Designation: D6038 – 21

Standard Test Methods for Determining the Compatibility of Resin/Solvent Mixtures by Precipitation Temperature (Cloud Point)[1]

This standard is issued under the fixed designation D6038; 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 These test methods cover the manual and automatic procedures for testing the compatibility of lithographic ink resins in high boiling ink solvents by precipitation temperature (cloud point) in a range from 35 to 210°C.

1.2 The manual procedure in this test method uses laboratory equipment generally available in a normal, well-equipped laboratory. The automated procedure uses a programmable cloud point tester.

1.3 This test method is for use with ink resins intended mainly for oil-based offset and letterpress inks. The type of resins are typically, but not limited to C_9 aromatic hydrocarbon resins, modified dicyclopentadiene resins, rosin pentaerythritol or glycerol esters, phenolic modified rosin esters, maleic anhydride modified-rosin esters, and naturally occurring resins such as gilsonite.

1.4 A resin solution or ink vehicle could also be used in this test instead of the resin.

1.5 The typical high boiling solvents to be used are C_{12} to C_{16} petroleum distillates.

1.6 To avoid fire or injury, this test method should not be used with low flash point solvents such as toluene or xylene. The minimum flash point of the solvents used should be 60°C as determined by Test Method D56.

NOTE 1—Users of this test method should be aware that the flash point of many solvents used for this test (as defined in Test Methods D56 and D1310) is exceeded in the heating cycle of this test method. Safety precautions should be taken since there is the potential for vapor ignition. The method outlined should be done in a shielded exhaust hood, where there is access to a fire extinguisher if needed.

1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.9 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D56 Test Method for Flash Point by Tag Closed Cup Tester

D1310 Test Method for Flash Point and Fire Point of Liquids by Tag Open-Cup Apparatus

E180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial and Specialty Chemicals (Withdrawn 2009)[3]

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

3. Terminology

3.1 *Definitions:*

3.1.1 *cloud point, n*—(precipitation temperature) the temperature at which a resin/solvent mixture changes from clear to turbid and opaque.

3.1.2 *compatibility, n*—resin and solvent mixture forms a clear, homogeneous, and stable solution at room temperature.

3.1.3 *incompatibility, n*—resin and solvent mixture does not form a uniform solution and may be in two phases or opaque.

3.1.4 *n*—degrees of freedom.

3.1.5 *precipitation, n*—resin separates from the resin/solvent mixture.

[1] These test methods are under the jurisdiction of ASTM Committee D01 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.37 on Ink Vehicles.

Current edition approved Feb. 1, 2021. Published February 2021. Originally approved in 1996. Last previous edition approved in 2014 as D6038 – 14. DOI: 10.1520/D6038-21.

[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] The last approved version of this historical standard is referenced on www.astm.org.

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

3.1.6 *solubility, n*—the degree of resin compatibility in a given solution.

4. Summary of Test Method

4.1 A 10 % by weight mixture of the resin (or ink vehicle) to be tested in the reference solvent (or vice versa) is heated in a test tube and stirred until a clear solution is obtained.

4.2 The solution is allowed to cool. The cloud point is the lowest temperature that can be read on a thermometer, positioned at the back wall of the test tube, or in the case of the automated tester, before the solution reaches the preset turbidity level.

4.3 If the solution remains clear at room temperature, the test tube is cooled (cold water, ice water, or dry ice/acetone bath) until the cloud point can be recorded.

5. Significance and Use

5.1 These test methods provide a means of determining the compatibility of a resin (or vehicle), at low concentrations, in a high boiling ink solvent.

5.2 Resin-solvent mixtures that exhibit a high precipitation temperature are less compatible than those exhibiting a low precipitation temperature.

5.3 Resin-solvent mixtures that exhibit precipitation temperatures at or close to the cloud point of the pure solvent are considered infinitely compatible or the resin is infinitely soluble in that solvent.

6. Reagents and Materials

6.1 *Resins,* (for example, modified hydrocarbon, or rosin ester resins, or both) or Ink Vehicle.

6.2 *Solvents,* will be those commonly used for making lithographic ink vehicles, for example, hydrocarbon petroleum distillate C_{12} to C_{16} as agreed upon by producer and user.

NOTE 2—The use of ink industry recognized Test Ink Oils are recommended for evaluating resins. The Test Ink Oils are controlled from lot to lot to ensure meaningful data. (Contact NAPIM[4] for a list of approved Test Ink Oils).

6.3 *Calibration Materials,* PRIFRAC 2981 stearic acid and Haltermann N-C16 oil[5] designated for automatic method only but can be used for manual method.

MANUAL METHOD

7. Apparatus

7.1 *Balance or Scale,* weighing to ±0.02 g accuracy and a minimum 250 g capacity.

7.2 *Heat-Resistant Test Tube,* 25–mm width by 150–mm height.

7.3 *Thermometer,* 0 to 250°C range.

7.4 *Thermometer,* -100 to 50°C range.

7.5 *Hot Air Gun,* suitable to reach 260°C.

7.6 *Auxiliary Equipment,* (that is, mixing loop (if desired), lab stand, and test tube clamp, 500-ml beaker for cooling, etc.).

8. Sample Preparation

8.1 Crush the solid resin to particles not larger than 2 mm^3 and preferably not larger than 1 mm^3.

9. Procedure

9.1 Weigh 2.00 ± 0.02 g of crushed resin and 18.00 ± 0.02 g of the solvent to be tested into the test tube.

9.2 Place test tube with the sample, the thermometer, and the optional mixing loop in a tube clamp.

9.3 Aim the heat gun from below at the bottom of the test tube (keep nozzle at least 25 mm from tube), and turn on.

9.4 Mix resin and solvent slowly as heat rises.

NOTE 3—To avoid loss of solvent while stirring, do not remove the stirring apparatus from the mixture.

9.5 **Warning—**Care must be taken in stirring the resin/solvent mixture not to drop the thermometer or stirring loop. This could cause the test tube to break, which could cause a serious fire or accident.

9.6 Heat to 200°C; hold this temperature for 2 min, and then remove the heat source.

NOTE 4—Low softening point and very compatible resins will dissolve at temperatures well below 200°C. The maximum temperature to dissolve such resin and solvent mixtures can be much lower than 200°C. In such cases the maximum temperature for the test should be one agreed upon between the customer and the supplier and recorded.

9.7 Check to see that all resin is dissolved.

9.8 Start to blow ambient air from the heat gun onto the test tube.

9.8.1 If the mixture is not clear after heating for 2 min at 200°C, continue to heat up to 250°C or until dissolution has occurred and keep 2 min at that temperature, record the temperature at dissolution. Start the cooling procedure at this point.

9.8.2 If after the hold time of 2 min, in either case, the mixture is clear except for a very slight presence of precipitate, start the cooling procedure and note the presence of "slight precipitate."

9.8.3 If the mixture does not become clear or exhibits significant precipitation, record it as incompatible.

NOTE 5—Make sure that in case of 9.8.2 or 9.8.3 the cause of the lack of dissolution is NOT too large initial particles due to insufficient crushing.

9.9 Observe mixture closely as temperature drops and record the approximate rate of cooling, and the temperature at which solution becomes so cloudy that the thermometer, when held at the back wall of the test tube, can no longer be read (or the thermometer fluid can no longer be seen). This observation is defined as the precipitation temperature or cloud point (see Note 6 and Note 7).

NOTE 6—Cooling water, ice water, or a dry ice/acetone bath and a low temperature thermometer may be needed to reach the precipitation

[4] National Association of Printing Ink Manufacturers (NAPIM), 581 Main St., Woodbridge, NJ 07095, (napim@napim.org).

[5] Available from IGT Testing Systems, Randstad 22-2, P.O.box 22022, 1316 BX Almere, The Netherlands.

temperature for very soluble resins.

NOTE 7—This test method is recommended for resins and solvent mixtures exhibiting a precipitation temperature between 50 and 180°C at 10 % resin solids. If a mixture is out of that range, a change in resin concentration (for example, 20 % solids) or solvent is recommended.

10. Report

10.1 A report on precipitation temperature should note the following information:

10.1.1 *The Method Used*—MANUAL;

10.1.2 Sample identification;

10.1.3 Number of repeats and test run number;

10.1.4 Maximum temperature achieved;

10.1.5 Approximate rate of cooling;

10.1.6 Precipitation temperature;

10.1.7 *General Information*—Date, time, location; and

10.1.8 Any information on conditions which may have influenced the results of the test.

11. Precision and Bias[6]

11.1 *Precision*—An interlaboratory study of the precipitation temperature of two resins was run by seven laboratories.

11.1.1 The precision estimates here, are based on one analyst in each laboratory performing triplicate determinations on each of two materials.

11.1.2 Practice E180 was used in developing these precision statements.

11.2 *Repeatability (Within-Laboratory)*—The standard deviation of results obtained by the same analyst running the different analyses has been estimated to be 0.8° absolute at n = 20 at the 60°C level and 2.4° absolute at n = 20 at the 160°C level. The 95 % limit for the difference between two such averages is 2.1° absolute and 6.7° absolute, respectively.

11.3 *Reproducibility (Multilaboratory)*—The standard deviation of results obtained by analysts in different laboratories has been estimated to be 4.1° absolute at n = 6 at the 60°C level and 6.9° absolute at n = 6 at the 160°C level. The 95 % limit for the difference between two such averages is 11.5° absolute and 19.3° absolute, respectively.

11.4 *Bias*—The procedure in this test method has no bias because the value of the precipitation temperature is defined in terms of this test method.

AUTOMATED METHOD

12. Apparatus

12.1 *Balance or Scale,* weighing to ±0.02 g accuracy and a minimum 250 g capacity.

12.2 *Programmable Cloud Point Tester.*

12.3 *Glass Tube*[5], with external diameter 22 mm, length 20 cm.

12.4 *Mortar and Pestle.*

12.5 *Magnetic Stirring Bar*[5], PTFE coated, length 50 mm, diameter 7 mm.

12.6 *PTFE Tube Holder*[5], temperature insulating.

13. Materials

13.1 PRIFRAC 2981 stearic acid.[5]

13.2 Haltermann N-C16 oil.[5]

14. Sample Preparation

14.1 Crush the solid resin to a size no larger than 2 mm[3].

15. Instrument Calibration

15.1 Place the test tube with magnetic stirring bar and tube holder on the balance.

15.2 Weigh 10 ± 0.02 g of PRIFRAC 2981 stearic acid[5] with 10 ± 0.02 g of Haltermann N-C 16 oil [5] into the test tube.

15.3 Place the test tube into the cloud point tester.

15.4 Place the thermocouple assembly on the tube on the cloud point tester.

15.5 Start the test.

15.6 Tests should be repeated and the final cloud point for correct calibration of the instrument is 58°C ± 1. If the test result is outside this range, consult your manual for troubleshooting or call the manufacturer.

15.7 The manufacturers suggest calibration every two weeks.

16. Procedure

16.1 Insert the magnetic stirring bar into the test tube. Place the test tube in the tube holder.

16.2 Weigh 2.00 ± 0.02 g of crushed resin and 18.00 ± 0.02 g of the solvent to be tested into the test tube.

NOTE 8—Care should be taken to wash down the dust on the sides of the tube with solvent.

16.3 Place the test tube with test sample into the cloud point tester and insert the thermocouple assembly attached to the instrument.

16.4 Start the standard cloud point test sequence and the machine will run automatically.

NOTE 9—Resins that have not been tested before may require the operator to lift the tube once the test reaches 230°C. A visual inspection should confirm that the resin is in solution and that the solution is clear.

16.5 The instrument will indicate and record the cloud point temperature.

16.6 Remove the test tube from the instrument; discard sample, and clean using normal lab cleaning procedures.

17. Instrument Sequence

17.1 There are two common ways to operate these instruments: semi-automatic using the settings and timing as in Table 1 – Program 1 or the fully automatic sequence as in Table 1 – Program 2. Program 2 uses factory preset conditions. Other programs and sequences can be run as long as customer and supplier agree.

[6] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR:D01-1103. Contact ASTM Customer Service at service@astm.org.