



Standard Test Methods for Polyurethane Raw Materials: Determination of Viscosity of Crude or Modified Isocyanates¹

This standard is issued under the fixed designation D 4889; 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 determine the viscosity of crude or modified *isocyanates*. It is applicable to products derived from toluene diisocyanate, methylene-bis-(4-phenylisocyanate), and polymethylene polyphenylisocyanates. (See Note 1.)

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 hazards statements see Note 2 and 10.5.

NOTE 1—There is no equivalent ISO standard.

2. Referenced Documents

2.1 ASTM Standards:

- D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)²
- D 883 Terminology Relating to Plastics³
- E1 Specification for ASTM Thermometers⁴

3. Terminology

3.1 For definitions of terms used in these test methods see Terminology D 883.

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 uniformly continuous flow without turbulence or other forces.

5. Sampling

5.1 Since organic *isocyanates* react with atmospheric moisture, special precautions must be taken in sampling (**Caution**,

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, the sample must be blanketed with dry air or nitrogen at all times.

NOTE 2—**Caution:**—Organic *isocyanates* are toxic when they are absorbed through the skin, or when the vapors are breathed. Adequate ventilation should be provided and protective gloves and eyeglasses should be worn.

6. Test Conditions

6.1 Since isocyanates react with moisture, laboratory humidity should be kept low, preferably around 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 or RVF.⁵

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.

9. Apparatus

9.1 *Brookfield Synchro-lectric Viscometers*⁵, Model LVF with speeds of 60, 30, 12, and 6 r/min, or Model RVF with speeds of 20, 10, 4, and 2 r/min, are to be used. The instrument should be calibrated periodically with Brookfield Engineering Laboratories 10-cP or 100-cP Viscosity Standard Fluids.

9.2 *Spindle No. 1*, The No. 1 Spindles for the LVF and RVF 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°C subdivisions and standardized for the range of use to the nearest 0.01°C . An ASTM Saybolt Viscosity Thermometer

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.

Current edition approved July 10, 1998. Published January 1999. Originally published as D 4889 – 88. Last previous edition D 4889 – 93.

² *Annual Book of ASTM Standards*, Vol 05.01.

³ *Annual Book of ASTM Standards*, Vol 08.01.

⁴ *Annual Book of ASTM Standards*, Vol 14.03.

⁵ Available from Brookfield Engineering Laboratories, 240 Cushing St., Stoughton, MA 02072.

having a range of 19–27°C and conforming to the requirements for Thermometer 17C in Specification E1 is recommended.⁶

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°C constant-temperature bath.

10.2 Attach the No. 1 spindle to the machine, (see Note 3), following the directions supplied with instrument. Set speed indicator as follows:

10.2.1 *Viscometer Model LVF*—60 r/min.

10.2.2 *Viscometer Model RVF*—20 r/min.

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, the speed of viscometer will also have to be changed. Conversion factors for spindle number versus spindle speed are given in Table 1. It is important to obtain a reading as close to the center of the scale as possible. When there is a choice, choose the spindle which gives 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 reinsert the spindle.

10.4 Press down the viscometer clutch lever and start the motor. Release the clutch lever until the spindle has made 8 to 10 revolutions. Depress the clutch lever, stop the motor, and read the scale. If, at higher speeds, the point is not in view when the dial comes to rest, rapidly throw the motor switch on and off (with the clutch lever still depressed) until the pointer reaches the window.

TABLE 1 Correction Factors Corresponding to Various Combinations of Spindles and Rotational Speeds

Model	Spindle Number	Correction Factors			
Rotational speed, r/min		6	12	30	60
LVF	1	10	5	2	1
	2	50	25	10	5
	3	200	100	40	20
	4	1 000	500	200	100
Rotational speed, r/min		2	4	10	20
RVF	1	50	25	10	5
	2	200	100	40	20
	3	500	250	100	50
	4	1 000	500	200	100
	5	2 000	1 000	400	200
	6	5 000	2 500	1 000	500
	7	20 000	10 000	4 000	2 000

10.5 Repeat the procedure until the readings (after each 8 to 10 revolutions) agree to within one scale division. **Caution**—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, $cP = A \times F$.

11.2 For Model LVF— $A =$ reading on 100 scale, $F = 1$.

11.3 For Model RVF— $A =$ reading on 100 scale, $F = 5$.

11.4 If any other combination of spindle size and speed is used with either model viscometer, the factor F should be changed (see Table 1).

11.5 At 60 r/min, air resistance on the pointer has a certain effect. Values obtained should be reduced as follows: No. 1 spindle, deduct 0.4 cP; No. 2 spindle, deduct 2.0 cP; No. 3 spindle, deduct 8.0 cP, No. 4 spindle, deduct 40.0 cP.

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 D 445.⁶

14. Keywords

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

⁶ Supporting data are available from ASTM Headquarters. Request RR: D01-1132.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.



This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).