



Standard Test Method for Polyurethane Raw Materials: Determination of Monomer and Isomers in Isocyanates¹

This standard is issued under the fixed designation D7252; 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.

^{ε1} NOTE—Reapproved with editorial changes in September 2011.

1. Scope

1.1 This test method determines the percent by weight of monomeric isomers and total monomer in crude or modified isocyanates. The test method is applicable to methylene di(phenylisocyanate) (MDI) and polymeric (meththylene phenylisocyanate) (PMDI). (See [Note 1.](#))

1.2 *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.*

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

2. Referenced Documents

2.1 *ASTM Standards:*²

[D883 Terminology Relating to Plastics](#)

[E682 Practice for Liquid Chromatography Terms and Relationships](#)

[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

3. Terminology

3.1 For definitions of terms used in these test methods see Terminology [D883](#).

4. Summary of Test Method

4.1 The sample is reacted (derivatized) with methanol to form a mixture of methyl urethanes. The urethanes mixture is then separated by normal phase high performance liquid

chromatography (HPLC). The separated, derivatized isomers are quantified through the use of an internal standard.

5. Significance and Use

5.1 This test method can be used for research or for quality control to characterize isocyanates used in polyurethane products.

6. Apparatus

6.1 *High Performance Liquid Chromatograph*, consisting of:

6.1.1 *Binary (or greater) solvent pump*, capable of maintaining a pulse-free flow rate of 1-3 millilitres per minute

6.1.2 *Sample injector*, automatic or manual, capable of reproducibly injecting a 2 microlitre volume

6.1.3 *Column heater*, capable of maintaining a temperature of $30 \pm 0.2^{\circ}\text{C}$

6.1.4 *UV detector*, capable of measurements at 235 nm.

6.1.5 *Chart recorder or Data system*, capable of peak area integration.

6.2 *HPLC analytical column*, 250 mm by 4.6 mm by 5 μm cyano stationary phase

NOTE 2—Other chromatographic columns can be used provided it is ascertained that similar chromatographic performance is obtained.

6.3 *Magnetic Stirring Hotplate*.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent-grade chemicals are to be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades can 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.

7.1.1 *Acetanilide*, 99.9 % purity, to be used as an internal standard.

7.1.2 *Acetonitrile*, dry. Dry this and reagents below over molecular sieve for twenty-four hours.

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.

Current edition approved Sept. 1, 2011. Published October 2011. Originally approved in 2006. Last previous edition approved in 2006 as D7252 - 06. DOI:10.1520/D7252-06R11E01.

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.