Designation: D 5400 – 93 (Reapproved 1997)

Standard Test Methods for Hydroxypropylcellulose[1]

This standard is issued under the fixed designation D 5400; 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 hydroxypropylcellulose.

1.2 The test procedures appear in the following order:

	Sections
Moisture	4 to 9
Viscosity	10 to 16
pH	17 to 21
Residue on Ignition	22 to 28
Hydroxypropoxy Content	29 to 36

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.* For specific hazard statements, see Note 2.

2. Referenced Documents

2.1 *ASTM Standards:*

D 3876 Test Method for Methoxyl and Hydroxypropyl Substitution in Cellulose Ether Products by Gas Chromatography[2]

E 70 Test Method for pH of Aqueous Solutions With the Glass Electrode[3]

3. Reagents

3.1 *Purity of Reagents*—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.[4] 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 *Purity of Water*—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 hydroxypropylcellulose.

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 residue on ignition) is a measure of the amount of active polymer in the material and must be considered when determining the amount of hydroxypropylcellulose to use in various formulations.

6. Apparatus

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

6.2 *Weighing Dish*, glass or aluminum, with cover, 50 mm in diameter, 25 mm in height, or equivalent.

6.3 *Analytical Balance*.

7. Procedure

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

7.2 Place it in an oven at 105 ± 1°C for 3 h with the cover removed.

7.3 Remove the dish from the oven, immediately 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

[1] These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.36 on Cellulose and Cellulose Derivatives.

Current edition approved May 15, 1993. Published July 1993.

[2] *Annual Book of ASTM Standards*, Vol 06.03.

[3] *Annual Book of ASTM Standards*, Vol 15.05.

[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.

B = sample used, g.

8.2 Report the moisture content to the nearest 0.1 %.

9. Precision and Bias

9.1 *Precision*—Statistical analysis of intralaboratory test results on samples containing from 1 to 6 % moisture indicates a precision of ±0.2 % absolute at the 95 % confidence level.

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

VISCOSITY

10. Scope

10.1 This test method is an arbitrary method of determining the viscosity of aqueous solutions of hydroxypropylcellulose in the viscosity range of 10 to 16 000 cP/s at 25°C.

10.2 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.

10.3 The results for the viscosity of hydroxypropylcellulose by this test method will not necessarily check with results from other types of instruments used for viscosity measurement.

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

11. Significance and Use

11.1 This test method is intended for referee purposes. The Brookfield[5] 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.

TABLE 1 Brookfield[A] Viscometer Spindle and Speed Combinations

Viscosity Range, cP	Spindle Number	Speed, r/min	Scale	Factor
10–80	1	60	100	1
20–160	1	30	100	2
50–400	2	60	100	5
100–800	2	30	100	10
400–1600	3	60	100	20
800–3200	3	30	100	40
2000–8000	4	60	100	100
4000–16 000	4	30	100	200

[A]Brookfield Viscometer, Model LVF[5].

11.2 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.

12. Apparatus

12.1 *Viscometer*, Brookfield Type.[5]

12.2 *Container*, glass bottle, 12-oz (350-cm^3) approximately 2½ in. (64 mm) in outside diameter and 6 in. (152 mm) high.

12.3 *Mechanical Stirrer*, agitator as shown in Fig. 1, attached to a variable-speed motor capable of 1500 r/min.

12.4 *Water Bath*, constant-temperature, set at 25°C and capable of maintaining that temperature to within ± 0.5°C.

13. Procedure

13.1 Determine the moisture in accordance with Sections 4-9.

13.2 Calculate the sample mass, *S*, in grams necessary to make 250 g of test solution as follows:

$$S = \frac{A \times 100}{100 - B} \quad (2)$$

where:

A = desired dry mass of sample, g, and

B = percent moisture in the weighed sample.

13.3 Calculate the mass of water required, *W*, in grams as follows:

$$W = 250 - S \quad (3)$$

where:

S = sample mass, g.

13.4 Weigh the water into the glass bottle and place it on the stirrer, mixing at a speed sufficient to cause a vortex but not fast enough to splash.

13.5 Sprinkle the hydroxypropylcellulose sample into the water slowly, to prevent lumping. Increase stirring speed to

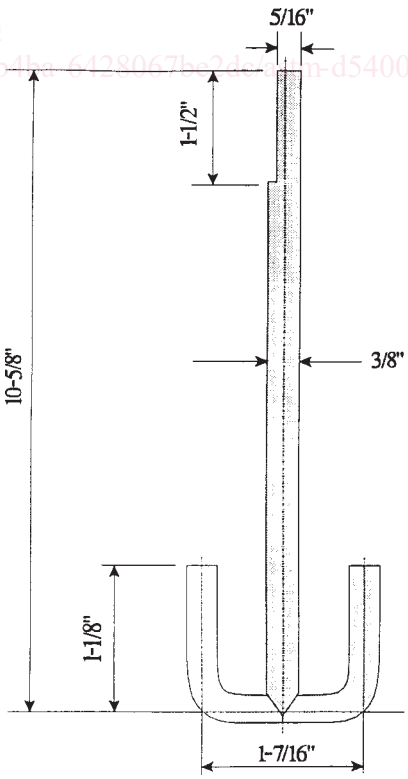

FIG. 1 Stainless Steel Agitator

[5] Model LVF, available from Brookfield Engineering Laboratories, Inc., Stoughton, MA has been found satisfactory for this purpose.