Designation: D7395 – 07 (Reapproved 2012)

Standard Test Method for Cone/Plate Viscosity at a 500 s^{-1} Shear Rate[1]

This standard is issued under the fixed designation D7395; 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 This test method covers the determination of the viscosity of paints and related products using a cone/plate viscometer at a shear rate of 500 s^{-1}.

1.2 Viscosity values obtained by this method may be used as an alternative to results from No. 4 Ford cup measurements. The values from this method do not replicate Ford cup results, but can be used for quality control, producer-user specifications and viscosity reduction in the same manner that Ford cups are used.

1.3 If viscosity at a higher shear rate is needed, Test Method D4287, which describes viscosity measurement at 10 000 to 12 000 s^{-1} may be used.

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

1.5 *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

D4287 Test Method for High-Shear Viscosity Using a Cone/Plate Viscometer

3. Summary of Test Method

3.1 The material to be tested is placed between the cone and plate of a cone/plate viscometer set at 25ºC, a solvent trap is lowered, and the specimen is subjected to a shear rate of 500 s^{-1} while the viscosity is measured.

4. Significance and Use

4.1 This test has been used to measure viscosities of automotive topcoats in place of No. 4 Ford cup measurements and has provided an equivalent ability to evaluate whether the paint meets specifications and is sprayable.

4.2 The method could be considered as an alternative to No. 4 Ford cup and other efflux cup measurements for other products as well.

4.3 The type of viscometer described in this method gives better temperature control than Ford and other efflux cups and is expected to give better precision.

4.4 This test method is suitable for all paints and related materials whether they are Newtonian in behavior or not.

5. Apparatus

5.1 *Cone/Plate Type Viscometer* with cone/speed combination producing a shear rate of 500 s^{-1} and fitted with a solvent trap that covers the cone. For automotive topcoats, the viscometer must provide a viscosity measurement range that includes 20 to 200 cP (= mPa.s) at the above-mentioned shear rate. A Brookfield CAP1000+ or CAP2000+ with a #10 cone at 100 rpm shaft speed has worked well.

6. Reagents and Materials

6.1 *Water or Solvent*—If the viscometer allows manual zeroing, this should be done in accordance with the manufacturer's directions. Zeroing procedures that require liquids may be satisfied with water or a low-viscosity solvent such as xylene or mineral spirits.

6.2 *Mineral Oils*—One to three standard mineral oils with known viscosities (certified by an approved laboratory) lying between 10 and 90 % full scale to be used for calibrating the instrument. If a single oil is used, its viscosity should be similar to that of the materials being tested.

NOTE 1—Silicone oils should be avoided because of their tendency to contaminate instruments, containers and other equipment.

7. Sampling

7.1 Take a representative sample of the product to be tested in accordance with Practice D3925. If the sample has a

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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.

tendency to settle or separate on standing, then it must be stirred or shaken until homogeneous before a test specimen is taken from it. However, it is appropriate to directly test a sample that has been under constant circulation and shear in a paint system. The specimen must be free of any foreign matter or air bubbles, and its volume must be sufficient to cover the portion of the viscometer plate under the cone when the latter is brought into contact with the plate.

8. Preparation of Apparatus

8.1 The viscometer must be zeroed on a daily basis or otherwise before use, in accordance with the operating manual for the instrument. It may need to be re-zeroed at other times in accordance with the manufacturer's instructions.

8.2 Verify the calibration of the apparatus by following the procedure in Section 9, but using a standard refined mineral oil having Newtonian characteristics and a known viscosity. If the viscometer reads the correct viscosity (or within 5 % of that value) with an oil whose viscosity is similar to that of the specimens to be tested, then the viscometer readings may be used as is. If the viscometer does not give the correct viscosity for the oil, then the instrument should be returned to the manufacturer for adjustment. If necessary, a calibration curve may be constructed by taking viscometer readings for three oils and plotting measured viscosity versus specified (true) viscosity for the oils. Subsequent measurements are corrected to true viscosities through use of the curve.

8.3 Check the cones periodically for wear. Replace any cone that shows obvious wear. If there is any question of as to whether the cone has changed, verify the apparatus as in 8.2.

8.4 The viscosity measurement must be done at a closely controlled temperature of 25 ± 0.1ºC.

8.5 Make sure that the combination of cone and speed that is selected for the measurements gives the shear rate of 500 s^{-1}.

9. Procedure

9.1 Check that the cone is attached properly and the set screw hand tightened to snug. Do not over tighten. With the cone in the down position (in contact with the plate), turn the instrument on and allow it to warm up for at least 10 min. If the viscometer is a multi-temperature instrument, be sure that it is set at 25.0ºC. Confirm that readings will be in cP (mPa.s).

9.2 Raise the cone to the up position. Transfer a suitable amount of the product to be tested to the center or near the center of the plate with a pipette, syringe or other precision transfer device. If the viscometer manufacturer specifies a target specimen volume, transfer this amount (±0.005 ml).

NOTE 2—Dispensing of 0.190 ± 0.005 ml with a precision pipette has been found to work for a Brookfield CAP1000+/2000+ using a #10 cone, and takes hold-up in the pipette tip into account. The actual target amount for a #10 cone is 0.170 ml.

Take care to avoid the inclusion of air bubbles. Lower the cone to the down position quickly to minimize solvent loss and confirm that the specimen is pressed out slightly (at least 1 mm) around the entire circumference of the cone. Lower the solvent trap over the cone and onto the plate.

NOTE 3—If specimen material is not observed around the entire circumference of the cone, Stop! Clean up, resample and deposit a new specimen onto the plate to avoid producing a low viscosity value. Verify that the entire specimen is being dispensed from the pipette, syringe or other transfer device.

9.3 Wait for 60 s to allow the specimen to reach 25.0ºC and equilibrate at that temperature. If the instrument has the capability of setting times for specific procedures, set the HOLD time for 60 s or make certain that the specimen has equilibrated at 25.0°C. Set a hold time of at least 20 s. Whichever HOLD time approach is used, make certain that the specimen actually is tested (RUN) at 25.0°C. Set the RUN time for 15 s (at 100 rpm; slower rotation speeds may require longer run times, in accordance with manufacturer's recommendation).

9.4 Start the cone rotating (make sure that the solvent trap is not touching the cone shaft) and record the reading after 15 s.

9.5 Raise the solvent trap, followed by the cone.

9.6 Clean both the cone (in-place) and the plate carefully, using a clean, lint-free soft cloth or tissue and a suitable solvent. Take care to remove all the test material and cleaning solvent, but minimize sideways pressure on the cone shaft to avoid bending it. Do not use cleaning utensils that may damage the apparatus. *Metal cleaning tools must never be used.*

10. Report

10.1 Report the following information:

10.1.1 Reference to this test method and the viscometer used.

10.1.2 Identification of the product under test.

10.1.3 Cone used.

10.1.4 Viscometer (rpm) speed used.

10.1.5 Test result in cP (= mPa.s) to the nearest 0.1 cP.

11. Precision and Bias

11.1 *Precision*—No ASTM interlaboratory testing has yet been carried out on this method, but estimates of precision have been made based on experience with the method.

11.1.1 *Repeatability*—Two test results in the range 20 to 200 cP obtained by the same operator should be considered suspect if they differ by more than 3 % relative.

11.1.2 *Reproducibility*—Two results in the range 20 to 200 cP obtained by operators in different laboratories should be considered suspect if they differ by more than 10 % relative.

11.2 *Bias*—Since there is no accepted reference material suitable for determining the bias for the procedure in this test method, bias has not been determined.

12. Keywords

12.1 No. 4 Ford cup-alternative to; rheology-paints/related coatings/materials; viscometer-cone/plate; viscosity-paints/related coatings/materials