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AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Test Method for Determining the Compatibility of Resin/Solvent Mixtures by Precipitation Temperature[1]

This standard is issued under the fixed designation D 6038; 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 test method covers the procedure for testing the compatibility of lithographic ink resins in high boiling ink solvents by precipitation temperature.

1.2 This test method uses laboratory equipment generally available in a normal, well-equipped laboratory.

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 glycerine esters, phenolic modified rosin esters, maleic anhydride modified-rosin esters, and naturally occurring resins such as gilsonite.

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

1.5 To avoid fire or injury to the operator, or both, 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 (140°F) as determined by Test Method D 56.

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 D 56 and D 1310) 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.

1.6 *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:*

D 56 Test Method for Flash Point by Tag Closed Tester[2]

D 1310 Test Method for Flash Point and Fire Point of Liquids by Tag Open-Cup Apparatus[3]

E 1 Specifications for ASTM Thermometers[4]

E 180 Practice for Determining the Precision of ASTM Methods for Analysis and Testing of Industrial Chemicals[5]

3. Terminology

3.1 *Definitions:*

3.1.1 *cloud point*—the point at which precipitation causes a resin/solvent mixture to become cloudy and opaque.

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

3.1.3 *incompatibility*—resin and solvent mixture is not compatible, an opaque or two-phase mixture results.

3.1.4 *precipitation*—resin separates from the resin/solvent mixture.

3.1.5 *precipitation temperature*—the temperature at which resin precipitation causes a cloud point.

3.1.6 *solubility*—the degree of resin compatibility, is solution compatible at all levels of resin and solvent.

4. Summary of Test Method

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

4.2 The solution is allowed to cool. The end point is the lowest temperature that can be read on a thermometer, positioned at the back wall of the test tube, before the solution gets cloudy.

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 This test method provides a means of determining the compatibility of a resin, 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

[1] This test method is under the jurisdiction of ASTM Committee D-1 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] *Annual Book of ASTM Standards*, Vol 05.01.

[3] *Annual Book of ASTM Standards*, Vol 06.01.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] *Annual Book of ASTM Standards*, Vol 15.05.

soluble in that solvent.

6. Apparatus

6.1 *Balance or Scale*, weighing to ±0.02 g accuracy.

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

6.3 *Thermometer*, AP style, 0 to 250°C, conforming to Specification E 1.

6.4 *Thermometer*, − 100 to 50°C range.

6.5 *Hot Air Gun*,[6] 260 to 399°C, 120 volts, alternating current (VAC), 60 HZ, 14 A.

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

7. Reagents and Materials

7.1 *Nonvolatile Resins*, (for example, modified hydrocarbon, or rosin ester resins, or both).

7.2 *Solvents*, used in this procedure will be those commonly used for making lithographic ink vehicles, for example, hydrocarbon petroleum distillate C_{12} to C_{16}.

7.3 *Resins and Solvents*, agreed upon between producer and user.

8. Procedure

8.1 Weigh 2.00 ± 0.02 g of pulverized crushed resin (typical size no larger than 6 by 6 mm) and 18.00 ± 0.02 g of the solvent to be tested into a test tube.

8.2 Place test tube in tube clamp with 0 to 250°C thermometer and mixing loop (optional).

8.3 Aim heat gun at bottom of test tube (keep nozzle at least 25.4 mm from tube), and turn on.

8.4 Mix resin and solvent slowly as heat rises.

NOTE 2—To avoid loss of solvent while stirring, do not remove the stirring apparatus from the mixture.

NOTE 3—**Caution:** 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.

8.5 Allow mixture temperature to rise 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.

8.6 Check to see that all resin is dissolved.

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

8.7.1 If the mixture is not clear after heating for 2 min at 200°C, continue to heat until dissolution has occurred. Start the cooling procedure at this point.

8.7.2 If the mixture is clear except for a very slight presence of precipitate, continue to end point and note the presence of "slight precipitate".

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

8.8 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 is called the precipitation temperature (see Note 5 and Note 6).

NOTE 5—Cooling water, ice water, or a dry ice/acetone bath and a low temperature thermometer may be needed to reach the precipitation temperature for very soluble resins.

NOTE 6—This test method is recommended for resins and solvent mixtures exhibiting a precipitation temperature between 50°C 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.

9. Evaluation

9.1 The degree of resin compatibility in a particular solvent is determined by the precipitation temperature. The higher the precipitation temperature, generally, the less compatible the resin and solvent mix is at ambient temperatures.

9.2 Resins and solvent mixes exhibiting precipitation temperatures well above 25°C are not considered highly compatible or highly soluble at ambient temperatures.

9.3 Resins and solvent mixes exhibiting precipitation temperatures below 25°C are considered very compatible and soluble at ambient temperatures.

9.4 It should be noted that many resins exhibiting precipitation temperatures above 25°C at the 10 % resin solids level form compatible resin solutions at higher resin solids. There is a concern, however, that upon dilution with more ink solvent these resins will precipitate.

10. Report

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

10.1.1 Sample identification number,

10.1.2 Test run number,

10.1.3 Maximum temperature achieved,

10.1.4 Approximate rate of cooling, and

10.1.5 Precipitation temperature.

11. Precision and Bias [7]

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

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

11.3 Practice E 180 was used in developing these precision statements.

11.4 *Laboratory Precision (Within-Laboratory, formerly called Repeatability)*—The standard deviation of results obtained by the same analyst running the different analyses has been estimated to be 0.8° absolute at 20 df at the 60°C level and 2.4° absolute at 20 df at the 160°C level. The 95 % limit

[6] The sole source of supply of the hot air gun, Model HG-501A known to the committee at this time is Master Appliance Corp., Racine, WI 53403. If you are aware of alternative suppliers, please provide this information to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[7] Supporting data are available from ASTM Headquarters. Request RR:D01-1103.

for the difference between two such averages is 2.1° absolute and 6.7° absolute, respectively.

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

11.6 *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.

12. Keywords

12.1 cloud point; compatibility; precipitation temperature