



Standard Practice for Preparing Resin Solutions for Viscosity Measurement by Bubble Time Method¹

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1. Scope

1.1 This practice provides instructions for preparing resin solutions viscosity measurement by bubble time method.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

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. For specific hazard statements, see Section 7.*

2. Referenced Documents

2.1 ASTM Standards:²

D154 Guide for Testing Varnishes

D1545 Test Method for Viscosity of Transparent Liquids by Bubble Time Method

D6440 Terminology Relating to Hydrocarbon Resins

E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

2.2 Other Document:

OSHA Regulations, 29 CFR, paragraphs 19.10.1000 and 1910.1200³

3. Terminology

3.1 For definitions related to hydrocarbon resins see Terminology D6440.

¹ This practice is under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and is the direct responsibility of Subcommittee D01.33 on Polymers and Resins.

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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 U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

4. Summary of Practice

4.1 Solid resins are dissolved in organic solvents by cold-cut or hot-cut methods in the laboratory. The viscosity of such prepared solutions, or of commercial solutions of resins is then determined by the bubble time method (Test Method D1545). The bubble seconds are approximately equal to stokes.

5. Apparatus

5.1 *Bath*, constant-temperature, consisting of a cylindrical glass jar of about 5-gal capacity, or an aquarium tank with controls capable of maintaining the temperature at $25 \pm 0.1^\circ\text{C}$ with water as the bath medium.

5.2 *Bottles*, 225-mL (8-oz), wide-mouth, screw cap.

5.3 *Cellophane*, sheet, cut into 102 or 127- mm (4 or 5-in.) squares.

5.4 *Corks*, No. 2 short taper, to fit viscosity tubes.

5.5 *Holder for Viscosity Tubes*, preferably a mechanical holder⁴ with rack and pinion for inverting the tubes. The holder should be checked with a small level and T-square to make certain it holds the tubes in an exact vertical position after being placed in the constant-temperature bath.

5.6 *Bottle Shaker*, preferably one which will give end-over-end agitation.

5.7 *Timing Device*, such as a stopwatch or electric stop clock capable of being read to a precision of 0.1 s.

5.8 *Viscosity Tubes*, of clear glass and flat bottoms, having 10.65 ± 0.025 -mm inside diameter and 114 ± 1 -mm outside length. Plainly legible lines shall be located on the tubes as follows:

27 ± 0.5 mm
 100 ± 0.5 mm
 108 ± 0.5 mm

All distances shall be measured from the bottom outside of the tube. The distance between the first and second lines shall be 73 ± 0.5 mm.

⁴ Suitable mechanical viscosity tube holders are available from various laboratory supply companies.

6. Solvent

6.1 The solvent used should be mutually agreed upon between the purchaser and the manufacturer. Toluene, xylene, mineral spirits, alcohols, etc., are used for the preparation of solutions of resins. For a given resin, the viscosity obtained will depend on the solvent used. In case of dispute, both laboratories should use portions of the same batch of solvent.

7. Hazards

7.1 Consult current OSHA regulations, supplier's Material Safety Data Sheets, and local regulations for all materials used in this practice.

8. Preparation of Resin Solutions

8.1 The procedure employed for dissolving solid resins will be dependent upon the chemical nature of the resin under test. Some resins will dissolve readily in cold solvent with moderate agitation, while others must be hot-cut in order to effect solution. The method of solution shall be agreed upon mutually between the purchaser and the seller since there may be differences in solubility or viscosity between solutions prepared with and without heat. The solvent concentration may vary from 30 to 90 % and is generally selected to give a solution having a viscosity between 5 and 300 s. With hard resin use only material taken from freshly broken lumps. Do not use crushed or powdered material from the resin sample container.

8.2 *Cold-Cut Solutions*—Because it is not easy to tell when solution of the resin is complete, proceed exactly in accordance with the following directions, unless experience has shown that short cuts can be safely taken:

8.2.1 Prepare 100 g of solution as follows: Weigh an 225-mL (8-oz), screw-cap bottle, cap, and cellophane sheet to the nearest 0.05 g. Weigh into the bottle to the nearest 0.05 g the appropriate amount of solvent (40 g for a 60 % solution).

8.2.2 Weigh into a beaker to the nearest 0.05 g the correct amount (60 g for a 60 % solution) of the freshly crushed resin, passing a No. 10 (2000- μ m) sieve.

8.2.3 Pour the resin slowly into the bottle containing the solvent, swirling gently so as to wet the resin as thoroughly as possible as it is added. Place the cellophane sheet over the mouth of the bottle and screw on the cap tightly.

8.2.4 Immediately shake vigorously by hand and place on a shaker to give end-over-end agitation for an overnight period.

8.2.5 Check the weight of the bottle, cap, cellophane and solution to make certain that none was lost during the mixing. If appreciable loss has occurred, discard the solution and prepare a fresh one.

8.3 Hot-Cut Solutions:

8.3.1 Prepare 100 g of solution as follows: Weigh a 250-mL Erlenmeyer flask with a ground joint to the nearest 0.05 g. Weigh into the flask to the nearest 0.05 g the appropriate amount of solvent (40 g for a 60 % solution).

8.3.2 Weigh into the flask to the nearest 0.05 g the correct amount (60 g for a 60 % solution) of the freshly crushed resin passing a No. 10 (2000- μ m) sieve. Connect the flask to a suitable air or water condenser and warm gently on a hot plate with swirling as necessary to prevent any charring or scorching of the resin. Usually, the solution will be complete in 15 or 20 min.

8.3.3 When the solution is complete, cool the flask, disconnect, and weigh. If over 0.1 g of solvent has been lost, add the necessary amount and swirl to mix.

8.3.4 If any air bubbles are present, allow the solution to stand until they disappear.

8.4 *Resins Supplied in Solution*—These resins ordinarily can be tested for viscosity as received. However, if modification is necessary, such as a reduction to a specified solids content, use a solvent or solvent combination suitable for use with resin under test, as agreed upon between the manufacturer and the purchaser.

9. Procedure

9.1 Determine the viscosity of the resin solution in accordance with Test Method **D1545**, repeating the timing determination until three readings are obtained that agree within 10 s or 3 % relative, whichever is larger.

9.2 For viscosities of solutions which have a timed bubble travel of 5 s or less, more precise results can be obtained by comparison against reference standards having predetermined viscosity (Guide **D154**) or timed bubble travel (Test Method **D1545**).

10. Report

10.1 Report the following information:

10.1.1 The mean bubble travel time, in seconds, as the viscosity at 25°C, including:

10.1.1.1 Solution concentration,

10.1.1.2 Solvent used, and

10.1.1.3 Whether cold- or hot-cut solutions were used.

11. Keywords

11.1 bubble time; resin solutions; viscosity

APPENDIX

(Nonmandatory Information)

X1. INTERLABORATORY STUDY

X1.1 An interlaboratory study using this standard practice in conjunction with Test Method **D1545** was conducted when the practice was originally approved in 1960.

X1.2 *Repeatability and Reproducibility*—Two results, each the mean of three readings, should be considered suspect if they differ by more than 7 % relative (95 % confidence level). See Practice **E691**.

NOTE X1.1—This estimate of precision is based on an interlaboratory study.⁵ Because the data showed no significant difference for between laboratory and within laboratory variances, the precision estimates for repeatability and reproducibility were pooled and a single value reported.

⁵ For the results of the interlaboratory study, see Report of Committee D01, *Proceedings*, ASTM Vol 60, 1960.

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