



Designation: D4273 – 18

Standard Test Method for Polyurethane Raw Materials: Determination of Primary Hydroxyl Content of Polyether Polyols¹

This standard is issued under the fixed designation D4273; 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 *Carbon-13 Nuclear Magnetic Resonance Spectroscopy* (¹³C NMR) measures the primary hydroxyl content of ethylene oxide (EO)-propylene oxide (PO) polyether polyols used in preparing flexible polyurethane foams. This method is best suited for polyether polyols with primary hydroxyl contents of 10 to 90 %.

1.2 The values stated in SI units are to be regarded as 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.*

NOTE 1—There is no known ISO equivalent to this standard.

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. Referenced Documents

2.1 *ASTM Standards:*²

D883 Terminology Relating to Plastics

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

E2977 Practice for Measuring and Reporting Performance of Fourier-Transform Nuclear Magnetic Resonance (FT-NMR) Spectrometers for Liquid Samples

¹ This test method is under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.22 on Cellular Materials - Plastics and Elastomers.

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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. Terminology

3.1 *Definitions*—For definitions of terms that appear in this method, refer to Terminology D883 and Practice E2977.

4. Summary of Test Method

4.1 Peaks of the primary and secondary hydroxyl carbons of polyether polyols used in flexible polyurethane foams are well-resolved in high-resolution ¹³C NMR spectra. The primary hydroxyl content is determined from the ratio of the primary hydroxyl area to the total hydroxyl (primary and secondary) area.

5. Significance and Use

5.1 The primary hydroxyl content provides information about the relative reactivities of polyols.

6. Interferences

6.1 Primary hydroxyl PO methylene carbons (where the methylene carbon is next to the hydroxyl group and the methine carbon is next to the ether oxygen) are integrated with the secondary hydroxyl carbons and are therefore not included in the primary hydroxyl content as measured by this method.

7. Apparatus

7.1 *Fourier-Transform NMR (FT-NMR) Spectrometer*, with carbon-13 capability and a carbon-13 resonance frequency of 50 MHz (proton resonance frequency of 200 MHz) or higher. The spectrometer is to have a minimum carbon-13 signal-to-noise ratio of 70:1 based on the benzene carbon signal in a 60 % benzene-d₆, 40 % p-dioxane (v/v) sample (ASTM NMR standard) that has been pulsed once using a 90° pulse angle under the conditions specified in Practice E2977.

7.2 *NMR sample tubes* having outside diameters of 5 mm or more.

7.3 *NMR spinners*.

8. Reagents and Materials

8.1 All reagents are to be spectroscopic-grade and free of magnetic materials.

8.1.1 *Deuterated chloroform or deuterated acetone*, containing tetramethylsilane (TMS) as an internal standard.

*A Summary of Changes section appears at the end of this standard