Designation: D2364 – 15

Standard Test Methods for Hydroxyethylcellulose[1]

This standard is issued under the fixed designation D2364; 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 hydroxyethylcellulose.

1.2 The test procedures appear in the following order:

	Sections
Moisture	4 – 9
Ash	10 – 17
Viscosity	18 – 23
Density	24 – 30

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1193 Specification for Reagent Water

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

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,[3] 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.

3.2 Unless otherwise indicated, reference to water shall be understood to mean reagent water, conforming to Specification D1193.

MOISTURE

4. Scope

4.1 This test method covers the determination of the volatile content of hydroxyethylcellulose.

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 a measure of the amount of active polymer in the material and must be considered when determining the amount of hydroxyethyl cellulose to use in various formulations.

6. Apparatus

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

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

6.3 *Analytical Balance.*

7. Procedure

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

7.2 Place it in an oven at 105°C for 2 h with the cover removed. Replace the cover, cool in a desiccator, and weigh.

[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] *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**

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 Statistical analysis of intralaboratory (repeatability) test results on samples containing from about 3.5 % moisture indicate a precision of ±0.5 % absolute at the 95 % confidence level.

9.2 No statement on bias can be made as no suitable reference material is available as a standard.

ASH—AS SULFATE

10. Scope

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

11. Summary of Test Method

11.1 A specimen is moistened with sulfuric acid, the excess acid evaporated, the carbonaceous matter burned off, and the residue ignited in a muffle furnace, cooled, and weighed.

12. Significance and Use

12.1 Excessive ash can affect solution clarity and film properties. The ash (along with moisture) is a measure of the amount of active polymer in the material and must be considered when determining the amount of hydroxyethyl cellulose to use in various formulations.

13. Apparatus

13.1 *Dishes,* platinum, 50 to 75-mL capacity.

13.2 *Muffle Furnace,* maintained at 825 ± 25°C.

14. Reagents

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

15. Procedure

15.1 Weigh, to the nearest 0.0001 g, about 2 g of the dried sample into a tared platinum dish. Moisten the entire specimen with about 2 mL of H_2SO_4. Then cautiously heat over a small flame until sulfur trioxide (SO_3) fumes cease to be evolved.

15.2 Increase the heat, ignite the specimen, and heat as necessary to burn off the volatile matter. Avoid spattering.

15.3 Place the dish in a 825°C muffle furnace for 1 h, or longer if required, to burn all of the carbon.

15.4 Remove the dish, allow to cool somewhat, place in a desiccator, and cool to room temperature. Weigh the dish and residue to the nearest 0.0001 g.

16. Calculation

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

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

where:

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

17. Precision and Bias

17.1 Statistical analysis of interlaboratory (reproducibility) test results on samples containing 2 to 5 % ash (as sulfate) indicates a precision of ±0.3 % absolute at the 95 % level.

17.2 No statement on bias can be made as no suitable reference material is available as a standard.

VISCOSITY

18. Scope

18.1 This test method is an arbitrary method of determining the viscosity of aqueous solutions of hydroxyethylcellulose in the viscosity range from 10 to 10 000 mPa·s (cP) at 25°C.

18.2 The concentration to be used for the test shall be agreed upon between the purchaser and the seller. It shall be such that the viscosity of the solution will fall within the range of this test.

18.3 The results for the viscosity of hydroxyethylcellulose by this test method will not necessarily agree with results from other types of instruments used for viscosity measurements.

18.4 The determinations are run on a calculated dry basis; that is, the amount of hydroxyethylcellulose required for the desired concentration on a dry basis is calculated from the known moisture content.

19. Significance and Use

19.1 This test method is intended for referee purposes.

19.2 This test method determines the relative ability of the polymer to thicken aqueous solutions and is therefore related to the concentration required in various formulations to achieve the desired finished product viscosity.

20. Apparatus

20.1 *Viscometer, Coaxial Rotational*—The essential instrumentation required providing the minimum rotational viscometer analytical capabilities for this method include:

NOTE 1—Manufacturers of cellulose derivatives usually specify the viscometer make, model, spindle, and speed to be used with their products. It is highly recommended that these specifications be followed. Use of a viscometer made by another company or even a different model by the same company may result in slightly different results.

20.1.1 A *drive motor* to apply a unidirectional rotational displacement to the specimen at a rate from 0.5 to 60 r/min constant to within ±1 %.

20.1.2 A *force sensor* to measure the torque developed by the specimen to the rotational displacement of the rotational element.

20.1.3 A *coupling shaft* or other means to transmit the rotational displacement from the motor to the specimen.

20.1.4 A *rotational element, spindle, or tool* to fix the specimen between the drive shaft and a stationary position.

NOTE 2—Each rotational element typically covers a range of about 2 decades of viscosity. The rotational element is selected so that the measured viscosity is between 10 and 90 % of the range of that rotational element.

NOTE 3—Coaxial cylinder rotational elements of the form shown in Fig. 1 are suitable for this standard.

20.1.5 A *specimen container*, approximately 64 mm in outside diameter, 152 mm in height, and 350 mL in volume, to contain the test specimen during testing.

20.1.6 A *data collection device*, to provide a means of acquiring, storing, and displaying measured or calculated signals, or both. The minimum output signals required for rotational viscometry are torque, rotational speed, temperature, and time.

20.1.7 A *stand*, to support, level, and adjust the height of the drive motor, shaft, and rotational element.

20.1.8 Auxiliary instrumentation considered necessary or useful in conducting this method includes:

20.1.8.1 *Data analysis* capability to provide viscosity, stress or other useful parameters derived from the measured signals.

20.1.8.2 A *level* to indicate the vertical plumb of the drive motor, shaft and rotational element.

20.2 *Mechanical Stirrer*—Agitator as shown in Fig. 2 or Fig. 3, attached to a variable-speed motor capable of 1500 r/min rotational speed.

NOTE 4—An agitator made with 38 mm (1.5 in) three-bladed propellers is satisfactory for this purpose.

20.3 *Water Bath,* constant-temperature, set at 25°C and capable of maintaining that temperature to within ±0.2°C.

20.4 A *temperature sensor* to provide an indication of the specimen temperature over the range of 19 to 27°C to within ±0.1°C.

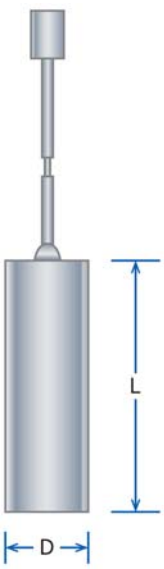


FIG. 1 Coaxial Cylinder Rotational Element

21. Procedure

21.1 Determine the moisture in accordance with Sections 4 – 9.

21.2 Calculate the dry-basis specimen mass, M, in grams necessary to make 250 g of test solution as follows:

$$M_s = 100\,A/(100 - B) \quad (3)$$

where:

A = desired dry mass of specimen, g, and
B = percent moisture in the weighed specimen.

21.3 Add the specimen to the jar. Then add sufficient distilled water to make a total of 250 g of solution. Calculate the mass of water, M_W, in grams as follows:

$$M_w = 250 - S \quad (4)$$

where:

S = sample mass, g.

21.4 Place the agitator in the solution allowing a minimum clearance between the agitator and the bottom of the container. Stir at approximately 1500 r/min until the specimen is completely dissolved. This may require several hours.

21.5 Remove the agitator from the motor and transfer the specimen container, with the agitator in it, to the constant temperature bath. Allow it to stand for 1 h.

21.6 Remove the specimen container from the bath and shake or stir vigorously for 10 s.

NOTE 5—If the room temperature is considerably greater or less than 25°C, the entire operation of stirring, standing, and measurement should be conducted with the specimen suspended in the water bath.

21.7 Measure the viscosity with the rotational viscometer at a rotational speed of 30 or 60 r/min using a coaxial spindle (see Fig. 1) suitable for the test specimen viscosity. Allow the spindle to rotate for 3 min before taking a reading.

21.7.1 **Warning**—Shaking or stirring the sample may cause entrainment of air bubbles. One must exercise care to avoid having a large air bubble under the viscometer spindle when taking the measurement.

22. Report

22.1 Report the following information:

22.1.1 Viscosity at 25°C,

22.1.2 Solution concentration,

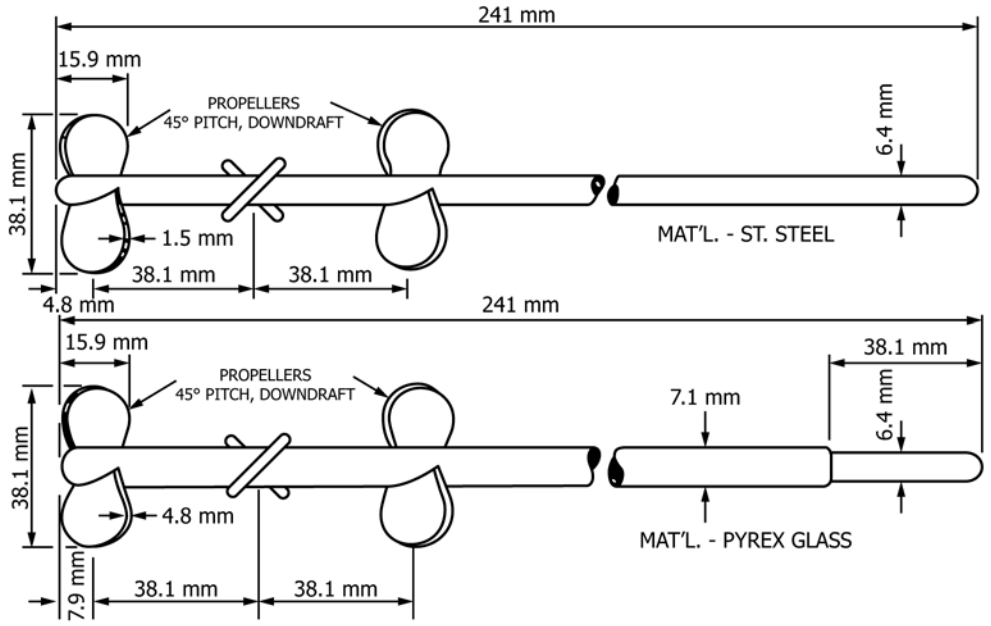
22.1.3 Complete description of the apparatus used including manufacturer, model number and spindle, and

22.1.4 Description of experimental conditions including rotational speed used.

23. Precision and Bias

23.1 *Precision*—Statistical analysis of interlaboratory (reproducibility) test results indicates a precision of ±10 % at the 95 % confidence level when using the same viscometer make and model.

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



in.	mm	in.	mm
1/16	1.5	5/16	7.9
3/16	4.8	5/8	15.8
1/4	6.4	1 1/2	38
9/32	7	9 1/2	241

FIG. 2 Agitator

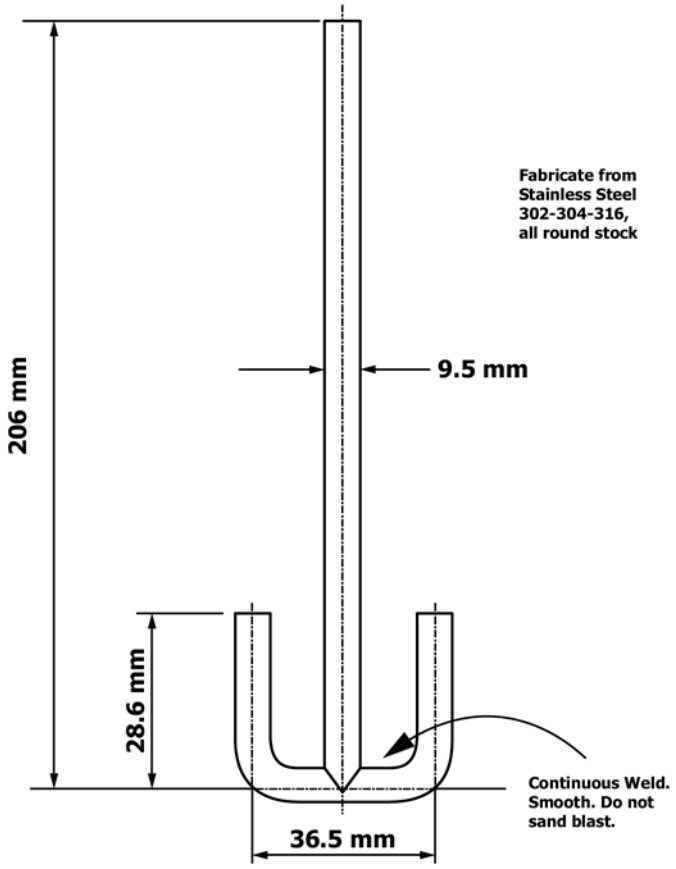


FIG. 3 Agitator

DENSITY

24. Scope

24.1 This test method covers the determination of the bulk density of hydroxyethylcellulose.

25. Summary of Test Method

25.1 A weighed amount of hydroxyethylcellulose is transferred to a 100-mL graduated cylinder and the graduate vibrated to settle the powder.

26. Significance and Use

26.1 Density can relate to dry flow properties, rate of dissolution, lumping, packaging, and storage space requirements.

27. Apparatus

27.1 *Vibrator*—A magnetic-type electric vibrator attached to the vertical support rod of a ring stand approximately 0.3 m above the base. A condenser clamp of sufficient size to hold a 100-mL graduated cylinder also shall be attached to the above rod. The base of the stand should be weighted.

28. Procedure

28.1 Place 50.0 g of hydroxyethylcellulose in a 100-mL graduated cylinder and place in the condenser clamp. Turn on the vibrator and allow the cylinder to vibrate for 3 min. Record the level (in millilitres) to which the specimen has compacted.

28.2 Alternatively, the specimen may be compacted manually. Tap it on a hard surface by dropping the cylinder repeatedly from a height of about 25 mm until the volume of the sample remains constant. In order to prevent cylinder breakage, cover the tapping surface with a 3 to 6-mm thick rubber sheet, or use a plastic graduated cylinder.

29. Calculation

29.1 Calculate the density, D, in grams per millilitre as follows:

$$D = 50/r_o \quad (5)$$

where R_o = observed reading, mL.

30. Precision and Bias

30.1 *Precision*—Statistical analysis of intralaboratory (repeatability) test results indicates a precision of ±0.04 g/mL at the 95 % confidence level.

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

MOLAR SUBSTITUTION: see Test Method D4794

31. Keywords

31.1 ash; density; hydroxyethylcellulose; moisture; molar substitution; rotational viscometer; viscosity

SUMMARY OF CHANGES

Committee D01 has identified the location of selected changes to this standard since the last issue (D2364-01(2007)$^{\varepsilon 1}$) that may impact the use of this standard. (Approved December 1, 2015.)

(1) Section 20 is revised to include a generic description of rotational viscometer apparatus in accordance with Practice E1953.

(2) Figure 1 is added. Figures 2 and 3 are revised to SI metric equivalence.