



Designation: **D4663—15 D4663 – 20**

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.

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, health, and environmental 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.

1.3 This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.

2. Referenced Document

2.1 *ASTM Standards:*²

[D883 Terminology Relating to Plastics](#)

[D1193 Specification for Reagent Water](#)

[E456 Terminology Relating to Quality and Statistics](#)

[E2935 Practice for Conducting Equivalence Testing in Laboratory Applications](#)

2.2 *ISO Standards:*

[ISO 15028 Plastics—Aromatic Isocyanates for Use in the Production of Polyurethanes—Determination of Hydrolyzable Chlorine](#)³

3. Terminology

3.1 *Definitions*—~~For definitions of terms~~ Terms used in this test method see standard are defined in accordance with Terminology [D883](#), unless otherwise specified. For terms relating to precision and bias and associated issues, the terms used in this standard are defined in accordance with Terminology [E456](#).

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 ~~May 1, 2015~~ April 1, 2020. Published ~~June 2015~~ April 2020. Originally approved in 1987. Last previous edition approved in 2010 as [D4663—10](#); [D4663 - 15](#). DOI: [10.1520/D4663-15](#); [10.1520/D4663-20](#).

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](#).

*A Summary of Changes section appears at the end of this standard

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *hydrolyzable chloride*—the ~~low level~~ 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 specifications are available.⁴ Other grades may 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.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I of Specification **D1193**.

8.3 *Concentrated Nitric Acid* (HNO₃ sp gr 1.42).

8.4 *Methanol*.

8.5 *Silver Nitrate Standard Solution* (0.01 N)—Standardize with standard hydrochloric acid, either gravimetrically or potentiometrically, frequently enough to detect changes of 0.0005 N.

9. Sampling

9.1 Since organic isocyanates react with atmospheric moisture, take special precautions in sampling. Usual sampling methods, even when conducted rapidly, can cause contamination of the sample with insoluble urea. Therefore, blanket the sample with dry air or nitrogen at all times. (**Warning**—Diisocyanates are eye, skin and respiratory irritants at concentrations above the occupational exposure limit (TLV or PEL). Diisocyanates can cause skin and respiratory sensitization (asthma) in some people. Once sensitized, it is essential to limit further exposure to diisocyanates. Use a combination of engineering controls and personal protective equipment, including respiratory, skin and eye protection, to prevent over-exposure to diisocyanates. Consult the product suppliers' Safety Data Sheet (SDS) for more detailed information about potential health effects and other specific safety and handling instructions for the product.)

10. Test Conditions

10.1 Since isocyanates react with moisture, keep laboratory humidity low, preferably around 50 % relative humidity. See Section 9 of this standard for guidance on how to keep moisture out of the sample.

11. Procedure

11.1 Weigh (by difference to the nearest 0.1 g) 9 to 11 g of sample from a sampling weighing bottle into a clean, dry 400-mL beaker (**Note 3**). Add 50 mL of methanol and stir. Stir continuously while the reaction starts at which point the beaker will become warm and crystals may form on the sides of the beaker (**Note 4**). Fill the beaker half-full with water (**Note 5**) and boil gently for 30 min.

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.