Designation: D 4889 – 04

Standard Test Methods for Polyurethane Raw Materials: Determination of Viscosity of Crude or Modified Isocyanates [1]

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 (A and B) determine the viscosity of crude or modified *iso*cyanates. They are applicable to products derived from toluene di*iso*cyanate, methylene-bis-(4-phenyl*iso*cyanate), and polymethylene polyphenyl*iso*cyanates (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, D 445.

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:* [2]

D 445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and the Calculation of Dynamic Viscosity)

D 883 Terminology Relating to Plastics

E 1 Specification for ASTM Thermometers

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 *iso*cyanates 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 *iso*cyanates 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 *iso* cyanates 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 ± 0.3°C with a Brookfield viscometer, either Model LVF, LVT, RVF, or RVT.[3]

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.

[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.

Current edition approved April 1, 2004. Published April 2004. Originally approved in 1988. Last previous edition approved in 1998 as D 4889 - 98.

[2] 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.

[3] 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*, [3] 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 ± 0.3°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 having a range of 19–27°C and conforming to the requirements for Thermometer 17C in Specification E 1 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 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 *iso*cyanates. 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 *iso*cyanates as well as other materials is published in Test Method D 445.[4]

14. Keywords

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

[4] Supporting data are available from ASTM Headquarters. Request RR: D01-1132.

TABLE 2 Conversion Factors for Various Combinations of Spindles and Rotational Speeds

Spindle Number		Conversion Factors									
LVF Rotational speed, rpm		6	12	30	60						
	1	10	5	2	1						
	2	50	25	10	5						
	3	200	100	40	20						
	4	1000	500	200	100						
RVF Rotational speed, rpm		2	4	10	20						
	1	50	25	10	5						
	2	200	100	40	20						
	3	500	250	100	50						
	4	1000	500	200	100						
	5	2000	1000	400	200						
	6	5000	2500	1000	500						
	7	20 000	10 000	4000	2000						
LVT Rotational speed, rpm		0.3	0.69	1.5	3	6	12	30	60		
	1	200	100	40	20	10	5	2	1		
	2	1000	500	200	100	50	25	10	5		
	3	4000	2000	800	400	200	100	40	20		
	4	20 000	10 000	4000	2000	1000	500	200	100		
RVT Rotational speed, rpm		0.5	1	2	2.5	4	5	10	20	50	100
	1	200	100	50	40	25	20	10	5	2	1
	2	800	400	200	160	100	80	40	20	8	4
	3	2000	1000	500	400	250	200	100	50	20	10
	4	4000	2000	1000	800	500	400	200	100	40	20
	5	8000	4000	2000	1600	1000	800	400	200	80	40
	6	20 000	10 000	5000	4000	2500	2000	1000	500	200	100
	7	80 000	40 000	20 000	1600	10 000	8000	4000	2000	800	400

SUMMARY OF CHANGES

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

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(*1*) Revised 1.1 to reflect industry experience and to provide more information about the standard so that web-searching reviewers will better understand the applicability of standard and what is contained therein.

(*2*) Added statement about SI units in 1.2.

(*3*) Editorially changed word form in 4.2.

(*4*) Added 4.3 for more technical information and counsel for comparison of results.

(*5*) Grammatically corrected 5.1.

(*6*) Added additional information to term Brookfield viscometer in 7.1.

(*7*) Updated Viscometer Model references; converted to SI units; and reorganized speed and spindle information into tables in 9.1.

(*8*) Updated procedure in 10.2 to utilize information recorded in tables which are proposed to be added.

(*9*) Added technical details to assist user in 10.3 and 10.4.

(*10*) Revised calculations to reflect reference to proposed tables; and removed air resistance statement to reflect current practice and instrument designs in Section 11.

(*11*) Revised Table 2 to reflect current manufacturer recommendations.

(*12*) Added Table 1 to reflect current manufacturer data and recommendations.