



Standard Test Methods for Polyurethane Raw Materials: Determination of the Isocyanate Content of Aromatic Isocyanates¹

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1. Scope

1.1 These test methods measure the isocyanate content of aromatic isocyanates used as polyurethane raw materials.

1.1.1 *Test Method A*—Unheated toluene-dibutylamine determines the amount of toluene diisocyanate in refined toluene-2,4-diisocyanate and toluene-2,6-diisocyanate, or mixtures of the two. Other isomers, if present, will be included in the determination. This test method may also be applied to other isocyanates of suitable reactivity and solubility.

1.1.2 *Test Method B*—Heated toluene-dibutylamine determines the amine equivalent of crude or modified isocyanates derived from toluene diisocyanate, methylenebis-(4-phenylisocyanate), and polymethylene polyphenylisocyanate.

1.1.3 *Test Method C*—Unheated trichlorobenzene-toluene-dibutylamine determines the isocyanate content of crude or modified isocyanates derived from toluene diisocyanate, methylene-bis-(4-phenylisocyanate), and polymethylene polyphenylisocyanate. This test method can also be used to assay isomer mixtures of toluene diisocyanate and methylene-bis-(phenylisocyanate).

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 warning and precautionary statements, see Note 2.

NOTE 1—There is no equivalent ISO standard.

2. Referenced Documents

2.1 ASTM Standards:

D 841 Specification for Nitration Grade Toluene²

D 883 Terminology Relating to Plastics³

¹ These test methods are under the jurisdiction of ASTM Committee D-20 on Plastics and are the direct responsibility of Subcommittee D20.22 on Cellular Plastics. These test methods were recommended to ASTM by the Society of the Plastics Industry Polyurethane Raw Materials Analysis Committee.

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Revisions to this edition include the addition of Note 1 as well as the lowering of the normal acidity level of crude isocyanates from 0.3 % to 0.05 %. The designation of pH in a nonaqueous mixture was changed to “apparent pH.” Section 24, Report, was added, and Precision and Bias, Sections 14, 19, and 25, were rewritten to reflect the statistical significance of the data available.

² Annual Book of ASTM Standards, Vol 06.03.

³ Annual Book of ASTM Standards, Vol 08.01.

D 1193 Specification for Reagent Water⁴

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals⁵

3. Terminology

3.1 Definitions:

3.1.1 *polyurethane, n*—a polymer prepared by the reaction of an organic diisocyanate with compounds containing hydroxyl groups.

3.1.1.1 *Discussion*—Polyurethanes or urethanes, as they are sometimes called, may be thermosetting, thermoplastic, rigid or soft and flexible, or cellular or solid (see Terminology D 883).

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *amine equivalent*—the weight of sample that will combine with 1.0-g equivalent weight of dibutylamine.

3.2.2 *assay*—the percent by weight of toluene diisocyanate present in the sample.

3.2.3 *isocyanate (NCO) content*—the weight percent of the NCO groups present in the sample.

4. Summary of Test Methods

4.1 All three test methods react the isocyanate sample with an excess of dibutylamine to form the corresponding urea. The NCO content is determined from the amount of dibutylamine consumed in the reaction. Each test method varies solvent or heating times to yield accurate assays of the sample in question.

4.1.1 *Test Method A*—The sample is added to an excess of dibutylamine in toluene and allowed to stand at room temperature for 15 min. The reaction mixture is diluted with isopropyl alcohol, and the excess dibutylamine is back-titrated colorimetrically with hydrochloric acid.

4.1.2 *Test Method B*—The sample is added to an excess of dibutylamine in toluene and stirred for 20 min. The resulting solution is then heated rapidly to 100°C, removed from the heat, and allowed to stand for 30 min. The reaction mixture is diluted with isopropyl alcohol, and the excess dibutylamine is back-titrated potentiometrically with hydrochloric acid.

4.1.3 *Test Method C*—The sample is added to a solution of dibutylamine in toluene and trichlorobenzene. The resulting

⁴ Annual Book of ASTM Standards, Vol 11.01.

⁵ Annual Book of ASTM Standards, Vol 15.05.