Designation: D4878 – 23

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*

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. Test Method A is a rotational procedure for determining dynamic viscosity. Test Method B is a general procedure for kinematic viscosity 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

NOTE 1—Test Method A is equivalent to ISO 3219. Test Method B is equivalent to ISO 3104.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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

E456 Terminology Relating to Quality and Statistics

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

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

E2935 Practice for Evaluating Equivalence of Two Testing Processes

2.2 *ISO Standards:*[3]

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

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 Terms used in this standard are defined in accordance with Terminology D883, unless otherwise specified. For terms relating to precision and bias and associated issues, the terms used in this standard are defined in accordance with Terminology E456.

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 Polyester and polyether polyols contain molecules covering an appreciable range of molecular weights. These can 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 polyol well before removing a sample for analysis. Many polyols are hygroscopic, therefore exercise care to provide minimum exposure to atmospheric moisture during the sampling.

[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.

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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

TEST METHOD A—ROTATIONAL VISCOSITY

6. Summary of Test Method

6.1 The viscosity is measured by determining the torque on a spindle rotating at constant speed in the liquid sample at 25 ± 0.1°C. Generation of comparative data using this method requires agreement on the speed, spindle, temperature, time of rotation, and torque range of the instrument used.

7. Apparatus

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

7.3 *Rotational Viscometer*—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 is to be checked periodically by measuring the viscosity of NIST traceable standard fluids.

8. Solvent

8.1 *Cleaning Solvent—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 non-uniform temperature distribution as well as the presence of air bubbles and traces of extraneous material 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 Follow the manufacturer's instructions to set up the instrument and ensure that the viscometer is level.

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 are to be 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 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 provide guidance for choosing the spindle size and speed to be used for a specific sample.

12.1.1 The combination chosen shall generate a torque value between 15 and 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.

13. Procedure

13.1 Using the smallest container recommended by the manufacturer, place sufficient sample to cover the immersion mark on the viscometer spindle. Cover the container and immerse it to the sample level in a constant temperature bath. Stir occasionally without trapping air bubbles. Check the temperature at several different locations in the container to 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 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. Reattach the spindle.

13.3 Follow the manufacturer's instructions to measure the viscosity for the sample using a 15-second rotation time.

13.4 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 the reading by the factor provided by 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.

15. Report

15.1 Report the following information:

15.1.1 Temperature of test,

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