



Designation: D5400 – 15 (Reapproved 2021)

Standard Test Methods for Hydroxypropylcellulose¹

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 (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 – 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see 26.6.

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*²

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

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. 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.³ 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.

³ *ACS Reagent Chemicals, Specifications and Procedures for Reagents and Standard-Grade Reference Materials*, 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.

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.

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 mPa · s (cP) 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 agree 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 The rotational elements and speeds given in Table 1 are recommended for this purpose. Slight deviations from Table 1 may be found convenient for an individual application.

TABLE 1 Cylindrical Rotational Elements and Speeds
(see Fig. 1)

Viscosity Range, (mPa·s)	Diameter (D) (mm)	Length (L) (mm)	Speed (r/min)	Torque Scale Multiplier
10 to 80	19	65	60	1
20 to 160	19	65	30	2
50 to 400	10	54	60	5
100 to 800	10	54	30	10
400 to 1600	5.9	43	60	20
800 to 3200	5.9	43	30	40
2000 to 8000	3.2	31	60	100
4000 to 16 000	3.2	31	30	200

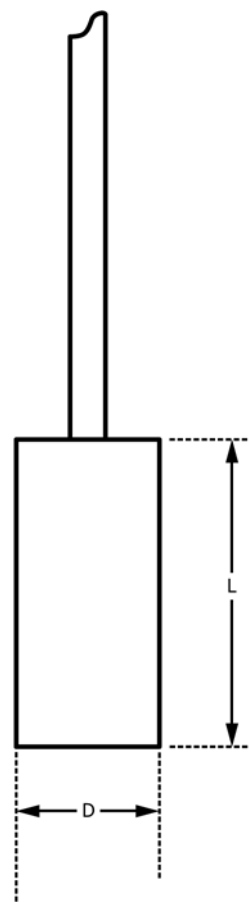


FIG. 1 Cylindrical Rotational Elements and Speeds

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

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

12.1.2 A *force sensor* to measure the torque developed by the specimen.

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

NOTE 1—It is helpful to have a mark on the shaft to indicate the appropriate level of the specimen.

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

NOTE 2—The rotational element shall be of the immersion type. This standard is not intended for cone-and-plate or parallel plate type viscometers.

NOTE 3—Each rotational element typically covers a range of about two decades of viscosity. The rotational element is selected so that the measured viscosity is between 15 and 95 % of the range of the rotational element.