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Standard Test Method for VISCOSITY OF EPOXY RESINS AND RELATED COMPONENTS[1]

This standard is issued under the fixed designation D 2393; 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.

This method has been approved for use by agencies of the Department of Defense and for listing in the DoD Index of Specifications and Standards.

1. Scope

1.1 This test method covers the measurement of the viscosity of epoxy resins, other epoxidized compounds, modifiers, and diluents used in formulating epoxy systems, liquid curing agents that effect the hardening of epoxy resins, and epoxy resin-curing agent systems or mixtures.

1.2 The viscosity of other liquid materials, either clear or opaque, can be determined by this test method.

1.3 While the test method described is valid for viscosities between 0.1 and 2000 Pa·s (100 and 2 000 000 cP), the use of a kinematic method of measurement is recommended for viscosities between 0 and 0.5 Pa·s (0 and 500 cP).

NOTE 1—For unfilled systems, more precise results may be obtained by using a kinematic procedure for viscosities up to 50 Pa·s (50 000 cP).

1.4 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems 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:*

D 1824 Test Method for Apparent Viscosity of Plastisols and Organosols at Low Shear Rates by Brookfield Viscometer[2]

E 1 Specifications for ASTM Thermometers[3]

3. Significance and Use

3.1 The procedures for testing permit the accurate determination of the viscosity of materials within the range from 0.1 to 2000 Pa·s (100 to 2 000 000 cP).

3.2 The tests may be used for both the characterizing and the quality control testing of liquid materials.

3.3 This procedure is related to Test Method D 1824, but is of more general application.

4. Apparatus

4.1 *Viscometer*, Brookfield Model RVF or equivalent.

NOTE 2—This test method is based on the use of a Brookfield viscometer.[4] Any other comparable viscometer may be used, provided that the limitations and procedures specified by the manufacturer are followed.

NOTE 3—Any viscometer must be checked for accuracy against standard liquids covering the normal range of operation of the instrument. The time lapse between checks must not exceed 6 months. A defective instrument must be recalibrated before further use, preferably by the manufacturer of the instrument.

4.2 *Bath*, temperature-controlled, controllable to ±0.1°C (±0.2°F), either oil or water type.

4.3 *Thermometer*, Celsius, with 0.1 divisions,

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[2] *Annual Book of ASTM Standards*, Vol 08.02.

[3] *Annual Book of ASTM Standards*, Vols 05.03 and 14.01.

[4] Available from Brookfield Engineering Laboratories, Inc., Stoughton, MA.

or Fahrenheit, with 0.2 divisions. A suitable thermometer is ASTM Solidification Point Thermometer having a range from 20 to 50°C and conforming to the requirements for Thermometer 91C as prescribed in Specifications E 1. For other temperature ranges, other ASTM standard thermometers are available.

4.4 *Glass Beaker*, 600-mL capacity, or 1-qt open-top paint can, approximately 10.4 cm (4.125 in.) in diameter and 11.8 cm (4.625 in.) deep.

5. Conditioning

5.1 Cover the sample and place in a temperature-controlled bath at the test temperature for at least 4 h prior to being tested, or for however much longer time is needed for all parts of the sample to reach the test temperature within ±0.1°C (±0.2°F). This conditioning may be carried out in the beaker or can in which the measurements are to be made.

NOTE 4—If the sample is a reacting mixture, such as a mixture of a resin with a hardener or catalyst, the resin component and the hardener component shall be brought to the test temperature separately. When both components have reached the test temperature, the resin and hardener shall be combined by slow agitation with a stirring rod or mixing paddle, avoiding the stirring-in of air. Three minutes of careful mixing is usually sufficient to yield a uniform blend. Immediately after mixing is complete, the spindle and the guard shall be attached to the viscometer. Readings shall commence 1 min after completion of mixing.

Greater precision in determining the viscosity of reacting systems can be obtained by use of Gardner tubes in a constant-temperature bath.

5.2 Periods of conditioning shorter than 4 h may be used, if experience has shown results to be comparable to those obtained after 4 h of conditioning. If shorter than 4 h, show the time of conditioning in the report.

5.3 Bring the spindle and guard to the test temperature before beginning testing.

6. Procedure

6.1 Using the Brookfield Viscometer Model RVF, the procedure is as follows:

6.1.1 Level the instrument.

6.1.2 Place a 500-mL portion of the previously conditioned sample in the clean beaker or can.

6.1.3 Insert the recommended spindle and guard into the sample, taking care to avoid the trapping of air under the spindle plate (Table 1).

NOTE 5—When the sample consists of a mixture of a liquid and a solid, for example, a filled resin, the solid material must be uniformly dispersed throughout the liquid phase.

If the sample is a mixture of liquids, for example, a mixture of a liquid resin with a liquid hardener, the liquids must be thoroughly interspersed, and the mixture must be visibly free of stirred-in air before inserting the spindle and guard.

NOTE 6—Since the accuracy of the viscometer is greatest in the middle of the dial range, it may be desirable to change the speed setting or the spindle, or both, to obtain a better reading. In general, greater accuracy is obtained by reading RVF values only on the "100" scale, and adjusting the Brookfield factor accordingly.

6.1.4 Start the viscometer 1 min after completion of any mixing (Note 7). Allow the spindle to rotate for 30 s. Stop the instrument through use of the clutch, and read the dial.

NOTE 7—Place the container on a nonconducting surface. Do not hold it in the hand, since heat may be transferred to or from the material being tested, thus affecting the viscosity. If extreme accuracy is needed, the measurements may be made while the sample is in its container in the constant-temperature bath.

The rigid, level mounting of the viscometer on a ring stand or equivalent, and the use of a height-adjusting platform for the sample container will be advantageous.

6.1.5 After recording the first reading, allow the spindle to rotate an additional three to four cycles, and take a second reading.

6.1.6 If the second reading agrees with the first, record this figure. If, however, the two readings differ, allow the spindle to rotate three or four more times, and read the value again. Continue this procedure for ten readings, or until a constant reading is obtained.

NOTE 8—If the sample is a reacting mixture, for example, a resin and a hardener, it will often be impractical or impossible to obtain a constant reading. In this case, the first reading is generally the one to record, unless it is desired to conduct a time versus viscosity study of the reaction.

6.1.7 Convert the reading obtained from the dial to viscosity in pascal seconds (or centipoises), in accordance with the conversion table in Table 1.

6.2 It is often of interest to determine the thixotropic characteristics of the sample under test. The viscosity is measured at different speeds, and a ratio relationship is set up as an index of relative thixotropicity or so-called thixotropic index. For example:

(Viscosity at Speed 2)/(Viscosity at Speed 20)
= 80 Pa·s (80 000 cP)/
20 Pa·s (20 000 cP) = 4