Designation: D7867 – 13

Standard Test Methods for Measurement of the Rotational Viscosity of Paints, Inks and Related Liquid Materials as a Function of Temperature[1]

This standard is issued under the fixed designation D7867; 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 cover the use of rotational viscometers to determine the dependence of apparent viscosity of paints, inks and related liquid materials on temperature. The first method uses a standard rotational viscometer with concentric cylinder geometry running at a fixed rotational speed as the temperature is increased or decreased. The second method uses a rotational viscometer with cone and plate geometry running at a fixed rotational speed as the temperature is increased or decreased. The third method uses concentric cylinder or cone/plate geometry operated with a shear rate ramp at several discrete temperatures.

1.2 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 This standard may involve hazardous materials, operations and equipment. *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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings

3. Terminology

3.1 *Definitions:*

3.1.1 *apparent viscosity, n*—viscosity that is not a true property of the fluid, but a variable depending on the shear rate.

3.1.2 *shear thinning fluid*—fluid in which the apparent viscosity decreases with increasing shear rate.

3.1.3 *thixotropic fluid, n*—fluid whose viscosity is time dependent, that is, its viscosity decreases with the time it is subjected to shear.

3.1.4 *viscosity, n*—the ratio between an applied shear stress to the resulting shear rate (velocity gradient) is defined as the dynamic viscosity; it is a measure of the resistance to flow of a fluid.

4. Summary of Test Methods

4.1 *Test Method A* is run with a viscometer with concentric (coaxial) cylinder capability, either built-in or as an accessory that allows use of small specimen size (2 to 20 mL). This test is used for measuring apparent viscosity as a function of temperature at low to medium shear rates. The viscosity-temperature profile of the paint, ink or other material may be determined at a fixed shear rate or the viscosity-shear rate profile may be determined at several discrete temperatures.

4.2 *Test Method B* is accomplished with a viscometer that has cone/plate test geometry. It is used to measure apparent viscosity as a function of temperature at low to medium shear rates (high shear rates may cause shear heating that interferes with temperature control). Shear heating is more likely with cone/plate geometry because it permits use of a wider range of shear rates than does the concentric cylinder instrumentation in Method A. The upper limit of shear rate will depend on the material and its viscosity. The viscosity-temperature profile of the paint/coating may be determined at a fixed shear rate or the viscosity-shear rate profile may be determined at several discrete temperatures. The smaller specimen size compared to Method A permits better temperature control and more rapid characterization of the viscosity-temperature profile.

4.3 *Test Method C* is used for determining apparent viscosity as a function of a shear rate ramp at multiple discreet temperatures. This method can be used with either coaxial cylinder geometry or cone/plate geometry. A shear rate ramp is defined and run at a discreet temperature; the viscosity values at each shear rate are recorded. The shear rate ramp is then repeated at a series of discreet temperatures and the viscosity values are measured to characterize the viscosity-temperature profile for the sample specimen.

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[1] These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints & Paint Materials.

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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.

4.4 Temperature control for Test Methods A, B, and C requires use of an apparatus that maintains test sample temperature within ±0.2°C of the specified set point. Control to within ±0.1°C is preferred if achievable.

5. Significance and Use

5.1 The viscosity of paint, inks and many related liquid materials is dependent on temperature. It is useful to know the extent of this dependence. One use of such information is to prepare a viscosity-temperature table or curve. Then, if ambient conditions do not allow the measurement of viscosity at the exact temperature stated in a specification or regulation, the viscosity measured at ambient temperature can be used to determine the viscosity at the temperature of interest through the use of the previously prepared table or curve. Viscosity measurements that cover a range of shear rates as well as temperatures could include shear rates associated with paint application or allow extrapolation to such shear rates. This information would enable a producer or user to estimate the effect on application of heating the paint.

6. Apparatus

6.1 *Multi-speed Rotational Viscometer,* either with coaxial (concentric) cylinder geometry (either built-in or as an attachment) or cone/plate geometry.

7. Sampling

7.1 Take a representative sample of the product to be tested in accordance with Practice D3925. If the sample has a tendency to settle or separate on standing, it must be stirred or shaken until homogeneous before a test specimen is taken from it. The specimen must be free of any foreign matter or air bubbles.

TEST METHOD A—CONCENTRIC CYLINDER

8. Procedure

8.1 Make all measurements at agreed upon temperatures or temperature increments.

8.2 Verify calibration of the viscometer according to the manufacturer's instructions.

8.3 Select the correct inner cylinder.

8.4 Place the proper amount of material in the cup of the instrument. Make sure to introduce the sample in a consistent way (that is, via a syringe or by pouring).

8.5 Allow the specimen and inner cylinder to equilibrate to the first temperature. Verify the temperature at the instrument.

8.6 Select proper rpm and allow the rotating cylinder to rotate for an agreed upon time interval and taking a reading or wait until the reading stabilizes and note that value.

8.7 Record viscosity reading, temperature and rotational speed. Record shear rate if measurable and needed.

NOTE 1—Many paints and inks are shear thinning and thixotropic. They have structure that is broken down by shearing. By allowing the same time interval between readings for measurements on given product, viscosity differences due to differences in the degree of break-down of structure should be minimized.

8.8 Turn off motor after data point is collected. Increase temperature to next level. Allow the specimen and inner cylinder to come to equilibrium. The time for thermal equilibration will vary with the instrument and the size and mass of the cylinders.

8.9 Repeat steps 8.4 – 8.8 as needed until the desired temperature range had been covered.

NOTE 2—In the case of highly structured materials or those that dry rapidly, it may be necessary to change the specimen between each temperature change.

9. Report

9.1 Report the following information:

9.1.1 Reference to this test method, the viscometer model and specific concentric cylinder geometry used.

9.1.2 The viscosity at each rotational speed/temperature.

9.1.3 The time interval between measurements.

TEST METHOD B—CONE/PLATE VISCOMETER

10. Procedure

10.1 Make all measurements as agreed upon temperatures or temperature increments.

10.2 Select the cone needed for the shear rate or rates of interest.

10.3 Zero the viscometer and verify calibration according to the manufacturer's instructions.

10.4 Make sure that the gap between the cone and the plate is set properly.

NOTE 3—If temperature is changed in increments of 10°C or more, then the gap setting should be rechecked.

10.5 Place the correct amount of material on the plate. Make sure to introduce the sample in a consistent way (that is, via a syringe or by pouring).

10.6 Allow the specimen and cone to equilibrate to the first temperature. Verify temperature at the instrument.

10.7 Select proper rpm and allow cone to rotate for an agreed upon time before taking a reading or wait until reading stabilizes.

NOTE 4—See Note 1.

10.8 Record viscosity reading, temperature and rotational speed. Record shear rate if applicable.

10.9 After taking a data point, turn off motor. Increase temperature to next level. Allow proper amount of time for specimen and cone to come to equilibrium.

10.10 Repeat steps 10.5 – 10.9 as needed until the desired temperature range has been covered.

NOTE 5—Cone/plate specimens have a tendency to dry out around the edges, particularly if they contain volatile solvents. Heating makes this worse. Therefore, it may be necessary to change the specimen between each temperature change. If temperature is changed in increments of 10°C or more, then the gap setting should be rechecked.

11. Report

11.1 Report the following results:

11.1.1 Reference to this test method and the viscometer model and the cone used.

11.1.2 The viscosity at each rotational speed/temperature.

11.1.3 The time interval between measurements.

TEST METHOD C—SHEAR RATE RAMP AT DISCRETE TEMPERATURES

12. Procedure

12.1 Make all measurements at agreed upon shear rate ramp and discreet temperatures.

12.2 Select the system (coaxial cylinder or cone/plate) needed for the shear rate ramp of interest.

12.3 Zero the viscometer and verify calibration according to the manufacturer's instructions.

12.4 If using the cone/plate system, make sure that the gap between the cone and the plate is set properly.

NOTE 6—If temperature is increased by a total change of 10°C or more, the gap between the cone and plate should be reset.

12.5 Place the correct amount of sample material into the chamber if using coaxial cylinder geometry. If using cone/plate geometry, place the proper amount of material on the plate. Make sure to introduce the sample in a consistent way (that is, via a syringe or by pouring).

12.6 Allow the specimen and spindle to equilibrate to the test temperature.

12.7 Select the first rotational speed and allow the spindle to rotate for an agreed upon time before taking a viscosity reading or wait until the viscosity reading stabilizes.

NOTE 7—Many paints and inks are shear thinning and thixotropic. They have structure that is broken down by the shearing action. Allowing the same time interval between viscosity readings at different shear rates may provide better repeatability of test data.

12.8 Record viscosity readings at each rotational speed in the shear rate ramp.

12.9 After taking all data points in the shear rate ramp, turn off motor. Increase temperature to next level. Allow proper amount of time for specimen to come to equilibrium.

12.10 Repeat steps 12.5 – 12.9 as needed until the desired temperature range has been covered and each temperature has been tested at the shear rate ramp.

NOTE 8—Cone/plate specimens have a tendency to dry out around the edges, particularly if they contain volatile solvents. Heating makes this worse. Therefore, it may be necessary to change the specimen between each temperature change. If temperature is changed in increments of 10°C or more, then the gap setting should be rechecked.

NOTE 9—If concentric cylinder geometry is being used and if the material is highly structured or may dry rapidly, it may be necessary to change the specimen between each temperature change.

13. Report

13.1 Report the following results:

13.1.1 The viscometer model and geometry used.

13.1.2 The viscosity at each rotational speed/temperature.

13.1.3 The time interval between measurements.

14. Precision and Bias

14.1 *Precision*—No precision statement has yet been developed.

14.2 *Bias*—Since there is no accepted reference material suitable for determining bias for the procedures in this standard, bias has not and will not be determined.

15. Keywords

15.1 coaxial cylinder viscometer; cone/plate viscometer; rotational viscosity; viscosity; viscosity as a function of temperature