Designation: D5166 – 97 (Reapproved 2016)

Standard Practice for Laboratory Preparation of Gelled Vehicle Samples Using a Microwave Oven[1]

This standard is issued under the fixed designation D5166; 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 outlines a procedure for preparing gelled vehicle samples using a microwave oven.

1.2 The test samples can be used for characterizing the gelability or reactivity of resins, gelling agents, and vehicles used in the manufacture of oil based printing inks, or both.

1.3 Evaluation of the gelled vehicles may, depending upon preference, be either visual or instrumental.

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]

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 of Terms Specific to This Standard:*

3.1.1 *gel, n*—Any resin solution, or more complex blend of resins and alkyds that has been cooked or reacted with a gelling or cross-linking agent to build molecular weight and that exhibits a pseudoplastic rheology. (Also called "gelled vehicle.")

3.1.2 *gelling agent, n—in the context of ink resins and vehicles*, gelling agents or gellants are typically organo-aluminum compounds that react with carboxylic acid and hydroxyl groups present on the backbone of resins and alkyds to form cross-linked networks. (Also called "gellant.")

3.1.3 *gel length, n*—References to long, medium, and short gel length refer to the length of a string of gelled vehicle observed when pulling apart a small sample of vehicle with a spatula (that is, a long gel is very fluid and forms a "long" filament; a short gel has little flow and forms a "short" filament).

3.1.4 *pregel, n*—The resin solution or vehicle components comprising the vehicle prior to the addition of gelling agent and viscosity adjusting solvent. (Also called "pregel vehicle.")

4. Summary of Practice

4.1 Prepare a pregel resin solution or vehicle.

4.2 Separate the pregel vehicle into five or more aliquots to which various levels of gellant are added. Alternately, various gellants can be added at a given concentration to compare their effectiveness.

4.3 Place the test samples on a turntable in the microwave oven and heat the samples at full power until the desired test temperature is attained.

4.4 After the prescribed waiting period, test and rate samples for gelability.

5. Significance and Use

5.1 This practice provides a means of preparing a number of gel vehicle samples with minimum use of materials and time. It provides a means of quickly characterizing and comparing the gelability or reactivity of resins, vehicles, and gelling agents.

6. Apparatus

6.1 *Mercury Thermometer,* capable of reading from 0 to 250°C and conforming to Specification E1.

[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.

Current edition approved April 1, 2016. Published April 2016. Originally approved in 1991. Last previous edition approved in 2008 as D5166 – 97 (2008)ε1. DOI: 10.1520/D5166-97R16.

[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.

6.2 *Jars,* (40 mm in diameter by 83 mm in height), 70 cm^3 with nonmetallic screw-on lids.

6.3 *Microwave Oven,* common 750-watt.[3]

6.4 *Microwave Oven Carousel,* if not contained in the microwave oven.

6.5 *Glass Dish,* 15.2 cm in diameter.

6.6 *Balance or Scale,* capable of weighing to ±0.1 g accuracy.

6.7 *Ink Knife or Rigid Spatula.*

6.8 *Rheology Testing Apparatus,* at the discretion of the user.

7. Reagents and Materials

7.1 Nonvolatile materials including 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 typically used in the manufacture of lithographic ink vehicles, for example, hydrocarbon petroleum distillate with carbon numbers in the range of C12 to C16 and initial boiling point above 150°C.

8. Procedure

8.1 Place 45 g (or the calculated weight) of previously prepared pregel vehicle into each of five or more 70-cm^3 bottles.

NOTE 1—Any vehicle, even a very complex formulation, without the gellant added may be used as the pregel vehicle portion for the microwave oven gelability process.

8.1.1 In the absence of a vehicle formula agreed upon between user and seller, the following test vehicle formula is suggested:[4,5]

	Parts
Pregel Vehicle	
Resin	35
Alkaline refined linseed oil (ARLO)	30
C12-C16 hydrocarbon petroleum distillate	25
	90
Gellant/Solvent Addition	
33.3 % oxyaluminum octoate/66.7 % C12-C16 hydrocarbon petroleum distillate	10
	100

8.2 Estimate the amount of gellant required to make an acceptable gel vehicle. Add gellant solution (between 30 and 50 % gellant in major vehicle solvent recommended) in 1 % increments plus additional solvent to adjust the vehicle to a 100 % formula. If the amount of gellant required can not be estimated, use a larger range of gellant concentration.

8.2.1 If using the pregel formula as just recommended, the required amount of gellant can be estimated from Table 1.

8.2.2 A sample containing no (0 %) gellant solution is always prepared as the standard for each resin.

TABLE 1 Microwave Gelability Test, Weights and Concentrations for 50-g Samples

Gellant Solution, %	Actual Gellant Concentration, %	Weight Gellant Solution, g[A] (at 33.3 % Gellant in Ink Oil)	Weight Ink Oil, g[A]
0	0.00	0.0	5.0
1	0.33	0.5	4.5
2	0.67	1.0	4.0
3	1.00	1.5	3.5
4	1.33	2.0	3.0
5	1.67	2.5	2.5
6	2.00	3.0	2.0
7	2.33	3.5	1.5
8	2.67	4.0	1.0
9	3.00	4.5	0.5
10	3.33	5.0	0.0

[A] Weight added to 45-g pregel sample.

8.3 Mix the added liquids into the pregel very well using an ink knife or rigid spatula.

8.4 Place the five sample bottles, without tops, into the 15.2-cm glass dish and place on the carousel in the microwave oven.

8.5 Turn on the carousel and ensure that it is turning before starting the heating cycle. Turn on the light in the oven to observe the process.

8.6 Set the timer on the oven for "5.00" min and start the cooking cycle.

8.7 Upon completion of the cooking cycle, check the temperature of the samples to determine whether a temperature of 105 to 110°C was obtained. If not, continue the heating cycle in 1-min intervals until the samples have reached this temperature.

8.8 Remove the samples from the microwave oven, and allow to cool.

8.9 Seal cooled samples with jar lids.

NOTE 2—If more than one set of samples is to be run, allow the microwave oven to cool between runs. Heat is retained in the oven and if a second set of samples is run immediately it will be subjected to more heat than the first set.

9. Evaluation

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

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 Method D1545 or D1725. These test methods both use Gardner bubble tubes to determine the viscosity of liquids.

[3] Any common microwave oven, available at most appliance stores, can be used. A 750–watt oven with a built–in turntable is preferred. Ovens with lower power will require adjustments in the test cycle.

[4] This formula works best with higher molecular weight ink resins (for example, phenolic modified rosin esters). It is suggested that the resin solids be increased for low molecular weight resins (for example, simple rosin esters, modified hydrocarbons, etc).

[5] When a resin is insoluble, reformulate replacing 5 parts of C12-C16 hydrocarbon petroleum distillate with TXIB (2,2,4-trimethyl-1,3-pentanediol, diisobutyrate) or TDA (tridecyl alcohol). **Caution:** TDA may hinder the reactivity of the resin system tested.

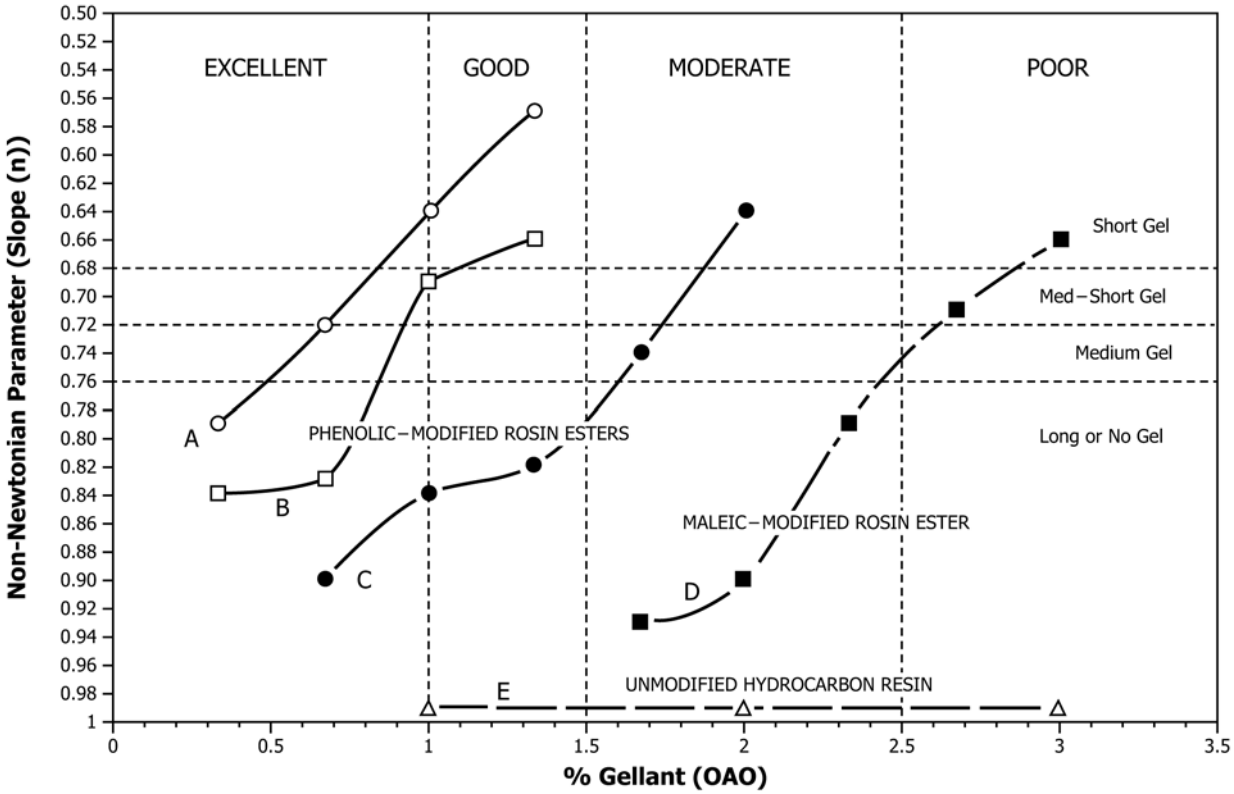

FIG. 1 Determination of Lithographic Ink Resin Gelability

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

9.5 For the trained analyst the occurrence of gelation is evident and gels can be rated visually (for example, long, medium, medium short, short gel body).

10. Report

10.1 Report the following information:

10.1.1 Sample identification,

10.1.2 Test run,

10.1.3 Brief summary of procedure,

10.1.4 Conditions of test procedure, and

10.1.5 Results of the analyses made. In the case of falling rod viscometer determinations, the following data could be obtained using Test Method D4040 (see also the Apparatus Section of this test method):

10.1.5.1 Apparent viscosity at 2500 at 25°C, s,

10.1.5.2 Pseudo-Bingham Yield value at 25°C,

10.1.5.3 Shortness ratio (yield value/viscosity), and

10.1.5.4 Slope (non-Newtonian parameter).

NOTE 3—For a measurement of relative resin, vehicle, or gellant reactivity, lots of rheology versus gellant concentration (see Fig. 1) provide an excellent comparison.

NOTE 4—Falling rod viscosity slope (non-Newtonian parameter), shortness ratio, and visual degree of shortness, if used properly, are all suitable measures of rheology.

11. Keywords

11.1 gel; gelability; gelled varnish; gelled vehicle; microwave oven; non-Newtonian fluid; pregel; pregel varnish; pregel vehicle; shortness ratio; slope; yield value