



Designation: D4889 – 04

# Standard Test Methods for Polyurethane Raw Materials: Determination of Viscosity of Crude or Modified Isocyanates<sup>1</sup>

This standard is issued under the fixed designation D4889; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope\*

1.1 These test methods (A and B) determine the viscosity of crude or modified *isocyanates*. They are applicable to products derived from toluene diisocyanate, methylene-bis-(4-phenylisocyanate), and polymethylene polyphenylisocyanates (see **Note 1**).

**NOTE 1**—Test method A includes a procedure for measuring dynamic viscosity using a rotational Brookfield instrument. Test method B is simply a reference to a general procedure for measuring kinematic viscosity using a Cannon-Fenske instrument, **D445**.

1.2 The values stated in SI units are to be regarded as standard.

1.3 *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 hazards statements see **Warnings** at the end of **5.1** and **10.5**.

**NOTE 2**—There is no equivalent ISO standard.

## 2. Referenced Documents

- 2.1 *ASTM Standards*:<sup>2</sup>
- D445** Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
  - D883** Terminology Relating to Plastics
  - E1** Specification for ASTM Liquid-in-Glass Thermometers

## 3. Terminology

3.1 For definitions of terms used in these test methods see Terminology **D883**.

<sup>1</sup> 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.

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<sup>2</sup> 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.

## 4. Significance and Use

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

4.2 Viscosity measures the resistance of a fluid to uniform continuous flow without turbulence or other forces.

4.3 Some isocyanates exhibit non-Newtonian behavior under certain conditions. Whenever possible, generate results for comparison under the same conditions, that is, the same spindle/speed combination for Brookfield viscosity and the same tube size for Cannon-Fenske viscosity.

## 5. Sampling

5.1 Since organic *isocyanates* react with atmospheric moisture, special precautions must be taken in sampling (see **Warning** at the end of this paragraph). Usual sampling methods (for example, sampling an open drum with a thief), even when carried out rapidly, can cause contamination of the sample with insoluble urea. Therefore, the sample must be blanketed with dry air or nitrogen at all times. **Warning**—Organic *isocyanates* are harmful when they are absorbed through the skin, or when the vapors are breathed. Provide adequate ventilation and protective gloves and wear eyeglasses.

## 6. Test Conditions

6.1 Since isocyanates react with moisture, keep laboratory humidity low, preferably about 50 % relative humidity.

### TEST METHOD A—BROOKFIELD VISCOSITY

## 7. Summary of Test Method

7.1 The viscosity is measured at  $25 \pm 0.3^\circ\text{C}$  with a Brookfield viscometer, either Model LVF, LVT, RVF, or RVT.<sup>3</sup>

## 8. Interferences

8.1 The temperature and container size are important factors in measuring Brookfield viscosity accurately. Deviation from the prescribed conditions will affect the accuracy of the results.

<sup>3</sup> Available from Brookfield Engineering Laboratories, 240 Cushing St., Stoughton, MA 02072.

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

**TABLE 1 Guide to Selecting the Rotational Speed Using the Number 1 Spindle**

Viscosity range of sample in mPa s (cP)	LVF Rotational speed (rpm)	LVT Rotational speed (rpm)	RVF Rotational speed (rpm)	RVT Rotational speed (rpm)
10-80	60	60	20	100
80-160	30	30	20	50
160-400	12	12	20	20

## 9. Apparatus

9.1 *Brookfield Synchro-lectric Viscometers*,<sup>3</sup> Model LVF, LVT, RVF, or RVT is to be used. Calibrate the instrument periodically with Brookfield Engineering Laboratories 10 mPa s (cP) or 100 mPa s (cP) Viscosity Standard Fluids.

9.2 *Spindle No. 1*, The No. 1 Spindles for the LV and RV instruments are different and, therefore, it is important to use the correct spindle type.

9.3 *Constant-Temperature Bath*, capable of maintaining a temperature of  $25 \pm 0.3^\circ\text{C}$ .

9.4 *Bath and Sample Thermometers*, graduated in  $0.1^\circ\text{C}$  subdivisions and standardized for the range of use to the nearest  $0.01^\circ\text{C}$ . An ASTM Saybolt Viscosity Thermometer having a range of  $19\text{--}27^\circ\text{C}$  and conforming to the requirements for Thermometer 17C in Specification E1 is recommended.<sup>6</sup>

9.5 *Wide-Mouth 1-Quart Paint Can*—Alternatively, a 1-quart jar or a 600-mL beaker can be used.

## 10. Procedure

10.1 Place sufficient sample in a 1-quart paint can to cover the immersion mark on the viscometer spindle. Equilibrate the sample in the  $25^\circ\text{C}$  constant-temperature bath.

10.2 Attach the No. 1 spindle to the viscometer following the directions supplied with the instrument. Set the speed using the guidelines in Table 1 (see Note 3). If the viscosity range of the sample is unknown, selection is accomplished by trial and error.

NOTE 3—The No. 1 spindle is suitable for the majority of crude or modified isocyanates. If it is necessary to use a different spindle and speed combination, conversion factors for spindle number versus spindle speed are given in Table 2. A dial reading as close to the center of the scale as possible is recommended. When there is a choice between two combinations with the same conversion factor, choose the combination with the lower speed.

10.3 Immerse the viscometer spindle and guard into the sample to the immersion line marked on the spindle. Avoid forming air bubbles under the spindle during immersion. If bubbles are observed, detach the spindle, keeping it in the sample, and stir until the bubbles are released, then reattach the spindle. Center the spindle in the test material.

10.4 Press down the viscometer clutch lever and start the motor. Release the clutch lever after the spindle has made 8 to

10 revolutions. Depress the clutch lever, stop the motor, and read the scale. If the pointer is not in view or if it is below 10 % torque when the dial comes to rest, rapidly throw the motor switch on and off (with the clutch lever still depressed) until the pointer is in view and is above 10 % torque.

10.5 Repeat the procedure until the readings (after each 8 to 10 revolutions) agree to within one scale division. **Warning**—Always release the clutch lever when the spindle is still immersed in the liquid so that pointer will float back rather than snap back to zero.

## 11. Calculation

11.1 Viscosity, mPa s (cP) =  $A \times F$ .

11.1.1  $A$  = dial reading on the 100 scale.

11.1.2  $F$  = conversion factor (see Table 2).

## 12. Precision and Bias

12.1 *Precision*—Attempts to develop a precision and bias statement for this test method have not been successful; however, the precision is expected to be equivalent to that reported by the instrument manufacturer. 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 D 20.22 (Section D 20.22.01), ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

12.2 *Bias*—The bias of this test method has not yet been determined.

## TEST METHOD B—CANNON-FENSKE

### 13. General:

13.1 A general test method for Cannon-Fenske viscosity which applies to isocyanates as well as other materials is published in Test Method D445.<sup>4</sup>

### 14. Keywords

14.1 Brookfield; Cannon-Fenske; isocyanates; polyurethane raw materials; viscosity

<sup>4</sup> Supporting data are available from ASTM Headquarters. Request D01-1132.