Designation: D5165 – 12

Standard Practice for Laboratory Preparation of Gelled Vehicles Using a Resin Kettle[1]

This standard is issued under the fixed designation D5165; 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 practice covers a laboratory procedure for the gelation of a resin solution, ink varnish, or vehicle using a resin kettle.

1.2 Guidance in preparing gelled vehicle samples suitable for use in laboratory sample quantity oil-based printing inks is provided.

1.3 The procedure outlined is not intended as a means of rating or evaluating resin or vehicle gelability, and is applicable only if the solutions, varnishes, or vehicles produced are of a rheology that is measurable by conventional ink and varnish industry viscometers or rheometers.

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

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]

D154 Guide for Testing Varnishes

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

D1725 Practice for Preparing Resin Solutions for Viscosity Measurement by Bubble Time Method

D4040 Test Method for Rheological Properties of Paste Printing and Vehicles by the Falling-Rod Viscometer

E1 Specification for ASTM Liquid-in-Glass Thermometers

3. Terminology

3.1 *Definitions:*

3.1.1 *gel or gelled vehicle*—any resin solution, or a more complex blend of resins and alkyds that has been cooked to build molecular weight or reacted with a gelling or crosslinking agent (for example, organo-aluminum compounds) and exhibits a pseudoplastic rheology.

3.1.2 *pregel or pregel vehicle*—the resin solution or vehicle components comprising the vehicle prior to the addition of gelling agent and viscosity adjusting solvent.

3.1.2.1 *Discussion*—The terms "vehicle" and "varnish" are commonly used interchangeably. Some vehicle and varnish chemists, however, may consider a varnish to be a vehicle that undergoes a chemical reaction during a cooking cycle.

3.1.3 *gel seed, n*—a non-homogeneous, gelatinous particle in a gel vehicle, often the result of poor mixing or localized over-reaction during gelation.

4. Summary of Practice

4.1 Prepare a pregel vehicle by dissolving all vehicle or varnish components, or both, in a heated 1-L, resin kettle with inert gas blanket.

4.2 Add gellant solution into the pregel vehicle. Mix using the suggested stirring rate at the prescribed reaction temperature for 1 h.

4.3 Remove the resin kettle from heat, evaluate, adjust if necessary, and re-evaluate.

5. Significance and Use

5.1 This practice provides a means of preparing gel vehicles in laboratory equipment that most closely resembles production reactors. It can be used to predict the performance of gel vehicle components (resins, gellants, alkyds, etc.) in the user's equipment.

5.2 An ample amount of gel vehicle can be prepared for use in preparing inks for press testing.

6. Apparatus (see Fig. 1)

6.1 *Balance or Scale,* accurate to 0.1 g.

6.2 *Resin Kettle,* 1-L with 5-neck kettle head.

[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 D01.37 on Ink Vehicles.

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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.

***A Summary of Changes section appears at the end of this standard**

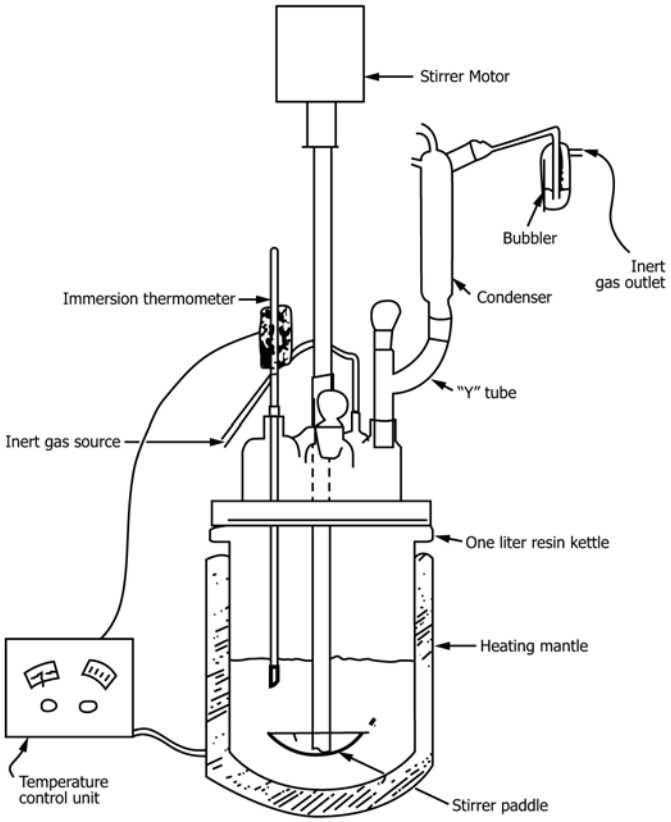

FIG. 1 Gel Vehicle Preparation Apparatus

6.3 *Electric Stirrer,* equipped with speed control.

6.4 *Glass Stirring Rod.*

6.5 *Paddle,* 6.4 cm (2.5 in.) in width.

6.6 *Heating Mantle,* to fit 1-L resin kettle.

6.7 *Electronic Temperature Control Device.*

6.8 *Thermometer, capable of reading,* 0 to 250°C and conforming to Specification E1. Alternately, temperature measuring devices such as liquid-in-glass thermometers, thermistors, thermocouples, or platinum resistance thermometers that provide equivalent or better accuracy and precision, that cover the temperature range specified, may be used.

6.9 *Inert Gas Blanket Source,* (for example, nitrogen, carbon dioxide).

6.10 *Auxiliary Equipment,* (that is, stopper, Y tube, condenser, bubbler, etc.).

6.11 *Insulated Gloves.*

7. Reagents and Materials

7.1 Nonvolatile materials will include resins, alkyds, gellants, etc., consistent with those used in the manufacture of lithographic ink vehicles (for example, aromatic hydrocarbon resins, modified rosin ester resins, long oil linseed isophthalic alkyds, and aluminum compounds).

7.2 Solvents used in this procedure will be those most often used in the manufacture of lithographic ink vehicles, for example, hydrocarbon petroleum distillate C12–C16.

8. Procedure

8.1 Weigh oil, solvent, and alkyd components for a 500-g sample into resin kettle. Do not add resin and gellant components at this time.

8.2 Separately prepare a gellant solution to be added after resin dissolution. Dilute gellant to between 30 and 50 % gellant with the major vehicle solvent.

8.3 Start mixer; be sure that all materials are mixing.

8.4 Start heating the mixture to 160°C, (or the defined reaction temperature) at full power, under inert gas blanket and refluxing. Record time.

8.5 Slowly add the resin component.

8.6 At 150°C, reduce to approximately ¾ power and continue heating to 160°C.

8.7 When the temperature reaches 160°C, check that all systems components have been dissolved into a homogeneous solution. If yes, record the time and the r/min of the stirrer (if possible). If not, allow system to mix until all components are dissolved.

8.8 After a homogeneous vehicle is obtained, reduce the solution temperature to 135°C.

8.9 Add the desired quantity of gellant solution to the pregel vehicle and mix thoroughly.

8.10 Raise reaction temperature to 160°C (or defined reaction temperature), stirring at 380 to 420 r/min (if able to determine stirring speed), and record time.

NOTE 1—Different gellants have different activation temperatures, and different resins have different reactivity. Adjust temperature accordingly to complete reaction while preventing gel seeding.

8.10.1 Hold at 160°C, or other reaction temperature, for 1 h, with constant stirring.

8.11 After 1 h, or the prescribed reaction cycle, discontinue heating.

8.12 Allow kettle to cool to a safe handling temperature.

8.13 Collect test sample from the kettle and evaluate.

8.14 Based on the results of the evaluation, make adjustments as necessary to the gel varnish before discharging (for example, adding resin, adding gellant, adding solvent). Repeat steps from above to achieve desired test results.

8.15 Once gel vehicle passes test requirements, transfer into a 1-qt can or other container.

9. Evaluation

9.1 Sample should be tested immediately after preparation, and after 24 h, to determine the effects of post gelation.

9.2 Guide D154 outlines many methods applicable to the testing of varnishes. It is an excellent guide for those looking for specific areas of evaluation.

9.3 Lightly gelled resin solutions and vehicles can be tested for changes in viscosity by using either Test Methods D1545 or D1725. These test methods both use Gardner bubble tubes to determine the viscosity of liquids.

9.4 Another test method for testing gelled ink vehicles is Test Method D4040. This test method utilizes a falling-rod viscometer and can be used to determine the non-Newtonian nature of vehicles. The test method gives poor precision, however. Other viscometers used for determining the rheology of gelled vehicles include: variable shear rate viscometers, and cone and plate viscometers.

10. Report

10.1 Report the following information on vehicle preparation:

10.1.1 Sample identification,

10.1.2 Test run,

10.1.3 Starting time of heat-up,

10.1.4 Initial stirring rate in r/min,

10.1.5 Time when vehicle temperature reaches 160°C, or defined reaction temperature,

10.1.6 Time at which all pregel material is dissolved,

10.1.7 Time at which cooling cycle is initiated,

10.1.8 Time at which pregel vehicle reached 120°C or below,

10.1.9 Time when gellant solution is added to pregel vehicle,

10.1.10 Time when heat up cycle toward 160°C has begun,

10.1.11 Length of time that the reaction is held at 160°C or the optimum reaction temperature, and

10.1.12 Time and temperature when gel vehicle is poured for storage.

10.2 A report on gel vehicles properties should contain:

10.2.1 Sample identification,

10.2.2 Test run identification, and

10.2.3 Results of the analyses made. In the case of falling rod viscometer determinations the following data could be obtained using Test Method D4040:

10.2.3.1 Apparent viscosity at 2500/s at 25°C,

10.2.3.2 Pseudo-Bingham Yield value at 25°C,

10.2.3.3 Shortness ratio (yield value/apparent viscosity), and

10.2.3.4 Slope (non-Newtonian parameter) as defined in Test Method D4040.

11. Keywords

11.1 falling rod viscometer; gel; gelability; gelled varnish; gelled vehicle; non-Newtonian fluid; pregel; pregel varnish; pregel vehicle; slope; yield value shortness ratio

SUMMARY OF CHANGES

Committee D01.37 has identified the location of selected changes to this standard since the last issue (D5165 – 93 (2004)) that may impact the use of this standard. (Approved November 1, 2012.)

(1) Terminology — added definition for “gel seed.”
(2) Procedure — clarified optimum order of addition. Added commentary/caution regarding gel reactivity and the potential to generate gel seeds during the reaction.