



Designation: D5400 – 03(Reapproved 2013)

## Standard Test Methods for Hydroxypropylcellulose<sup>1</sup>

This standard is issued under the fixed designation D5400; 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 ( $\epsilon$ ) 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 – 9
Viscosity	10 – 16
pH	17 – 21
Residue on Ignition	22 – 28
Hydroxypropoxy Content	29 – 36

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 26.6.

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D3876 Test Method for Methoxyl and Hydroxypropyl Substitution in Cellulose Ether Products by Gas Chromatography

E70 Test Method for pH of Aqueous Solutions With the Glass Electrode

### 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,

<sup>1</sup> 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 June 1, 2013. Published June 2013. Originally approved in 1993. Last previous edition approved in 2008 as D5400 – 03 (2008). DOI: 10.1520/D5400-03R13.

<sup>2</sup> 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.

where such specifications are available.<sup>3</sup> 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  $105 \pm 3^\circ\text{C}$ .

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  $105 \pm 1^\circ\text{C}$  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.

<sup>3</sup> *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.

**TABLE 1 Brookfield<sup>4</sup> 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

<sup>4</sup> Brookfield Viscometer, Model LVF<sup>4</sup>.

## 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.

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<sup>4</sup> 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.

<sup>4</sup> Model LVF, available from Brookfield Engineering Laboratories, Inc., Stoughton, MA has been found satisfactory for this purpose.

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.<sup>4</sup>

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

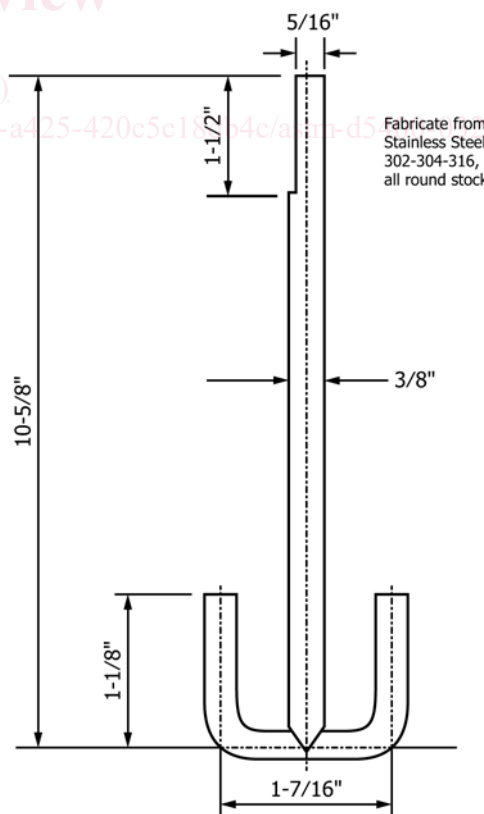
13.2 Calculate the sample mass, *S*, in grams necessary to make 250 g of test solution as follows:

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

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:



**FIG. 1 Stainless Steel Agitator**

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

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 approximately  $900 \pm 100$  r/min and stir until the sample is completely dissolved (1 to 2 h).

13.6 Remove the glass bottle from the stirrer, cap the bottle, and place it in the constant temperature bath for 30 min or as long as necessary to bring the solution to  $25 \pm 0.5^\circ\text{C}$ .

13.7 Remove the glass bottle from the water bath and invert it slowly 4 to 5 times, taking care to minimize the introduction of air bubbles.

13.8 Measure the viscosity with the Brookfield viscometer, following the manufacturer's operating instructions. Select the proper spindle and speed from **Table 1**. Allow the spindle to rotate for 3 min before taking the reading. Use the spindle guard.

**NOTE 1**—If the room temperature is considerably greater or less than  $25^\circ\text{C}$ ; the entire operation of stirring, standing, and measurement should be conducted with the glass bottle suspended in the constant temperature bath.

#### 14. Calculation

14.1 Calculate the viscosity,  $V$ , in centipoises as follows:

$$V = r \times f \quad (4)$$

where:

$r$  = scale reading and

$f$  = factor from **Table 1**.

#### 15. Report

15.1 Report the following information:

15.1.1 Results, as Brookfield viscosity at  $25^\circ\text{C}$ ,

15.1.2 Solution concentration,

15.1.3 Spindle used, and

15.1.4 Speed used.

#### 16. Precision and Bias

16.1 *Precision*—Statistical analysis of interlaboratory test results indicates a precision of  $\pm 10\%$  at the 95 % confidence level.

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

### pH

#### 17. Scope

17.1 This test method covers the determination of the pH of aqueous solutions of hydroxypropylcellulose.

#### 18. Significance and Use

18.1 This test method determines the relative acidity or alkalinity of aqueous solutions of hydroxypropylcellulose. This

must be taken into consideration when determining materials of construction for equipment and packaging for handling and storing these solutions. Also, pH may affect suitability for certain end-use applications.

#### 19. Apparatus

19.1 *pH Meter*, with glass electrodes.

#### 20. Procedure

20.1 Determine the pH of the viscosity solution from **13.5**, using a suitable pH meter that has been standardized according to Test Method **E70**.

#### 21. Precision and Bias

21.1 *Precision*—Statistical analysis of intralaboratory test results on samples with pH values of 4.5 to 8.5 indicates a precision of  $\pm 5\%$  at the 95 % confidence level.

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

### RESIDUE ON IGNITION

#### 22. Scope

22.1 This test method covers the determination of the amount of residue left from igniting a sample of hydroxypropylcellulose after being moistened first with sulfuric acid and then with hydrofluoric acid.

#### 23. Significance and Use

23.1 Excessive residue on ignition can affect solution clarity and film properties. The residue on ignition (along with moisture) 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.

23.2 Commercially available hydroxypropylcellulose most often has up to 0.60 % silica added as an anti-caking agent. Therefore, a hydrofluoric acid ashing step is required to remove the silica.

#### 24. Apparatus

24.1 *Dishes*, platinum, 75-mL capacity.

24.2 *Hot Plate*.

24.3 *Muffle Furnace*, capable of reaching  $1000 \pm 25^\circ\text{C}$ .

#### 25. Reagents

25.1 *Sulfuric Acid (sp gr 1.84)*—concentrated sulfuric acid ( $\text{H}_2\text{SO}_4$ ).

25.2 *Hydrofluoric Acid* (48 %).

#### 26. Procedure

26.1 Weigh, to the nearest 0.0001 g, about 1 g of hydroxypropylcellulose into a tared platinum dish. Using a hot plate, heat the dish containing the sample (gently at first) until the sample is thoroughly charred. Cool the dish for several minutes.