



Standard Test Method for Polyurethane Raw Materials: Determination of Hydrolyzable Chlorine of Isocyanates¹

This standard is issued under the fixed designation D4663; 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 This test method determines the hydrolyzable chlorine content of toluene-2,4-diisocyanate, toluene-2,6-diisocyanate, or mixtures of the two. It is acceptable to apply this test method to other isocyanates of suitable solubility. (See **Note 1**.) The main sources of hydrolyzable chlorine in the isocyanates are carbamoyl chloride and dissolved phosgene. Both of these compounds react with alcohols and water, forming ureas, carbamates, carbon dioxide, and hydrochloric acid. (See **Note 2**.)

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—It is possible that this test method is applicable to crude polymeric isocyanates. However, the precision with crude polymeric isocyanates has not been established.

NOTE 2—This standard is identical to ISO 15028.

2. Referenced Document

2.1 ASTM Standards:²

D883 Terminology Relating to Plastics

D1193 Specification for Reagent Water

2.2 ISO Standards:

ISO 15028 Plastics—Aromatic Isocyanates for Use in the Production of Polyurethanes—Determination of Hydrolyzable Chlorine³

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 Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, <http://www.ansi.org>.

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method see Terminology **D883**.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *hydrolyzable chloride*—the low level chlorine-containing components of the isocyanate, such as carbamoyl chlorides, which react with water or alcohol to form HCl.

4. Summary of Test Method

4.1 The hydrolyzable chlorine reacts with methanol, liberating hydrochloric acid. The titratable chlorides are then determined potentiometrically using a standard silver nitrate solution.

5. Significance and Use

5.1 This test method can be used for research or for quality control to characterize toluene diisocyanates. Hydrolyzable chlorine correlates with performance in some polyurethane systems.

6. Interferences

6.1 Thiocyanate, cyanide, sulfide, bromide, iodide, or other substances capable of reacting with silver ions, as well as substances capable of reducing silver ions in acid solution, will interfere with the determination.

7. Apparatus

7.1 *Weighing Bottle*, or any device capable of weighing a liquid by difference to the nearest 0.1 g.

7.2 *Hot Plate*, with magnetic stirrer.

7.3 *Potentiometric Titrator*, or pH meter.

7.4 *Combination Silver Billet Electrode*.

8. Reagents and Materials

8.1 *Purity of Reagents*—Use reagent grade chemicals 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