Designation: D4794 – 94 (Reapproved 2022)ε1

Standard Test Method for Determination of Ethoxyl or Hydroxyethoxyl Substitution in Cellulose Ether Products by Gas Chromatography[1]

This standard is issued under the fixed designation D4794; 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.

ε[1] NOTE—Editorial update made to 1.3 and 9.1.8 in June 2022.

1. Scope

1.1 This test method is applicable to the determination of ethoxyl or hydroxyethoxyl substitution in cellulose ether products by a Zeisel gas chromatographic technique.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statements, see Section 5 and 9.1.8.1.

1.4 *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. Summary of Test Method

2.1 When a cellulose ether containing ethyl or hydroxyethyl substitutes is allowed to react with hydriodic acid, one mole of iodoethane is liberated for each mole of ethoxyl or hydroxyethoxyl ether substituted on the cellulose chain. The iodoethane is extracted in situ with *o*-xylene and quantitated by gas chromatography using an internal standard technique. It is recommended to run duplicate samples.

3. Apparatus

3.1 *Gas Chromatograph* with thermal conductivity detector and heated injection port.

3.2 *Electronic Integrator.*

3.3 *Column*—stainless steel, 1829 mm in length, 3.2 mm in outside diameter, packed with the reagent in 4.6, coiled to fit the chromatograph used; or equivalent column and packing as appropriate.

3.4 *Syringe,* 10 µL.

3.5 *Reaction Vials, Caps, and Heating Block.* [2]

3.6 *Syringe,* 100 µ L.

3.7 *Cover,* stainless steel, fabricated to cover the heating block.

4. Reagents and Materials

4.1 *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.

4.2 *Iodoethane,* 99 % minimum.

4.3 *O-xylene.*

4.4 *Toluene.*

4.5 *Hydriodic Acid,* 57 % (sp gr 1.69 to 1.70).

4.6 *Column Packing,* 10 % methyl silicone stationary phase USP code [G1] coated on inert diatomite solid support USP code [S1A], 100/120 mesh.

[1] 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.

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[2] The sole source of supply of a heating block, Silli-Therm Heating Module, Reacti-Block 21, Reacti-Vials, and Minnert valve tops, known to the committee at this time is the Pierce Chemical Company P.O. Box 117, Rockford, IL 61105–9976. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[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.