



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

This standard is issued under the fixed designation D 4663; 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. This test method may also be applied 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.* For specific hazards statements see Note 3.

NOTE 1—This test method may be applicable with crude polymeric isocyanates. However, the precision with crude polymeric isocyanates has not been established.

NOTE 2—There is no equivalent ISO standard.

2. Referenced Document

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics²

D 1193 Specification for Reagent Water³

3. Terminology

3.1 Definitions—For definitions of terms used in this test method see Terminology D 883.

3.2 *Description of Term 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 ion, as well as substances capable of reducing silver ion 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 *Silver-Silver Chloride Electrode*.

7.5 *Silver or Glass Electrode*.

7.6 *Calomel 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

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² *Annual Book of ASTM Standards*, Vol 08.01.

³ *Annual Book of ASTM Standards*, Vol 11.01.

