



## Standard Test Method for Methoxyl and Hydroxypropyl Substitution in Cellulose Ether Products by Gas Chromatography<sup>1</sup>

This standard is issued under the fixed designation D 3876; 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 This test method is applicable to the determination of methoxyl and hydroxypropyl substitution content in cellulose ether products by a Zeisel-gas chromatographic technique.

1.2 This test method is not suitable for use for the analysis of hydroxypropyl-cellulose due to its very high substitution level.

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 Section 2, 11.1.4, and 11.1.7.

### 2. Summary of Test Method

2.1 When methyl cellulose or hydroxypropyl methyl cellulose is reacted with hydriodic acid, 1 mol of methyl iodide and 1 mol of isopropyl iodide are liberated for each mole of methoxyl and hydroxypropoxyl that is substituted on the cellulose chain. The methyl iodide and isopropyl iodide are extracted in situ with *o*-xylene and quantitated by gas chromatography using an internal standard technique.

### 3. Significance and Use

3.1 This test method determines the methoxyl and hydroxypropoxyl content of cellulose ethers by a Zeisel-gas chromatographic technique.

3.2 Substitution levels affect solution properties, rheology, viscosity, and many other properties of the polymer.

### 4. Apparatus

4.1 *Gas Chromatograph*,<sup>2</sup> with thermal conductivity detector and heated injection port.

4.2 *Electronic Integrator*<sup>3</sup>

4.3 *Stainless Steel Tubing*,<sup>4</sup> 9.5 mm in outside diameter and

1981 mm in length, packed with reagent in 5.8.

4.4 *Syringes*, 10 and 100 µL.

4.5 *Reaction Vials, Caps, and Heating Block*.<sup>5</sup>

### 5. Reagents

5.1 *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.<sup>6</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.

5.2 *o*-Xylene, ACS.

5.3 Toluene, ACS.

5.4 Iodomethane, 99% min.

5.5 2-Iodopropane, 97% min.

5.6 Hydriodic Acid (sp. gr. 1.69 to 1.70) 57%.

5.7 Acetone.

5.8 *Packing Material*.<sup>7</sup>

### 6. Hazards

6.1 Safety precautions must be taken for handling of hydriodic acid.

6.2 During the reaction, the glass vials are under pressure. Exercise caution in handling the hot vials.

### 7. Sampling

7.1 A specific sampling method is currently under study by the subcommittee.

### 8. Apparatus Preparation and Conditioning

8.1 Install prepackaged columns in the chromatograph and condition them by heating to 150°C over 1 h and then holding

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D01 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 Jan. 10, 1996. Published March 1996. Originally published as D 3876 – 79. Last previous edition D 3876 – 79 (1989)<sup>ε1</sup>.

<sup>2</sup> Hewlett-Packard Model 5700, available from Hewlett-Packard, Route 41, Starr Rd, P.O. Box 900, Avondale, PA 19311, has been found satisfactory for this purpose.

<sup>3</sup> Hewlett-Packard Model 3380 has been found satisfactory for this purpose.

<sup>4</sup> Tubing from Supelco, Inc., Supelco Park, Bellefonte, PA 16823 has been found satisfactory for this purpose.

<sup>5</sup> Reacti-therm Heating module, Reacti-Block Reacti-vials and Mininert valve tops from Pierce Chemical Co., Box 117, Rockford, IL 61105 have been found satisfactory for this purpose.

<sup>6</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.

<sup>7</sup> Columns packed with 10 % SP2100 on 100/120 Supelcoport® have been found satisfactory for this purpose.