Standard Test Methods for Polyurethane Raw Materials: Determination of the Isocyanate Content of Aromatic Isocyanates[1]

This standard is issued under the fixed designation D 5155; 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 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, methylene*bis*-(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—Method C of this test method is equivalent to Method B of ISO 14896.

2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics[2]
D 1193 Specification for Reagent Water[3]
E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals[4]

2.2 *ISO Standard:*

ISO 14896 Polyurethane Raw Materials-Determination of Isocyanate Content

3. Terminology

3.1 *Definitions:*

3.1.1 *polyurethane*, *n*—a polymer prepared by the reaction of an organic diisocyanate or polyisocyanate 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 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 with hydrochloric acid.

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

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

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

[4] *Annual Book of ASTM Standards*, Vol 15.05.

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

4.1.3 *Test Method C*—The sample is added to a solution of dibutylamine in toluene and trichlorobenzene. The resulting solution is allowed to stand until it has cooled to room temperature. The reaction mixture is diluted with methanol and back-titrated with hydrochloric acid.

5. Significance and Use

5.1 These test methods can be used for research or for quality control to characterize isocyanates used in polyurethane products.

6. Interferences

6.1 Phosgene, the carbamyl chloride of the isocyanate, hydrogen chloride, and any other acidic or basic compounds will interfere. In refined isocyanates, these impurities are usually present in such low amounts that they do not affect the determination. While some crude or modified isocyanates contain acidities of up to approximately 0.05 %, the NCO content is not normally corrected.

7. Reagents and Materials

7.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. 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.[5] 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.

7.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Types I through IV of Specification D 1193.

8. Sampling

8.1 Since organic isocyanates react with atmospheric moisture to form ureas, take special precautions in sampling (see Note 2). Usual sampling methods (for example, sampling an open drum with a thief), 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.

NOTE 2—**Warning:** Organic isocyanates are toxic when absorbed through the skin, or when the vapors are breathed. **Precaution**: Provide adequate ventilation and wear protective gloves and eyeglasses.

9. Test Conditions

9.1 Since isocyanates react with moisture, keep the laboratory humidity low, preferably below 50 % relative humidity.

TEST METHOD A—UNHEATED TOLUENE-DIBUTYLAMINE

10. Apparatus

10.1 Any weighing device that can weigh a liquid by difference to the nearest 0.001 g.

10.2 *Cooling Bath*—Any container approximately 50 mm deep filled with ice and water.

10.3 Pipet capable of reproducibly delivering 50 ± .05 mL.

11. Reagents

11.1 *Bromcresol Green Indicator Solution*—Using 1.5 mL of 0.1 *N* sodium hydroxide, extract the bromcresol green from 0.100 g of bromcresol green indicator-grade powder, stirring vigorously until the amount of insoluble residue remains constant. Decant the aqueous portion into a 100-mL volumetric flask and dilute to the mark with water.

11.2 *Dibutylamine Solution (260 g/L)*—Dilute 260 g of dry dibutylamine to 1 L with dry toluene. Dry the solution with a drying agent.[6]

11.3 *Hydrochloric Acid* (1 *N*)—Prepare 1 *N* HCl (hydrochloric acid) and standardize frequently enough to detect changes of 0.001 *N*.

11.4 *Isopropyl Alcohol*.

11.5 *Toluene*, dry with a drying agent.[6]

12. Procedure

12.1 Run sample and blank determinations side by side. Run the blank determination exactly as described as follows, but without adding the sample.

12.2 Add a magnetic stirring bar and 40 mL of dry toluene to a 500-mL Erlenmeyer flask that has been rinsed successively with water, alcohol, and high-purity acetone, dried at 100°C, and allowed to cool in a desiccator. Accurately add, by pipet or buret,[7] 50 mL of dibutylamine solution and mix carefully.

12.3 While stirring the contents of the flask, slowly add 6.5 to 7.0 g of the sample weighed to the nearest 0.001 g (Note 3). Wash down the sides of the flask with 10 mL of dry toluene, then stopper the flask loosely and allow it to stand at room temperature for 15 min.

NOTE 3—If spattering is anticipated, cool the flask and contents in the cooling bath before adding the sample and continue to cool until the heat of reaction is dissipated. Add 10 mL of dry toluene, stopper the flask loosely, and allow the contents to come to room temperature.

12.4 Add 225 mL of isopropyl alcohol and 0.8 mL of bromcresol green indicator solution. Titrate with 1 *N* HCl solution in a 50 or 100-mL buret[8] while stirring the flask contents with the magnetic stirring bar. Near the end point, slowly add the HCl dropwise. The end point is reached when the blue color disappears and a yellow color appears that persists for at least 15 s (Note 4).

NOTE 4—Alternatively, the end point may be determined using a potentiometer and electrodes. When using this apparatus, it may be

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[5] *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.

[6] The Linde 4A Molecular Sieve, or its equivalent, has been found suitable. The Linde 4A Molecular Sieve is available from Dow Chemical Corp., Specialty Gas-Linde Division, 2 Greenway Plaza, Suite 901, Houston, TX 77046, or from other Dow Chemical locations nationwide.

[7] Pipets and burets shall conform to National Institute of Standards and Technology tolerances, as given in Peffer, E. L., and Mulligan, G. C., "Testing of Glass Volumetric Apparatus," *NIST Circular C 434*, 1941, available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20025.

[8] If an isocyanate other than TDI is used, substitute the equivalent weight of the material being analyzed.

necessary to transfer the solution to a 600-mL beaker prior to titration. After transfer, rinse the Erlenmeyer flask with 25 mL of isopropyl alcohol and add the rinse to the 600-mL beaker. To titrate, immerse the calomel and glass electrodes of the pH meter (standardized with pH 4.0 and pH 7.0 standard buffers) and titrate the sample to the break that occurs at approximately pH 4.2 to 4.5 with 1.0 *N* HCl while stirring the solution with a stirring bar.

13. Calculation

13.1 Calculate the assay, %, as follows:

$$\text{Assay} = (B - S)(N)(87.08)(100)/1000\ W \quad (1)$$

When constants are combined, this equation reduces to

$$\text{Assay} = 8.708\ N(B - S)/W \quad (2)$$

where:

B = HCl required for titration of the blank, mL,
S = HCl required for titration of the sample, mL,
N = normality of the HCl, meq/mL,
W = sample used, g,
87.08 = equivalent weight of TDI, mg/meq,[10]
1000 = conversion from g to mg, and
100 = conversion to percent.

14. Precision and Bias [9]

14.1 Attempts to develop a precision and bias statement for this test method have not been successful due to the limited number of laboratories participating in round-robin tests. Data on precision and bias cannot be given for this reason. Anyone wishing to participate in the development of precision and bias data should contact the Chairman, Subcommittee D20.22 (Section D20.22.01), ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

14.2 A limited round robin was conducted.

14.2.1 It has been estimated that duplicate results by the same analyst should be considered suspect if they differ by 0.4 % TDI.

14.2.2 It has been estimated that results reported by different laboratories should be considered suspect if they differ by 0.8 % TDI.

14.3 There are no recognized standards by which to estimate the bias of this test method.

TEST METHOD B—HEATED TOLUENE-DIBUTYLAMINE

15. Apparatus

15.1 *Potentiometric Titrator*, or pH meter.

15.2 *Calomel Electrode or a combination electrode*.

15.3 *Glass Electrode*.

15.4 Any weighing device suitable for weighing a liquid sample by difference to the nearest 0.001 g.

15.5 *Magnetic Stirrer*.

15.6 *Thermometer*, from – 10 to 100°C range.

15.7 Pipet capable of reproducibly delivering 25 ± .025 mL.

16. Reagents

16.1 *Dibutylamine Solution (260 g/L)*—Dilute 260 g dry dibutylamine to 1 L with dry toluene.

16.2 *Hydrochloric Acid* (1 *N*)—Prepare 1 *N* hydrochloric acid (HCl) and standardize frequently enough to detect changes of 0.001 *N*.

16.3 *Isopropyl Alcohol*, 99 % minimum purity.

16.4 *Toluene*, dry, dried with a drying agent.[6]

17. Procedure

17.1 Add 50 mL of dry toluene to a dry 600-mL beaker. Pipet 25[7] mL of the dibutylamine solution into the beaker. Swirl the beaker to mix the contents.

17.2 Transfer to the beaker 0.02 to 0.03 equivalents of the sample weighed to the nearest 0.001 g. The amount of sample needed can be calculated from the following equation:

$$\textit{weight of sample}\ (g) = 105/\textit{expected}\ \%\ NCO \quad (3)$$

Start the magnetic stirrer carefully and rinse the sides of the beaker with an additional 10 mL of dry toluene. Cover the beaker and continue mixing for an additional 20 min.

17.3 Place the beaker on a hot plate with the – 10 to 100°C thermometer in the sample. Heat the sample mixture rapidly with stirring, so that the solution reaches a temperature of 95 to 100°C in 3½ to 4½ min. Do not overheat. Quickly remove the beaker from the hot plate, cover it with a watchglass, and allow it to stand for 30 min.

17.4 Cool the beaker and contents to room temperature and add 225 mL of isopropyl alcohol.

17.5 Titrate potentiometrically with 1.0 *N* HCl to the break that occurs at apparent pH approximately 4.2 to 4.5 (for manual titration see Note 5, below).

17.6 Prepare and titrate a blank exactly as described in 17.1-17.5, but without adding the sample.

18. Calculation

18.1 Calculate the amine equivalent as follows:

$$\text{amine equivalent} = \frac{1000(W)}{N(B - S)} \quad (4)$$

18.2 Calculate the percent NCO as follows:

$$\%\ NCO = 42.02\ (B - S)\ (N)\ (100)\ /\ 1000\ (W) \quad (5)$$

When constants are combined, this equation reduces to:

$$\%\ NCO = 4.202\ (B - S)\ N\ /\ W \quad (6)$$

B = HCl required for titration of blank, mL,
S = HCl required for titration of sample, mL,
N = normality of HCl, meq/mL,
W = sample used, g, and

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[9] Supporting data are available from ASTM Headquarters. Request RR:D20-1089.

4.202 = constant combining the equivalent weight of NCO (42.02) mg/meq, conversion of g to 1000 mg, and conversion to 100%

19. Precision and Bias [10]

19.1 Attempts to develop a precision and bias statement for this test method have not been successful due to the limited number of laboratories participating in round-robin tests. Data on precision and bias cannot be given for this reason. Anyone wishing to participate in the development of precision and bias data should contact the Chairman, Subcommittee D20.22 (Section D20.22.01), ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

19.2 A limited round robin was conducted.

19.2.1 It has been estimated that duplicate results by the same analyst should be considered suspect if they differ by 0.80 amine equivalents (0.2 % at 30.0 % NCO).

19.2.2 It has been estimated that results reported by different laboratories should be considered suspect if they differ by 2.0 amine equivalents (0.4 % at 30 % NCO).

19.3 There are no recognized standards by which to estimate the bias of this test method.

TEST METHOD C—UNHEATED TRICHLOROBENZENE-TOLUENE-DIBUTYLAMINE

20. Apparatus

20.1 *Potentiometric Titrator*, or pH meter (Note 5).

20.2 *Calomel Electrode or a combination electrode*.

20.3 *Glass Electrode*.

20.4 Any weighing device suitable for weighing a liquid sample by difference to the nearest 0.001 g.

20.5 *Magnetic Stirrer*.

20.6 Pipet capable of reproducibly delivering 20 ± .02 mL.

NOTE 5—If a potentiometric titrator is not available, the titration can be performed using a conventional 50-mL buret and bromophenol blue indicator (0.04 % aqueous bromophenol blue, sodium salt, reagent grade). Titrate the blank and the sample solutions to the first appearance of a stable yellow color. (The solution will change from a blue color at the start of the titration, to a bluish-green intermediate color, to a yellow color at the end point. Recognition of the end point is a matter of experience, but better defined color changes are obtained when the acid is titrated rapidly into the solution until the first flash of yellow color is observed. This flash of color normally appears within a few tenths of a millilitre of the end point.)

21. Reagents

21.1 *Dibutylamine*.

21.2 *Methanol*.

21.3 *Toluene*, dry, dried with a drying agent.[6]

21.4 *Trichlorobenzene-1,2,4 (TCB)*—Dry over Type 4A molecular sieves.

21.5 *Dibutylamine Solution* (2 *N*)—Dilute 260 g of dibutylamine to 1 L with dry toluene and dry over Type 4A molecular sieves.

21.6 *Methanolic Hydrochloric Acid* (1 *N*)—Prepare 1 *N* hydrochloric acid from methanol concentrated HCl and standardize frequently enough to detect changes of 0.001 *N* (Note 6).

NOTE 6—In order to have homogenous titrations, it is recommended that methanolic HCl be used in this procedure. If desired, aqueous HCl can be used. However, turbidity will be encountered in some titrations. It is recommended that 200 to 250 mL of methanol be added to the reacted product to minimize the formation of two layers. Experience has shown that if the mixtures are agitated vigorously, inhomogeneity can be tolerated without adversely affecting the results.

22. Procedure

22.1 Add 25 mL of TCB to a dry 250-mL wide-mouth Erlenmeyer flask. Pipet 20 mL of the dibutylamine solution into the flask. Swirl to mix the contents.

22.2 Transfer the approximate amount of sample required weighed to the nearest 0.001 g to the flask. The approximate amount of sample required is calculated from the following equation:

$$\text{weight of sample, g} = \frac{84}{\text{expected \% NCO}} \tag{7}$$

22.3 Cover the flask and swirl the contents until the solution is homogeneous. The reaction mixture will warm to approximately 40°C.

22.4 Let the sample stand until the reaction mixture reaches room temperature (20 to 25 min) and add 100 mL of methanol to the flask (see Note 5).

22.5 Titrate potentiometrically with 1.0 *N* HCl to the break that occurs at apparent pH approximately 4.2 to 4.0.

22.6 Prepare and titrate a blank exactly as described in 22.1-22.5, but without adding the sample.

23. Calculation

23.1 Calculate the NCO, %, as follows:

$$\% \, NCO = 42.02\,(B - S)\,(N)\,(100)\,/\,1000\,(W) \tag{8}$$

When constants are combined, this equation reduces to:

$$\% \, NCO = 4.202\,(B - S)\,N\,/\,W \tag{9}$$

where:
B = HCl required for titration of blank, mL,
S = HCl required for titration of sample, mL,
N = normality of HCl, meq/mL,
W = sample used, g, and
4.202 = constant combining the equivalent weight of NCO (42.02) mg/meq, conversion of g to 1000 mg, and conversion to 100 %.

24. Report

24.1 The result is reported as the average of duplicates, expressed as percent NCO, to the nearest 0.01 %. Any unusual conditions during operation also should be reported, such as any heating required to effect solution before titration, or end point identified different from that described in 22.5.

25. Precision and Bias [10]

25.1 Table 1 is based on a round robin involving nine laboratories and conducted in 1991 in accordance with Practice

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[10] Supporting data are available from ASTM Headquarters. Request RR: D20-1040. The precision estimates are based on an interlaboratory study performed in 1989 on one sample each of Lupranate M20S (BASF), PAPI 20 and Isonate 143L (Dow), Mondur PF (Bayer), and Rubinate HF185 (Rubicon). Eleven industrial laboratories participated in the test method evaluation.

TABLE 1 Round-Robin Percent NCO Data In Accordance with Practice E 180[A]

	Average	S_r[B]	S_R[C]	r[D]	R[E]	df[F]
Lubrinate M20S	31.30	0.082	0.206	0.230	0.577	9
Rubinate 1850	30.78	0.082	0.194	0.230	0.543	9
PAPI 20	29.57	0.080	0.172	0.224	0.482	8
Isonate 143L	28.83	0.126	0.230	0.353	0.644	9
Mondur PF	22.63	0.048	0.120	0.134	0.336	9
MDI	33.53	0.011	0.080	0.031	0.224	6
TDI (see 25.2.4)	48.18	0.078	0.126	0.218	0.353	2

[A] Values in units of percent NCO.

[B] S_r = within-laboratory standard deviation of the replicates.

[C] S_R = between-laboratories standard deviation of the average.

[D] r = within-laboratory repeatability limit = $2.8 \cdot S_r$.

[E] R = between-laboratories reproducibility limit = $2.8 \cdot S_R$.

[F] df = degrees of freedom in the data.

E 180. All labs used potentiometric titration for the generation of the data used in this study. Except for MDI and TDI, all the samples were prepared at one source, but the individual specimens were prepared at the laboratories that tested them. The MDI and TDI samples were freshly produced material at the laboratory site. Each test result was the average of two individual determinations.

NOTE 7—**Caution:** The following explanations of *r* and *R* (25.2.1-25.2.4) are intended only to present a meaningful way of considering the approximate precision of this test method. The data in Table 1 should not be rigorously applied to the acceptance or rejection of material, as those data are specific to the round robin and may not be representative of other lots, conditions, materials, or laboratories. Users of this test method should apply the principles outlined in Practice E 180 or E 691 to generate data specific to their laboratory and materials or between specific laboratories. The principles of 25.2.1-25.2.4 then would be valid for such data.

25.2 *Precision*

25.2.1 *Repeatability, (r)*—Comparing two replicates for the same material, obtained by the same operator, using the same equipment on the same day. The two replicate results should be judged not equivalent if they differ by more than the *r* value for that material.

25.2.2 *Reproducibility, (R)*—Comparing two results, each the mean of replicates, for the same material, obtained by different operators, using different equipment in different laboratories on different days. The two test results should be judged not equivalent if they differ by more than the *R* value for that material.

25.2.3 Any judgment in accordance with 25.2.2 and 25.2.3 would have an approximate 95 % (0.95) probability of being correct.

25.2.4 There are insufficient degrees of freedom to make a statistically acceptable determination for TDI. The data in Table 1 are provided for information only. The precision for TDI isomers is expected to be similar to results obtained for MDI.

25.3 There are no recognized standards by which to estimate the bias of this test method.

26. Keywords

26.1 isocyanates; isocyanates aromatic; methylene-*bis*-(4-phenylisocyanate); polymethylene polyphenylisocyanate; polyurethane; raw materials; test method; titration; toluene diisocyanate

SUMMARY OF CHANGES

This section identifies the location of selected changes to this test method. For the convenience of the user, Committee D20 has highlighted those changes that may impact the use of this test method. This section may also include descriptions of the changes or reasons for the changes, or both.

D 5155 – 01:
(*1*) Changed ISO equivalency statement.
(*2*) Removed reference to Specification D 841, as well as its corresponding footnote.
(*3*) Added reference to ISO 14896.
(*4*) Added the term polyisocyanate to 3.1.1.
(*5*) Removed references to detection methodology in 4.1.1, 4.1.2, and 4.1.3.
(*6*) Changed the accepted types of water in 7.2.
(*7*) Removed any reference to Lunge Pipet.
(*8*) Added 10.3, 15.7, 20.6.
(*9*) Removed any reference to glassware in 12.4.
(*10*) Revised Note 4.
(*11*) Numbered equations in 13.
(*12*) Updated company name in Footnote 6.
(*13*) Revised 15.2, 17.5, 17.6, 18.2, 20.2, 20.4, 21.3, 21.5, 22.5, 22.6, 23.1, 24.
(*14*) Added text and equation to 17.2.
(*15*) Re-organized 25.2.
(*16*) Added reference to text about one sample in Table 1.

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