



Standard Test Method for Unreacted Toluene Diisocyanates in Urethane Prepolymers and Coating Solutions by Gas Chromatography¹

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This standard has been approved for use by agencies of the Department of Defense.

^{ε1} NOTE—Editorial changes were made throughout in November 1996.

1. Scope

1.1 This test method covers the determination of free toluene diisocyanates in urethane prepolymers and unpigmented coating solutions.

NOTE 1—This test method may also be useful for the determination of free isocyanate monomers of other types upon taking precautions to confirm suitability.

1.2 There is no reason to believe this test method would not work for pigmented coatings.

1.3 The values stated in inch-pound 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 and health practices and determine the applicability of regulatory limitations prior to use.* For a specific hazard statement, see Section 8.

2. Referenced Documents

2.1 ASTM Standards:

D 2372 Practice for Separation of Vehicle from Solvent-Reducible Paints²

D 2698 Test Method for Determination of Pigment Content of Solvent-Reducible Paints by High-Speed Centrifuging²

E 260 Practice for Packed Column Gas Chromatography³

3. Summary of Test Method

3.1 A standard solution of the diisocyanate is analyzed by gas chromatography using an internal standard technique. The unknown specimen is then chromatographed, again using an internal standard, and a response factor is calculated from the standard solution. The unreacted diisocyanate content of the

unknown is calculated from the chromatogram of the specimen.

4. Significance and Use

4.1 This test method provides a measurement of the unreacted diisocyanate content of urethane prepolymers and coating solutions. This measurement is of basic importance to the health of the users.

5. Interferences

5.1 Under the test conditions described in this test method, other diisocyanates or impurities having identical or nearly identical elution times may interfere. It is generally necessary to know the identity of the diisocyanate being used in the specimen being tested, either by knowledge of the composition or by a separate analysis.

5.2 The temperature specifications for the injection port and column oven must be strictly followed to prevent thermal decomposition of the specimen. Decomposition can be detected by the failure of the recorder pen to return to the baseline after elution of the diisocyanate monomer.

5.3 Since resin solution specimens leave a nonvolatile residue in the injection port, the use of glass injection port liners is recommended. The glass liner should be changed daily or after each group of 10 specimens that are analyzed.

5.4 Since diisocyanates are highly reactive with water, a suitable drying cartridge should be used in the carrier gas line.

6. Apparatus

6.1 *Gas Chromatograph* equipped with flame ionization detector (FID) and capable of linear temperature programming to 300°C.

6.2 *Chromatographic Column*, 6.6 ft (2 m) by 0.125-in. (3.2-mm) outside diameter, 0.085-in. (2.1-mm) inside diameter stainless steel (or glass, 2-mm inside diameter), packed with a 10 % methyl vinyl silicone on 80/100 mesh high performance, white, flux-calcined diatomaceous earth. The packing is prepared by dissolving 1.0 g of the methyl vinyl silicone in 50 mL of methylene chloride. This solution is placed in an evaporating dish and 9 g of the diatomaceous earth is added with gentle

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² *Annual Book of ASTM Standards*, Vol 06.01.

³ *Annual Book of ASTM Standards*, Vol 14.02.

