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Standard Test Methods for Polyurethane Raw Materials: Determination of Viscosity of Polyols¹

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1. Scope^{*}

1.1 These test methods (A and B) determine the ~~viscosities~~ viscosity 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~~ 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 Liquid-in-Glass 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 $25 \pm 0.1^\circ\text{C}$. Samples with viscosities exceeding 100 000 mPa·s(cP) at 50°C are dissolved in *n*-butyl acetate

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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, <http://www.ansi.org>.

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

(or other solvent) and the viscosity is determined at $25 \pm 0.1^\circ\text{C}$.

7. Apparatus

7.1 *Constant-Temperature Bath*, capable of maintaining temperatures of $25 \pm 0.1^\circ\text{C}$ and $50 \pm 0.1^\circ\text{C}$ 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 conforming to the requirements for Thermometers 17C and 19C, respectively, as prescribed in Specification E 1 are recommended.

7.3 *Brookfield Synchroelectric 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.)

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.

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

⁴The sole source of supply of the apparatus known to the committee at this time is Brookfield Engineering Laboratories, 240 Cushing Street, Stoughton, MA 02072. If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend.