



Standard Test Methods for Hydroxyethylcellulose¹

This standard is issued under the fixed designation D 2364; 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	18-24
Density	25-31
Molar Substitution	32-42

1.3 *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:

- D 1193 Specification for Reagent Water²
- D 1347 Test Methods for Methylcellulose³
- D 1695 Terminology of Cellulose and Cellulose Derivatives³
- D 2363 Test Methods for Hydroxypropyl Methylcellulose³
- D 4794 Test Method for Determination of Ethoxyl or Hydroxyethoxyl Substitution in Cellulose Ether Products by Gas Chromatography³
- E 1 Specification for ASTM Thermometers⁴

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.⁵ Other grades may be used, pro-

vided 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 D 1193.

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 $105 \pm 3^\circ\text{C}$.
- 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.

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

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² *Annual Book of ASTM Standards*, Vol 11.01.

³ *Annual Book of ASTM Standards*, Vol 06.03.

⁴ *Annual Book of ASTM Standards*, Vol 14.03.

⁵ *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.

indicate a precision of $\pm 0.5\%$ 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 $825 \pm 25^\circ\text{C}$.

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 $\pm 0.3\%$ 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 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 check 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. The Brookfield spindles and speeds given in Table 1 are recommended for this purpose, but slight deviations from Table 1 may occasionally be found convenient for individual application.

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*, Rotational type.

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 will result in slightly different results.

20.2 *Container*, glass jar, 350-cm³ approximately 64 mm in outside diameter and 152 mm high.

20.3 *Mechanical Stirrer*—Agitator as shown in Fig. 1 or Fig. 2,⁶ attached to a variable-speed motor capable of 1500 r/min.

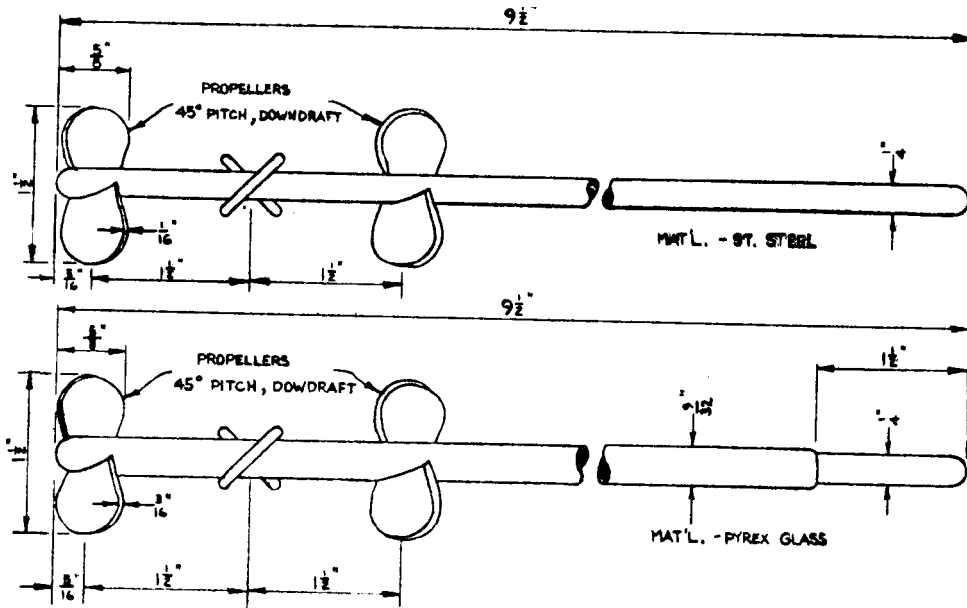
20.4 *Water Bath*, constant-temperature, set at 25°C and capable of maintaining that temperature to within $\pm 0.2^\circ\text{C}$.

20.5 *Thermometer*—An ASTM Saybolt Viscosity Thermometer having a range from 19 to 27°C and conforming to the requirements for Thermometer 17C, as prescribed in Specification E 1.

⁶ An agitator made with 1 1/2 in. (38 mm), three-bladed propellers, No. 9240K, available from A. H. Thomas Co., P.O. Box 779, Philadelphia, PA 19105 has been found satisfactory for this purpose.

TABLE 1 Viscometer Spindles Required for Given Speeds

Viscosity Range, cP	Spindle No.	Speed, rpm	Scale	Factor
10 to 100	1	60	100	1
100 to 200	1	30	100	2
200 to 1000	2	30	100	10
1000 to 4000	3	30	100	40
4000 to 10 000	4	30	100	200



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

FIG. 1 Agitator

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:

$$M_s = 100 A / (100 - B) \tag{3}$$

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:

$$M_w = 250 - S \tag{4}$$

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. Measure the viscosity with the rotational viscometer, following the manufacturer's instructions, selecting the proper spindle and speed from Table 1. Allow the spindle to rotate for 3 min before taking a reading.

NOTE 2—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.

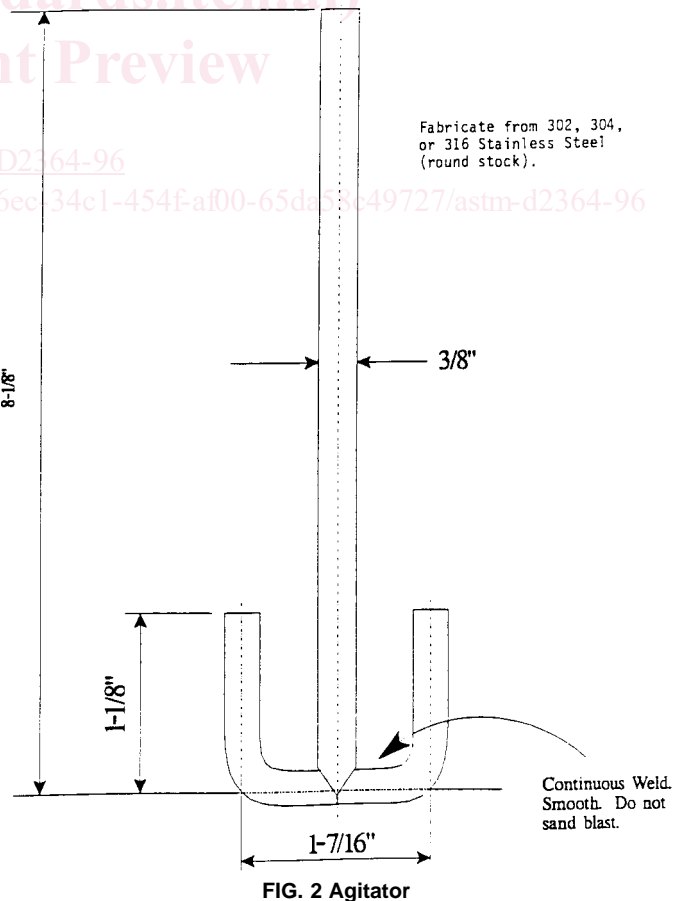


FIG. 2 Agitator