Designation: D4889 – 21

Standard Test Methods for Polyurethane Raw Materials: Determination of Viscosity of Crude or Modified Isocyanates [1]

This standard is issued under the fixed designation D4889; 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 crude or modified *iso*cyanates. They are applicable to products derived from toluene di*iso*cyanate, methylene di(phenylisocyanate), and polymeric (methylene phenylisocyanate) (see Note 1).

NOTE 1—Test method A includes a procedure for measuring dynamic viscosity using a rotational instrument. Test method B is a general procedure for kinematic viscosity of isocyanate.

1.2 The values stated in SI units are to be regarded as standard.

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.* For specific hazards statement, see **Warning** at the end of 5.1.

NOTE 2—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

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

E2935 Practice for Conducting Equivalence Tests for Comparing 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 or 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 can be used for research, quality control, or specification tests to characterize *iso*cyanates used in polyurethane products.

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

4.3 Some isocyanates exhibit non-Newtonian behavior under certain conditions. Whenever possible, generate results for comparison under the same conditions, that is, the same spindle/speed combination for rotational viscosity and the same tube size for kinematic viscosity.

5. Sampling

5.1 Since organic *iso*cyanates react with atmospheric moisture, take special precautions in sampling. Usual sampling

[1] These test methods are under the jurisdiction of ASTM Committee D20 on Plastics and are 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

methods, even when conducted rapidly, can cause contamination of the sample with insoluble urea. Therefore, blanket the sample with dry air or nitrogen at all times. (**Warning—** Di*iso*cyanates are eye, skin and respiratory irritants at concentrations above the occupational exposure limit (TLV or PEL). Di*iso*cyanates can cause skin and respiratory sensitization (asthma) in some people. Once sensitized, it is essential to limit further exposure to diisocyanates. Use a combination of engineering controls and personal protective equipment, including respiratory, skin and eye protection, to prevent overexposure to di*iso*cyanates. Consult the product suppliers' Safety Data Sheet (SDS) for more detailed information about potential health effects and other specific safety and handling instructions for the product.)

6. Test Conditions

6.1 Since isocyanates react with moisture, keep laboratory humidity low, preferably about 50 % relative humidity. See Warning in 5.1.

TEST METHOD A—ROTATIONAL VISCOSITY

7. Summary of Test Method

7.1 The viscosity 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. Generation of comparative data using this method requires agreement on the speed, spindle, temperature, time of rotation and torque range of the instrument used.

8. Apparatus

8.1 *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.

8.2 *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.

8.3 *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.

9. Solvent

9.1 *Cleaning Solvent—dichloromethane or acetone*, reagent grade. Any solvent in which the isocyanate is completely miscible is acceptable.

10. Preparation of Sample

10.1 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.

11. Preparation of Apparatus

11.1 Follow the manufacturer's instructions to set up the instrument and ensure that the viscometer is level.

12. Choice of Temperature

12.1 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.

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

13. Choice of Spindle and Rotational Speed

13.1 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.

13.1.1 The combination chosen shall generate a torque value between 15 and 90% of full scale, or that specified by the instrument manufacturer.

13.1.1.1 If more than one speed/spindle combination will fulfill the requirement of 13.1.1, 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.

13.1.1.2 There must be agreement between the testing laboratory and the submitter on the spindle/speed selection.

14. Procedure

14.1 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 make sure uniformity has been achieved.

14.2 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.

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

14.4 After the analysis, spindles are cleaned with a solvent appropriate for the isocyanate and equipment used, for example, dichloromethane or acetone.

15. Calculation

15.1 Multiply the reading by the factor provided by the manufacture for the speed/spindle combination used to convert the instrument reading to the viscosity in mPa.s (cP). Most instruments automatically perform this calculation.

16. Report

16.1 Report the following information:

16.1.1 Temperature of test,

16.1.2 Model of viscometer,

16.1.3 Speed of rotation,

16.1.4 Spindle number, and

16.1.5 Viscosity in millipascal seconds (centipoises) [mPa·s(cP)].

17. Precision and Bias

17.1 *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 D 20.22 (Section D 20.22.01), ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

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

TEST METHOD B—KINEMATIC VISCOSITY

18. General

18.1 The viscosity of isocyanates 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.

19. Apparatus

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

19.2 *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.

19.3 *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.

19.4 *Glass Capillary Viscometer,* calibrated by the manufacturer.

19.5 *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.

20. Solvent

20.1 *Cleaning Solvent—dichloromethane or acetone*, reagent grade. Any solvent in which the isocyanate is completely miscible is acceptable.

21. Preparation of Sample

21.1 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.

22. Preparation of Apparatus

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

22.2 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.

23. Choice of Temperature

23.1 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.

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

24. Choice of Viscometer Type and Size

24.1 The type of viscometer to be used depends on the transparency and the viscosity range of the material to be analyzed. Test Method E2935 lists the types of viscometers and their typical ranges. For transparent liquid isocyanates, Ostwald types are typically used.

24.2 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.

24.3 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.

25. Procedure

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