Designation: ~~D4878 – 08~~ D4878 – 15

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

This standard is issued under the fixed designation D4878; 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~~ Scope*

1.1 These test methods (A and B) determine the viscosity of polyols in the range from 10 to 100 000 mPa·s(cP) at ~~25°C or at 50°C.~~ 25°C. Test Method A ~~also applies to more viscous samples that are soluble in~~ is a rotational procedure for determining dynamic viscosity. ~~n-butyl acetate.~~ Test Method B is ~~simply a reference to a~~ a general procedure for kinematic ~~viscosity,~~viscosity ~~D445.~~ of transparent polyols. (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.~~is equivalent to ISO 3219. Test Method B is equivalent to ISO 3104.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)

D446 Specifications and Operating Instructions for Glass Capillary Kinematic Viscometers

D883 Terminology Relating to Plastics

~~E1~~E2251 Specification for ~~ASTM Liquid-in-Glass Thermometers~~Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

2.2 *ISO Standards:*[3]

ISO 3104 ~~ISO 3104~~ Petroleum Products—Transparent and Opaque Liquids—Determination of Kinematic Viscosity and Calculation of Dynamic Viscosity

ISO 3219 ~~ISO 3219~~ Plastics—Polymers/Resins in the Liquid State of as Emulsions or Dispersions—Determination of Viscosity Using a Rotational Viscometer with Defined Shear Rate

3. Terminology

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

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~~Polyester and ~~polyethers usually~~polyether polyols contain molecules covering an appreciable range of molecular weights. These ~~have a tendency to~~ can fractionate during solidification. Unless the material is a finely ground solid it is necessary

[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 Materials - Plastics and Elastomers.

Current edition approved ~~Nov. 1, 2008~~May 1, 2015. Published ~~November 2008~~June 2015. Originally approved in 1988. Last previous edition approved in ~~2003~~2008 as ~~D4878 - 03.~~D4878 - 08. DOI: ~~10.1520/D4878-08.~~10.1520/D4878-15.

[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**

to melt (using no higher temperature than necessary) and mix the ~~resin~~polyol 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~~A—ROTATIONAL 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~~at 25 ± 0.1°C. Generation of comparative data using this method requires agreement on the ~~n~~speed, ~~butyl acetate (or other solvent) and the viscosity is determined at 25 ± 0.1°C.~~ spindle, temperature, time of rotation, and torque range of the instrument used.

7. Apparatus

7.1 *Constant-Temperature Bath*, capable of maintaining ~~temperatures~~ a temperature of 25 ± 0.1°C ~~and 50 ± 0.1°C should~~ is to be used. Water, water and glycerin, or oil ~~may be~~ is used as the heating medium and the bath ~~should~~ is to be provided with heating, ~~stirring,~~circulating, and thermostatting 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~~S117C and ~~19C,~~S64C, respectively, as prescribed in Specification ~~E1~~E2251 are recommended. Any other thermometric device of equal or better accuracy is also acceptable.

7.3 ~~*Brookfield Synchrolectric Viscometer*~~*Rotational Viscometer*— ~~—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.~~ Capable of user defined speed and spindle combinations. An instrument that is capable of providing the shear rate is recommended. The calibration of the instrument ~~should~~ is to be checked periodically by measuring the viscosity of ~~Brookfield Engineering Laboratories viscosity~~ NIST traceable standard fluids. ~~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 *Cleaning Solvent*—~~*n-Butyl Acetate,*~~*methanol or acetone,* reagent grade. Any solvent in which the polyol is completely miscible is acceptable.

9. Preparation of Sample

9.1 The preparation of a homogeneous sample is of primary importance in viscosity measurements. A ~~nonuniform~~non-uniform 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~~are to be avoided. The sample must 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~~ Follow the manufacturer's instructions to set up the instrument and ensure that the viscometer is level.~~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~~ are to 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.

11.2 In cases of interlaboratory studies and higher viscosity samples, all parties are to agree upon a set measurement temperature.

12. Choice of Spindle and Rotational Speed

12.1 ~~The recommended Brookfield synchrolectric viscometer models~~ Rotational viscometers 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~~ provide guidance for choosing the 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).~~

12.1.1 The ~~lowest possible speed consistent with fulfilling the requirement given in~~combination chosen shall generate a torque value between 15 and ~~12.1.1 should be used in order to deemphasize certain types of non-Newtonian behavior.~~ 90 % of full scale, or that specified by the instrument manufacturer.

12.1.1.1 If more than one speed/spindle combination will fulfill the requirement of 12.1.1, the combination with the higher speed will provide higher accuracy and the combination with the lower speed will minimize certain types of non-Newtonian behavior.

12.1.1.2 There must be agreement between the testing laboratory and the submitter on the spindle, speed selection.

~~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.~~ Using the smallest container recommended by the manufacturer, place sufficient sample to cover the immersion mark on the viscometer spindle. Cover the ~~beaker with a watch glass~~ container and immerse it to the sample level in ~~the~~a constant temperature bath. Stir occasionally without trapping air bubbles. Check the temperature at several different locations in the ~~beaker~~container to ~~make sure~~ ensure 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~~ (and the guard when recommended by the manufacturer) 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~~Reattach 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.~~ Follow the manufacturer's instructions to measure the viscosity for the sample using a 15-second rotation time.

13.4 ~~Repeat the procedure until three readings on the 500 scale agree within five units.~~ After the analysis, spindles are cleaned with a solvent appropriate for the polyol and equipment used, for example, methanol or acetone.

14. Calculation

14.1 Multiply ~~readings on the 500 scale by the factors given in~~the reading by the factor provided by ~~Table 1 to obtain viscosity in mPa·s(cP). If the instrument scale is 0 to 100, multiply the calculated result by five.~~the manufacturer for the speed/spindle combination used to convert the instrument reading to the viscosity in mPa·s (cP). Most instruments automatically perform this calculation.

~~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,~~

15.1.2 Model of viscometer,

15.1.3 Speed of rotation,

15.1.4 Spindle number, and

15.1.5 Viscosity in millipascal seconds (centipoises) [mPa•s(cP)].

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.