Designation: D 4287 – ~~94~~00

Standard Test Method for High-Shear Viscosity Using ~~the ICI~~ a Cone/Plate Viscometer[1]

This standard is issued under the fixed designation D 4287; 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, varnishes, and related products at a rate of shear of 12 000 s^{-1}.

1.2 Paints and varnishes that dry very rapidly may not give reproducible results with this test method. Measurements made at elevated temperatures may also give poor precision due to loss of volatiles and to drying.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*

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[1] This test method is under the jurisdiction of ASTM Committee ~~D-1~~ D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints and Paint Materials.

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D 1210 Test Method for Fineness of Dispersion of Pigment-Vehicle Systems by Hegman-Type Gage[2]
D 3925 Practice for Sampling Liquid Paints and Related Pigmented Coatings[2]
D 4958 Test Method for Comparison of the Brush Drag of Latex Paints[3]

3. Summary of Test Method

3.1 The material to be tested is placed between the cone and plate of a cone/plate viscometer, then subjected to a high shear rate while the viscosity is determined.

4. Significance and Use

4.1 The viscosity value obtained by this test method gives information about the flow properties of the material under high-shear conditions similar to those encountered during application: brushing (see Test Method D 4958), spraying, electrostatic disk, or roll coating.

4.2 This test method is suitable for all paints and varnishes whether they are Newtonian in behavior or not. However, due to the narrow gap between the stationary and rotary parts of high-shear viscometers, this test method is more reproducible for paints having finer pigment dispersions as determined by Test Method D 1210.

5. Apparatus

5.1 *ICI Cone/Plate Type Viscometer,* (see Fig. 1) or other cone/plate viscometer withwith cone/speed combination producing a

[2] *Annual Book of ASTM Standards*, Vol 06.01.
[3] *Annual Book of ASTM Standards*, Vol 06.02.

FIG. 1 Analog ICI Cone and Plate Viscometer

rate of shear of 12 000 s^{-1}. The ~~ICI cone/plate~~ viscometer ~~gives this shear rate with~~ must provide a viscosity measurement rage of either 0 to ~~10 P (0 to 1 Pa·s) and~~ 10 (P) or 0 to 5~~.~~ (P ~~c~~) at the above mentioned shear rate. With higher viscosity materials, other cones and speeds may be used upon agreement between the producer and the user, but it should be noted that these may give lower shear rates not truly representative of application conditions. ~~Other cone/plate viscometers may be used on agreement between the producer~~ Refer to Fig. 1 and ~~the user as long as the same rate~~ Fig. 2 of ~~shear is measured and it is approximately 12 000 s^{-1}. However, results may differ from those produced with~~ an ~~ICI cone/plate~~ analog and digital cone and plate viscometer.

NOTE 1—The SI units for viscosity are pascal-seconds (Pa·s = 10 P, 1 mPa·s = 1 cP).

6. Reagents and Materials

6.1 *Water or Solvent*—~~W~~The viscometer should be zeroed according to the manufacturer's specification. Zeroing procedures that require liquid may be satisfied with water or a low viscosity solvent such as xylene or mineral ~~spirits to be used for zeroing the instrument.~~ spirits.

6.2 *Mineral Oils*—Three standard mineral oils with known viscosities (certified by an approved laboratory) lying between 10 and 90 % of full scale to be used for calibrating the instrument.[4]

NOTE 2—Silicone oils should be avoided because of their tendency to contaminate instruments, containers and other equipment and because of the possibility of shear thinning behavior at high shear rates.

7. Sampling

7.1 Take a representative sample of the product to be tested in accordance with Practice D 3925. 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 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 Zero the apparatus~~

8.1 The viscometer should be zeroed on a daily basis when in regular use~~,~~ or otherwise before use, ~~by following~~ according to the ~~procedure in Section 9, but using a low viscosity liquid.~~ viscometer operating manual. With the ~~ICI~~ analog-type viscometer,

~~[4] ICI cone/plate viscometers~~

[4] Such oils are ~~manufactured by Research Equipment (London) Ltd., 72 Wellington Rd., Twickenham, Middlesex TW12 5NX, England and are~~ available ~~in North America~~ from ~~BYK-Gardner, Inc. 2435 Linden Lane, Silver Spring, MD 20910.~~ The Cannon Instrument Co., P.O. Box 16, State College, PA 16801.

FIG. 2 Digital Cone and Plate Viscometer

if the pointer does not indicate zero, it may be adjusted by means of a lever on the left-hand side of the upper part of the instrument housing. If the instrument cannot be zeroed, ~~it~~ adjustment should ~~be returned~~ take place according to the ~~supplier for adjustment.~~ manufacturer's suggestions.

8.2 Verify the calibration of the apparatus by following the procedure in Section 9, but using standard refined mineral oils having Newtonian characteristics and known viscosities. If the viscometer reads the correct viscosity (or within 5 % of that value) with two or more oils whose viscosities bracket those of specimens to be tested, then the viscometer readings may be used as is. If the viscometer readings do not give the correct viscosities for the oils, then a calibration curve must be constructed by taking viscometer readings for three oils and plotting measured viscosity versus specified (correct) 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 a definite flattening of the apex. Some users have found it necessary to replace cones every year. Others have had to do so more often when abrasive paints or pastes were being tested.

8.4 The determination must be made at a closely controlled temperature of 25 ± 0.3°C, unless otherwise agreed. In order to check the temperature control, carry out the test as outlined in 9.1 with the standard refined mineral oil of the highest viscosity. Allow the viscometer to run with this oil for 5 min and determine whether the reading decreases. If the decrease is more than 10 %, the apparatus is unsuitable for the determination of viscosities at high rates of shear in accordance with this test method.

NOTE 3—Many multitemperature cone/plate viscometers have heating, but not cooling, capabilities. Therefore, runs with these viscometers at 25°C should only be done at room temperatures at or below 22°C to ensure that the plate temperature does not go above 25°C. ~~The instruments that operate at 25°C only do have cooling capabilities and give better temperature control.~~ .

9. Procedure

9.1 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 5 min. For a multitemperature instrument, set at 25°C or to an alternatively agreed upon temperature. Raise the cone to the up position. Transfer a suitable amount of the product to be tested to the plate, taking care to avoid the inclusion of air bubbles, and again lower the cone to the down position. Wait for 30 s to allow the specimen to attain the agreed upon temperature.

9.2 Start the cone rotating and record the reading ~~o~~. When using an analog viscometer, record the ~~scale (Note 4)~~ reading when the ~~pointer~~ point becomes steady (Note 4). When using a digital viscometer, record the reading directly from the digital display once it has been stabilized (Note 5).

NOTE 4—With an ~~ICI~~ analog cone/plate viscometer, whether the reading gives a direct indication of the viscosity or not, depends on the cone and scale used.

NOTE 5—In some cases it is difficult to judge whether a constant reading has been obtained. However, if the ~~pointer~~ reading does not become steady after 15 s, record the reading at 15 s and mention the lack of a constant reading in the test report. If highly accurate readings are required, make the readings below 90 % of the scale.

9.3 If the reading does not directly indicate the viscosity, multiply the reading by the appropriate conversion factor or use the appropriate calibration curve to obtain the viscosity.

9.4 Clean both the cone and the plate carefully, employing a cloth or tissue and a suitable solvent. Take care to remove all of the test material and cleaning solvent. Do not use cleaning utensils that may damage the apparatus. *Metal cleaning tools must never be used.*

9.5 Repeat the determination with a second specimen. If the two viscosity determinations differ by less than 7 %, calculate their mean and report as the high-shear viscosity for the material. If they differ by more than 7 %, make a third determination. If no two readings are within 7 % of each other, then the material is not suitable for testing by this test method.

10. Report

10.1 Report the following information:

10.1.1 Reference to this test method and the viscometer used,

10.1.2 Type and identification of the product under test,

10.1.3 Type of cone used,

10.1.4 Rate of shear at which the determination was made (in reciprocal seconds),

10.1.5 Temperature at which the determination was made,

10.1.6 Test results in poises, reported to the nearest 1 % of the total range, that is, 0.05 P for 0 to 5 P-cones, 0.1 P for 0 to 10 P-cones, etc.,

10.1.7 Any deviation, by agreement or otherwise, from the test procedures described, and

10.1.8 Date of the test.

11. Precision and Bias [5]

11.1 *Precision*—On the basis of an interlaboratory test of this test method in which eight operators in four laboratories tested six paints ranging in viscosity from 0.8 to 7.9 P on ~~ICI~~ analog cone/plate viscometers, the within-laboratory coefficient of variation

~~[5] Such oils~~

[5] Supporting data are available from ~~The Cannon Instrument Co., P.O. Box 16, State College, PA 16801.~~ ASTM Headquarters. Request RR:D01-1035.

was found to be 2.2 %, at 40 df. The between-laboratory coefficient of variation was found to be 6.9 % at 34 df. Based on these results, the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

11.1.1 *Repeatability*—Two test results, each the mean of two determinations, obtained by the same operator should be considered suspect if they differ by more than 6.3 % relative.

11.1.2 *Reproducibility*—Two results, each the mean of two determinations, obtained by operators in different laboratories should be considered suspect if they differ by more than 19.9 % 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 viscosity—paints/related coatings/materials; viscometer—ICI cone/plate; viscometer— cone/plate