Designation: D 2572 – 97 (Reapproved 2003)ε1

Standard Test Method for Isocyanate Groups in Urethane Materials or Prepolymers[1]

This standard is issued under the fixed designation D 2572; 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.

This standard has been approved for use by agencies of the Department of Defense.

ε[1] NOTE—Keywords were added editorially in July 2003.

1. Scope

1.1 This test method covers the determination of the isocyanate group (NCO) content of a urethane intermediate or prepolymer.

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 hazard statements, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

D 841 Specification for Nitration Grade Toluene[2]

3. Summary of Test Method

3.1 The urethane prepolymer is allowed to react with an excess of di-*n*-butylamine in toluene (Note 1). After the reaction is complete, the excess of di-*n*-butylamine is determined by back titration with standard hydrochloric acid.

NOTE 1—Solvents other than toluene may be needed in some cases, for example, anhydrous dimethylformamide (DMF), but the procedure has not been evaluated using them.

4. Significance and Use

4.1 The percent NCO is used to establish unit ratios for stoichiometric reactions with co-reactants.

5. Interferences

5.1 Phosgene, the carbamyl chloride of isocyanate, hydrogen chloride, or any other acidic or basic impurities of sufficient strength will interfere. The concentration of these compounds is usually sufficiently low that their effect on the determination is negligible.

6. Apparatus

6.1 *Polyethylene Stoppers (or corks)* covered with aluminum foil to fit 250-mL Erlenmeyer flasks.

6.2 *Magnetic Stirrer* with PTFE-fluorocarbon-covered stirring bar.

6.3 *Pipet*,[3] 25-mL.

6.4 *Buret*,[3] 50- or 100-mL.

6.5 *Analytical Balance*.

7. Reagents

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests unless otherwise specified. 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 specifications are available.[4] Other grades may be used, provided that it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Bromphenol Blue Indicator*, Prepare 0.1 % solution by mixing 0.10 g of acid, nonwater soluble bromphenol blue with 1.5 mL of 0.1 *N* sodium hydroxide solution and diluting to 100 mL with distilled water.

7.3 *Di-n-butylamine Solution*, 0.1 *N* in dry toluene. Not standardized.

7.4 *Hydrochloric Acid Solution*, 0.1 *N* standardized using bromphenol blue indicator solution.

7.5 *Isopropyl Alcohol*.

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[1] This test method is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.33 on Polymers and Resins.

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[2] *Annual Book of ASTM Standards*, Vol 06.04.

[3] Burets and pipets shall conform to National Institute of Standards and Technology tolerances.

[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. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

7.6 *Toluene*, anhydrous, conforming to Specification D 841 or equivalent dried with molecular sieve pellets.

8. Hazards

8.1 Diisocyanates are strong skin irritants and sensitizers. They are also hazardous to the lungs. (TLV for toluene 2,4-diisocyanate is 0.005 ppm.) Avoid contact with skin, eyes, and clothing, and avoid breathing these substances. Use with adequate ventilation. In case of contact, wash with plenty of water and flush eyes for 15 min. Call a physician before reuse. See Supplier's Material Safety Data Sheet for additional information.

9. Procedure

9.1 Weigh to 0.1 g a specimen containing approximately 1.1 milliequivalents of NCO (for example, 1.0 g of prepolymer containing approximately 5 % NCO) in a 250-mL Erlenmeyer flask.

9.2 Add 25 mL of dry toluene (Note 2), place a stopper in the flask, and swirl by hand or on a mechanical agitator to dissolve the prepolymer. Solution may be aided by warming on a hot plate.

NOTE 2—If the polymer is insoluble, add 10 mL of dry, analytical grade acetone to the toluene.

9.3 Using a pipet, add 25.00 mL of 0.1 *N* di-*n*-butylamine solution and continue swirling for 15 min with stopper in place.

9.4 Add 100 mL of isopropyl alcohol and 4 to 6 drops of bromphenol blue indicator solution. Titrate with 0.1 *N* hydrochloric acid to a yellow end point.

9.5 Run a blank titration including all reagents above but omitting the specimen.

10. Calculations

10.1 Calculate the NCO content as follows:

$$NCO, \% = \frac{[(B - V) \times N \times 0.0420]}{W} \times 100 \quad (1)$$

where:

B	=	volume of HCl for titration of the blank, mL,
V	=	volume of HCl for titration of the specimen, mL,
N	=	normality of HCl,
0.0420	=	milliequivalent weight of the NCO group, and
W	=	grams of specimen weight, g.

11. Precision and Bias

11.1 The following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1 *Repeatability*—Two results obtained by the same operator should be considered suspect if they differ by more than 0.11 % absolute NCO.

11.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 0.4 % absolute NCO.

11.2 *Bias*—No bias can be determined for this test method since no standard resin exists.

12. Keywords

12.1 dicyclohexylmethane-4,4-diisocyanate (rMDI); hexamethylene diisocyanate; isophorone diisocyanate (IPDI); polymeric MDI; toluene, 2,4-diisocyanate based intermediate; toluene, 2,4-diisocyanate based prepolymer; urethane intermediate; urethane prepolymer;2,4-diphenylmethane diisocyanate;4,4-diphenylmethane diisocyanate