Designation: 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*

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 and health 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.

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

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 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 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 and care should be taken to provide minimum exposure to atmospheric moisture during the sampling.

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

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[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**

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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 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—KINEMATIC VISCOSITY

17. Summary of Test Method

17.1 The viscosity of polyols is measured by determining the time it takes a fixed volume of liquid to flow under gravity control through a calibrated capillary glass viscometer under a reproducible driving head at 25 ± 0.1°C. The kinematic viscosity is determined by multiplying the flow time by the calibration factor of the viscometer.

18. Apparatus

18.1 Additional details of equipment can be found in Specifications D446.

18.2 *Constant-Temperature Bath,* capable of maintaining a temperature of 25 ± 0.1°C and 50 ± 0.1°C is to be used. Water, water and glycerin, oil, or any other transparent thermal transfer liquid is to be used as the heating medium and the bath is to be of sufficient depth and to be provided with heating, circulating, and thermostatting devices.

18.3 *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.

18.4 *Glass capillary viscometer,* calibrated by the manufacturer.

18.5 *Timing device,* capable of reading to the nearest 0.1 s or better, with an accuracy of ±0.07 % of the reading when tested over the intervals of 200 and 900 s.

19. Solvents

19.1 *Methanol or Acetone,* reagent grade. Any solvent in which the polyol is completely miscible is acceptable.

20. Preparation of Sample

20.1 The preparation of a homogeneous sample is of primary importance in viscosity measurements. The presence of air bubbles and traces of extraneous material are to be avoided. The sample is to be thoroughly mixed and the bubbles removed by heating and/or use of a sonication bath.

21. Preparation of Apparatus

21.1 The constant temperature bath is to be heated/cooled to the desired temperature and maintained prior to sample analysis.

21.2 The viscometer tube is to be clean and dry. The tube is to be suspended in the temperature bath using an appropriate tube holder such that the measurement area of the tube is at least 20 mm below the bath fluid surface, and the bottom of the tube is at least 20 mm from the bottom of the bath.

22. Choice of Temperature

22.1 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.

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

23. Choice of Viscometer Type and Size

23.1 The type of viscometer to be used depends on the transparency and the viscosity range of the material to be analyzed. Test Method D445 lists the types of viscometers and their typical ranges. For transparent liquid polyols, Ostwald types are typically used.

23.2 The size of viscometer to use depends again on the viscosity range of the material to be analyzed. Specifications D446 lists specific viscometer types and the sizes that are available. It is recommended that the size selected allow the sample to flow from the first timing mark to the final timing mark in a minimum of 200 s.

23.3 In cases of dispute between parties regarding kinematic viscosity measurements, all parties are to agree on the same type and size of viscometer to be used in any interlaboratory studies.

24. Procedure

24.1 The constant temperature bath is to be heated/cooled to the desired temperature and maintained prior to sample analysis.

24.2 Select a clean, dry viscometer covering the appropriate range for the sample. Suspend the viscometer in the temperature bath such that it hangs vertically in all directions (plumb).

24.3 Charge the viscometer with the proper amount of sample according to the design of the tube (Refer to documentation accompanying the viscometer and/or Specifications D446).

24.4 The viscometer containing the sample is to remain in the bath long enough for the sample to reach the bath temperature. This is typically done by drawing sample up into the capillary side of the tube via suction bulb and allowing the sample to flow back down completely via gravity flow. After several repetitions, the sample should be at equilibrium with the bath temperature. As different viscometer types and sizes will require varying equilibration times, determine a safe time by trial.

24.5 Using a bulb or other suction device, apply a vacuum to the capillary arm of the viscometer, drawing the sample head to a point a few mm above the first (top) timing mark. Remove the vacuum and start the timing device as the meniscus passes the first timing mark. Allow the liquid sample to flow freely via gravity, stopping the timing device as the meniscus passes the second timing mark. Record this time in seconds to the neares 0.1 s.

24.6 Repeat 24.5 to obtain a second measurement. If the duplicates agree within the repeatability listed in 27.1, report the average of the duplicates. If the duplicates do not agree, repeat 24.5 until two determinations agree within that specified in 27.1. The greatest source of variability is typically due to not allowing enough time for the sample temperature to come to equilibrium with the bath temperature (see 24.4).

24.7 Clean the viscometer after sample analysis by removing the viscometer from the bath and emptying its contents. Rinse several times with the cleaning solvent. Allow the tube to dry completely by storing in a warm oven, or by passing dry air slowly through the tube for two minutes.

25. Calculations

25.1 Calculate the kinematic viscosity, v, by using this equation:

$$v = C \times t \quad (1)$$

where:

v = kinematic viscosity, mm^2/s,
C = calibration constant of the viscometer, (mm^2/s)/s, and
t = mean flow time, s.

25.2 Calculate the dynamic viscosity, η, using the kinematic viscosity and the density, ρ, of the sample:

$$\eta = v \times \rho \quad (2)$$

where:

η = dynamic viscosity, mPa·s(cP),
ρ = density, g/cm^3, at the same temperature that the kinematic viscosity was determined, and
v = kinematic viscosity, mm^2/s.

26. Report

26.1 Report the following information:

26.1.1 Product identification,

26.1.2 Viscometer type and size used,

26.1.3 Measurement temperature, and

26.1.4 Result of the test.

27. Precision and Bias

27.1 *Precision*—Attempts to develop a full precision and bias statement for this test method have not been successful. For this reason, data on precision and bias cannot be given. Because this test method does not contain a round-robin-based 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 D20.00 (Section 20.00.00), ASTM International, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

27.2 Test Method D445 is similar to this standard and does include precision data. Precision statements from Test Method D445 for base oils are restated below as an estimate of the precision for this test method. An interlaboratory study (ILS) is expected to be performed on this method, specifically using base polyols, prior to the next five-year review.

27.2.1 *Repeatability*—Two results obtained within one laboratory shall be judged not equivalent if they differ by more than 0.11% (relative).

27.2.2 *Reproducibility*—Two test results obtained by different laboratories shall be judged not equivalent if they differ by more than 0.65 % (relative).

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

28. Keywords

28.1 dynamic viscosity; kinematic viscosity; polyols; polyurethane raw materials; rotational viscosity

SUMMARY OF CHANGES

Committee D20 has identified the location of selected changes to this standard since the last issue (D4878 – 08) that may impact the use of this standard. (May 1, 2015)

(1) Made various edits throughout to exclude the use of a specific instrument manufacturer's equipment and create a more general method for rotational viscosity of polyols.
(2) Made various edits throughout to exclude use of nonmandatory language.
(3) Updated ISO equivalency statement in Note 1.
(4) Added to 2.1, reference to D446 and changed reference from E1 to E2251 to allow for use of non-mercury thermometers.
(5) Updated ISO standard titles in 2.2 to current versions.
(6) Changed Section 8 to include more commonly used solvents.
(7) Replaced Method B, which contained only a reference to a general method for kinematic viscosity of liquids (D445), with an expanded method for kinematic viscosity of polyols.

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