1 *Titration pH Meter.*

19.2 *Mercury-Mercurous Sulfate Reference Electrode*—The electrode uses a potassium sulfate electrolyte to avoid chloride contamination from a chloride electrolyte.

19.3 *Silver-Silver Chloride Electrode*—The electrode is coated with silver chloride periodically. Prepare the electrode by polishing with fine steel wool, briefly soaking it in 5 % potassium cyanide solution, and rinsing it with water. Coat the electrode with silver chloride by electrodeposition from 0.1 *N* potassium chloride solution using a 3-V dry cell and a platinum wire electrode. Connect the silver electrode to the positive pole of the battery and electrolyze for 20 s; then reverse the connections for 5 s. Repeat these operations twice, and finally, chloridize the silver electrode for 20 s at the positive terminal. Store the silver electrode in 0.1 *N* potassium chloride solution. Rinse the electrode with water and wipe it with a soft tissue before each titration.

19.4 *Salt Bridge for Reference Electrode*—Fig. 1 shows one configuration in use. Exact dimensions are not important. The salt bridge is used to keep the reference electrode from plugging with the ethylcellulose slurry.

19.5 *Air-Driven Stirrer.*

20. Reagents

20.1 *Ethanol* (95 volume %), undenatured or specially denatured conforming to Formula 2B of the U.S. Bureau of Internal Revenue.

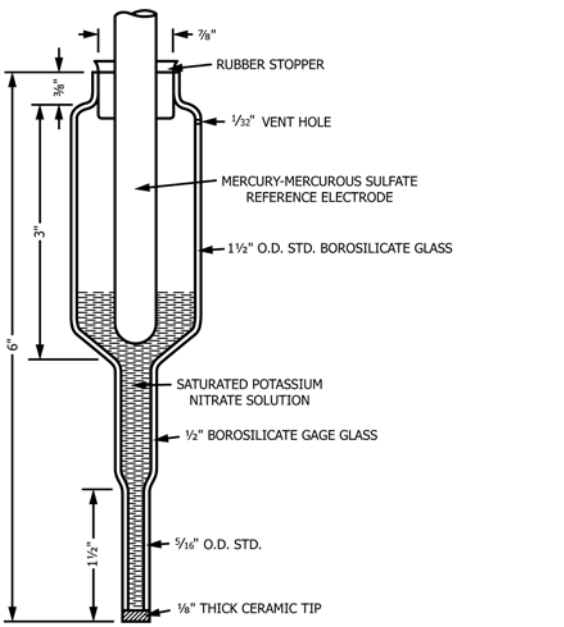

FIG. 1 Salt Bridge and Reference Electrode for Chloride Determination

20.2 *Ethanol-Distilled Water Solvent Mixture (80 + 20)*—Mix 800 g of 2B ethanol with 200 g of water. Add 7.5 g of aerosol OT 100 % surface-active agent per 3000 g of ethanol-water mixture.

20.3 *Potassium Nitrate* (KNO_3) *Solution* (saturated) for salt bridge (Fig. 1).

20.4 *Silver Nitrate, Standard Solution* (0.02 *N*)—Dissolve 3.4 g of silver nitrate ($AgNO_3$) in water, dilute to 1 L with water in a volumetric flask, and mix. Weigh exactly 0.5845 g of dry, primary standard sodium chloride (NaCl), dissolve in 25 mL of water, and dilute to 1 L with water in a volumetric flask. Add 10 mL of H_2SO_4 (1 + 16) to each aliquot before titrating. Titrate aliquots of this solution potentiometrically with the $AgNO_3$ solution. Calculate the normality, *N*, of the $AgNO_3$ solution as follows:

$$N = (A/B) \times 0.01 \quad (3)$$

where:

A = 0.01 *N* NaCl solution added, mL, and
B = $AgNO_3$ solution required for the titration, mL.

20.5 *Sulfuric Acid (1 + 16)*—Add 1 volume of concentrated sulfuric acid (H_2SO_4, sp gr 1.84) slowly with stirring into 16 volumes of water.

20.6 *Toluene,* meeting the requirements of Specification D362.

20.7 *Toluene-Ethanol Solvent Mixture (90 + 10)*—Mix 900 g of toluene with 100 g of ethanol.

21. Procedure

21.1 Weigh accurately 10 g of sample to the nearest 0.001 g (previously dried for 2 h at 100 to 105°C) and transfer to a 600-mL beaker containing 200 mL of the toluene-ethanol solvent mixture. Stir with an air-driven stirrer until solution is complete.

21.2 Add 200 mL of the ethanol-water mixture and agitate for 5 min to form a uniform emulsion. Immerse the electrodes in the emulsion using an air-driven stirrer for mixing. Add 10 mL of H_2SO_4 (1 + 16) and agitate for 3 to 4 min to allow the system to reach equilibrium.

21.3 Titrate slowly with the 0.02 *N* $AgNO_3$ solution. Make intermittent additions of 0.1 mL. It is advisable to allow longer periods of time between additions of titrant as the end point is approached to avoid passing the equivalence point. Run a blank by the same procedure.

22. Calculation

22.1 Calculate parts per million of chlorides as NaCl, *C*, as follows:

$$C = [(VN \times 0.05845)/W] \times 1\ 000\ 000 \quad (4)$$

where:

V = $AgNO_3$ solution, mL,
N = normality of $AgNO_3$ solution,
W = sample used, g, and
0.05845 = milliequivalent mass of NaCl.