



Standard Test Method for Polyurethane Raw Materials: Determination of Freezing Point of Toluene Diisocyanate Mixtures¹

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1. Scope

1.1 This test method determines the freezing points of mixtures of toluene-2,4-diisocyanate and toluene-2,6-diisocyanate. It should not be used to measure the isomer content of the mixture. (See Note 1.)

1.2 *This standard does not purport to address all of the safety problems, 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 and Note 3.

NOTE 1—There is no equivalent ISO standard.

2. Referenced Documents

2.1 *ASTM Standards:*

D 883 Terminology Relating to Plastics²

3. Terminology

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

4. Summary of Test Method

4.1 The toluene diisocyanate (TDI isomer) mixture is cooled slowly by stirring until it crystallizes. Since supercooling occurs, the temperature first falls, then rises to a maximum, and then falls again. This maximum temperature is the freezing point of the mixture.

5. Significance and Use

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

5.2 For toluene diisocyanate, the test methods for assay, freezing point, and specific gravity help determine purity, while the other test methods (isomer content, total chlorine, hydrolyzable chlorine, acidity, and color) may relate to reactivity or performance in polyurethane systems.

6. Apparatus

6.1 *Air Jacket*, glass test tube, 50-mm outside diameter and 175 mm long.

6.2 *Freezing Point Tube*, glass test tube, 25-mm outside diameter and 150 mm long.

6.3 *Ice Bath*, 1-L beaker or other container 180 mm deep.

6.4 *Stirrer*, made from a 2-mm glass rod (or 1-mm stainless steel wire) with one end made into a circle perpendicular to the shaft so that it moves freely between the thermometer stem and the freezing point tube. Operate the stirrer either manually or mechanically.

6.5 *Thermometer*, graduated to 0.1°C and calibrated from – 5 to + 30°C.³

6.6 *Timer*, read to the nearest 1 s.

7. Sampling

7.1 Since organic isocyanates react with atmospheric moisture, take special precautions in sampling (**Warning: Precaution**—Note 2). Usual sampling methods (for example, sampling an open drum thief), even when carried out 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 they are absorbed through the skin, or when the vapors are breathed. **Precaution**—Provide adequate ventilation and wear protective gloves and eyeglasses.

8. Test Conditions

8.1 Since isocyanates react with moisture, keep laboratory humidity low, preferably around 50 % relative humidity.

9. Procedure (Note 3)

9.1 Fit the small test tube with a two-hole cork stopper, and insert the thermometer through the center hole and shaft of the stirrer through the other hole. Place two 3-mm layers of absorbent cotton in the bottom of the large test tube. Insert the small test tube up to its lip into a cork stopper or cork ring that just fits into the large test tube.

9.2 Fill the ice bath with ice water, and sodium chloride (NaCl) if necessary, to bring the temperature to about 10°C below the expected freezing point of the test sample.

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

³ A thermometer of this range may be purchased from the Walter H. Kessler Co., 219 Westbury Ave., Carle Place, Long Island, NY.

9.3 Add the sample to the small test tube to a height of 90 to 100 mm.

NOTE 3—: Precaution—Carry out this test method in an exhaust hood and run the test method quickly to avoid reaction of toluene diisocyanate with atmospheric moisture.

9.4 Insert the cork carrying the stirrer and thermometer into the small test tube and adjust the thermometer so that its immersion mark is just level with the top of the sample. Cool the small test tube in the ice bath, while stirring, to about 4 to 5°C below the expected freezing point. Wipe the outside of the small test tube dry and immediately insert it into the large test tube. Place the assembled apparatus in the ice bath.

9.5 Stir the sample continuously and vigorously and observe the thermometer readings closely. The temperature should rise to a maximum, remain constant at this maximum for 5 to 10 min, then fall again (Note 4). The minimum temperature is due to supercooling before solidification starts and should not be more than 6°C below the maximum. Apply the thermometer calibration correction to the maximum constant temperature observed.

NOTE 4—If distinct minimum or maximum points are not evident, or the temperature does not remain constant at the maximum for at least 1 min, the determination should be repeated if the freezing temperature plateau is not obvious. It may be desirable to plot a temperature-versus-time curve. In this case, a timer should be used. The highest plateau in the

curve is the freezing point of the sample. For some samples, it may be helpful to add a seed crystal of the sample.

10. Report

10.1 Report the maximum constant temperature to the nearest 0.1°C as the freezing point.

11. Precision and Bias

11.1 *Precision*—Attempts to develop a precision and bias statement for this test method have not been successful. For this reason, data on precision and bias cannot be given. Because this test method does not contain a numerical precision and bias statement, it shall not be used as a referee test method in case of dispute. Anyone wishing to participate in the development of precision and bias data should contact the Chairman, Subcommittee D20.22 (Section D20.22.01), ASTM, 1916 Race Street, Philadelphia, PA 19103.

11.1.1 It is estimated that duplicate results by the same analyst should be considered suspect if they differ by more than 0.2°C.

11.2 *Bias*—The bias for this test method has not yet been determined.

12. Keywords

12.1 freezing point; isocyanates; polyurethane raw materials; TDI

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