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Standard Test Method for

**FREE TOLUENE DIISOCYANATE IN URETHANE
POLYMERS BY DISTILLATION¹**

This Standard is issued under the fixed designation D 2615; 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.

This method has been approved for use by agencies of the Department of Defense to replace Method 7426 of Federal Test Method Standard No. 141A and for listing in the DoD Index of Specifications and Standards.

[€] NOTE—An editorial change was made in the title in May 1976.

1. Scope

1.1 This method covers the determination of the free toluene diisocyanate (TDI) content of low molecular weight urethanes. As written, the method covers products containing from about 0.05% to about 2.0% of free toluene diisocyanate. Higher concentrations of TDI can be dealt with by altering the sample size, concentration of final solution, or absorption cell size.

2. Summary of Method

2.1 Solvent and toluene diisocyanate are removed from the urethane solution by vacuum distillation after the addition of a carrier solvent. The toluene diisocyanate is determined colorimetrically following conversion with sodium nitrite-Cellosolve² reagent to a product which has an absorption maximum at 440 nm.

3. Interferences

3.1 Other aromatic isocyanates are known to give a color with sodium nitrite-Cellosolve reagent. The wave length of maximum absorption and the extinction coefficients of these isocyanate derivatives are not known.

4. Apparatus

4.1 *Small-scale Vacuum Distillation Apparatus* (see Fig. 1).

4.2 *Lunge Weighing Bottle.*

4.3 *Cuvettes*, borosilicate, 1 and 2.5-cm.

4.4 *Spectrophotometer.*³

4.5 *Oil Bath*—A 600-ml tall-form beaker, without spout, containing heavy mineral oil.

4.6 *Dewar Flask*, 500 ml.

4.7 *Hot Plate.*

4.8 *Stopper*, standard taper, 14/35.

4.9 *Vacuum Pump.*

4.10 *Manometer.*

4.11 *Lab Jack.*

4.12 *Semilogarithmic Paper.*⁴

4.13 *Safety Shield.*

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 speci-

¹ This method is under the jurisdiction of ASTM Committee D-1 on Paint, Varnish, Lacquer, and Related Products.

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² Cellosolve is a registered trademark of Union Carbide Chemicals Co. The chemical name is ethylene glycol monoethyl ether.

³ Beckman Model B or equivalent has been found satisfactory.

⁴ Dietzgen D 340-L 112 or equivalent.