Designation: D 4878 – 03

Standard Test Methods for Polyurethane Raw Materials: Determination of Viscosity of Polyols [1]

This standard is issued under the fixed designation D 4878; 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 viscosities of polyols in the range from 10 to 100 000 mPa·s(cP) at 25°C or at 50°C. Test Method A also applies to more viscous samples that are soluble in *n*-butyl acetate. Test Method B is simply a reference to a general procedure for kinematic viscosity, D 445. (See Note 1.)

1.2 The values stated in SI units are to be regarded as the standard. Other equivalent units are provided because of current common usage.

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.*

NOTE 1—There is no equivalent ISO standard for Test Method A although ISO 3219 is similar. Test Method B is equivalent to ISO 3104.

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

2.2 *ISO Standards:*[3]

ISO 3104

ISO 3219

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 are suitable for research or as quality control or specification tests.

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

5. Sampling

5.1 Polyesters and polyethers usually contain molecules covering an appreciable range of molecular weights. These have a tendency to fractionate during solidification. Unless the material is a finely ground solid it is necessary to melt (using no higher temperature than necessary) and mix the resin well before removing a sample for analysis. Many polyols are hygroscopic and care should be taken to provide minimum exposure to atmospheric moisture during the sampling.

TEST METHOD A—BROOKFIELD VISCOSITY

6. Summary of Test Method

6.1 The viscosity of resins is measured by determining the torque on a spindle rotating at constant speed in the liquid sample which is adjusted to 25 ± 0.1°C. Samples with viscosities exceeding 100 000 mPa·s(cP) at 50°C are dissolved in *n*-butyl acetate (or other solvent) and the viscosity is determined at 25 ± 0.1°C.

7. Apparatus

7.1 *Constant-Temperature Bath*, capable of maintaining temperatures of 25 ± 0.1°C and 50 ± 0.1°C should be used. Water, water and glycerin, or oil may be used as the heating medium and the bath should be provided with heating, stirring, and thermostating devices.

7.2 *Bath and Sample Thermometers*, graduated in 0.1°C subdivisions and standardized for the range of use to the nearest 0.01°C. ASTM Saybolt Viscosity Thermometers having ranges from 19 to 27°C and 49 to 57°C, as specified, and

[1] These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and is the direct responsibility of Subcommittee D20.22 on Cellular Plastics.

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[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 American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

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

conforming to the requirements for Thermometers 17C and 19C, respectively, as prescribed in Specification E 1 are recommended.

7.3 *Brookfield Synchrolectric Viscometer*[4] —Model LVF with speeds of 60, 30, 12, and 6 r/min is to be used when available. It is applicable to the range of 10 to 100 000 mPa·s(cP). If this model is not available, Model RVF or HAF may be substituted. However, samples should be heated or dissolved in the standard way to keep the measured viscosity below 100 000 mPa·s(cP) so that the test may be repeated in other laboratories under similar conditions with Model LVF. The calibration of the instrument should be checked periodically by measuring the viscosity of Brookfield Engineering Laboratories viscosity standard fluids.[4] Standard fluids L-2, L-3, R-1, R-2, H-1 are suitable for the usual range. The calibration corrections should be applied to sample measurements.

8. Solvent

8.1 *n-Butyl Acetate,* reagent grade.

9. Preparation of Sample

9.1 The preparation of a homogeneous sample is of primary importance in viscosity measurements. A nonuniform temperature distribution as well as the presence of air bubbles and traces of extraneous material should be avoided. Resins are not easily made homogeneous with respect to temperature, therefore, the sample should be thoroughly mixed and the temperature measured at several locations in the sample vessel before determining the viscosity.

10. Preparation of Apparatus

10.1 Attach the viscometer with an adjustable clamp to a ring stand. Adjust the legs at the base of the ring stand until the bubble is in the center of the spirit level on the viscometer. Attach the spindle that applies to the range expected for the sample (see Section 12).

11. Choice of Temperature

11.1 Samples that are liquid and have a viscosity of less than 100 000 mPa·s(cP) at 25°C should be measured at that temperature. Materials that fulfill this requirement only when heated from 25 to 50°C should be measured at 50°C. If the sample viscosity exceeds 100 000 mPa·s(cP) at 50°C, the sample may be dissolved in *n*-butyl acetate (70 or 35 % solids) and the viscosity of the solution measured at 25°C.

12. Choice of Spindle and Rotational Speed

12.1 The recommended Brookfield synchro-lectric viscometer models offer a variety of spindle size and rotational speeds. In the case of non-Newtonian liquids, changing these factors will cause variation in the results obtained. In general, the following recommendations should guide in the choice of spindle size and speed to be used for a specific sample. (See Table 1.)

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	2	1	0.4	0.2
	2	10	5	2	1
	3	40	20	8	4
	4	200	100	40	20
Rotation speed, r/min		2	4	10	20
RVF	1	10	5	2	1
	2	40	20	8	4
	3	100	50	20	10
	4	200	100	40	20
	5	400	200	80	40
	6	1 000	500	200	100
	7	4 000	2 000	800	400
Rotational speed, r/min		1	2	5	10
HAF	1	40	20	8	4
	2	160	80	32	16
	3	400	200	80	40
	4	800	400	160	80
	5	1 600	800	320	160
	6	4 000	2 000	800	400
	7	16 000	8 000	3 200	1 600

12.1.1 The combination chosen should give an instrument reading near the center of the scale (that is, 175 to 325 on the 500 scale).

12.1.2 The lowest possible speed consistent with fulfilling the requirement given in 12.1.1 should be used in order to deemphasize certain types of non-Newtonian behavior.

12.1.3 If these two recommendations conflict, the requirements given in 12.1.1 have preference.

13. Procedure

13.1 Place sufficient sample in a 600-mL low-form beaker to cover the immersion mark on the viscometer spindle. Cover the beaker with a watch glass and immerse to the sample level in the constant temperature bath. Stir occasionally without trapping air bubbles. Check the temperature at several different locations in the beaker to make sure uniformity has been achieved.

13.2 After the desired temperature has been observed throughout the sample for 10 min, immerse the viscometer spindle and guard into a sample to the immersion line marked on the spindle. Exercise caution to avoid air bubbles gathering under the spindle during immersion. If bubbles are observed, detach the spindle, keeping it in the sample, and stir until the bubbles are released. Reinsert the spindle.

13.3 Press down the viscometer clutch lever and start the motor by snapping the toggle switch. Release the clutch lever and allow rotation to continue until the spindle has made eight or ten revolutions. Depress the clutch lever, stop the motor, and read the scale. If, when operating at higher speeds the pointer is not in view when the dial has come to rest, throw the motor switch on and off rapidly until the pointer reaches the window.

13.4 Repeat the procedure until three readings on the 500 scale agree within five units.

[4] Obtainable from Brookfield Engineering Laboratories, 240 Cushing Street, Stoughton, MA 02072.

14. Calculation

14.1 Multiply readings on the 500 scale by the factors given in Table 1 to obtain viscosity in mPa·s(cP). If the instrument scale is 0 to 100, multiply the calculation result by five to obtain viscosity in mPa·s(cP).

14.2 At 60 r/min, air resistance on the pointer has a certain effect. Values obtained should be reduced as follows:

14.2.1 No. 1 spindle, deduct 0.4 mPa·s(cP),

14.2.2 No. 2 spindle, deduct 2.0 mPa·s(cP),

14.2.3 No. 3 spindle, deduct 8.0 mPa·s(cP), and

14.2.4 No. 4 spindle, deduct 40.0 mPa·s(cP).

14.3 Apply all calibration corrections mentioned in 7.3.

15. Report

15.1 Report the following information:

15.1.1 Temperature of test,

15.1.2 Solids content and solvent,

15.1.3 Model of viscometer,

15.1.4 Speed of rotation,

15.1.5 Spindle number, and

15.1.6 Viscosity in millipascal seconds (centipoises).

16. Precision and Bias

16.1 *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 D20.22 (Section D20.22.01), ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

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

TEST METHOD B—CANNON-FENSKE

17. Test Method

17.1 A general method for Cannon-Fenske viscosity which applies to polyols as well as other materials is published in Test Method D 445.[5]

18. Keywords

18.1 Brookfield; Cannon-Fenske; polyols; polyurethane raw materials; viscosity

[5] Supporting data are available from ASTM Headquarters. Request RR: D02-1132.

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 may impact the use of these test methods. This section may also include descriptions of the changes or reasons for the changes, or both.

D 4878 - 03:

(*1*) Added a statement to paragraph 1.2 about common (non-SI) units that are currently used in the industry.

(*2*) Corrected an error in 7.1 (batch to bath).

(*3*) Inserted correct SI based units and made parenthetical the common units.