Designation: ~~D1439 – 03 (Reapproved 2008)$^{\varepsilon 1}$~~ D1439 – 15

Standard Test Methods for Sodium Carboxymethylcellulose[1]

This standard is issued under the fixed designation D1439; 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.

~~ε^1 NOTE—Editorial changes were made in Sections 11, 12, 15.5, and 31.1 in June 2008.~~

1. ~~Scope~~ Scope*

1.1 These test methods cover the testing of sodium carboxymethylcellulose.

1.2 The test procedures appear in the following order:

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 specific hazard statements, see 15.1 and 20.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1347 Test Methods for Methylcellulose (Withdrawn 2003)[3]

E1 Specification for ASTM Liquid-in-Glass Thermometers

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,[4] ~~,~~ where such specifications are available. 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.

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[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.

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[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] *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.

***A Summary of Changes section appears at the end of this standard**

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 sodium carboxymethylcellulose.

4.2 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. Significance and Use

5.1 Moisture analysis (along with purity) is used to calculate the amount of active polymer in the material and must be considered when determining the amount of sodium carboxymethylcellulose to use in various formulations.

6. Apparatus

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

6.2 *Weighing Bottles,* low-form, 50-mm inside diameter by 30-mm height, or equivalent.

6.3 *Analytical Balance.*

7. Procedure

7.1 Weigh 3 to 5 g of the sample to the nearest 0.001 g in a tared and covered weighing bottle.

7.2 Place the bottle in an oven at 105°C for 2 h with the cover removed. Cool the bottle in a desiccator, replace the cover, and weigh.

7.3 Replace the sample in the oven for 30 min, cool, and reweigh.

7.4 Continue this procedure to a mass loss of not more than 5 mg for 30 min drying time.

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 interlaboratory reproducibility test results on samples containing 2 to 10 % moisture indicates a precision of ±0.2 % absolute at the 95 % confidence level.

9.2 *Bias*—No justifiable statement can be made on the bias of the procedure for measuring moisture because no suitable reference material exists.

DEGREE OF ETHERIFICATION

10. Scope

10.1 These test methods cover the determination of the degree of etherification (D.E.) of sodium carboxymethylcellulose.

10.2 Two test methods are included as follows:

10.2.1 *Test Method A (Acid Wash),* for crude grades of sodium carboxymethylcellulose with degrees of etherification up to 0.85. Above 0.85 degree of etherification, slightly low results may be obtained.

10.2.2 *Test Method B (Nonaqueous Titration),* for purified grades of sodium carboxymethylcellulose of all degrees of etherification. It is not applicable to the crude grades.

11. Significance and Use

11.1 These test methods determine the amount of substituent groups added to the cellulose backbone. The level can greatly affect solution properties, rheology, viscosity, hygroscopicity, salt tolerance, and many other properties of the polymer.

Test Method A—Acid Wash

12. Summary of Test Method

12.1 The water-soluble sodium carboxymethylcellulose is converted to the insoluble acid form, purified by washing, dried, and then a weighed sample is reconverted to the sodium salt with a measured excess of sodium hydroxide.