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Standard Practices for Preparation of Oil-Based Ink Resin Solutions¹

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1. Scope *

1.1 These practices describe laboratory procedures for preparing an oil-based ink resin solution in a high-boiling solvent using four pieces of lab equipment; (1) a hot oil bath (Sections 4 to 11),

(2) a stirrer/hot plate (Sections 12 to 16),

(3) an industrial blender (Sections 17 to 22), and

(4) a hot air gun (Sections 23 to 27).

ASTM Subcommittee D01.37 recommends using the hot oil bath procedure (Practice D 5597) where possible.

1.2 These practices use laboratory equipment generally available in a normal, well-equipped laboratory.

1.3 One or several of these practices allows for rapid resin solution preparation (under 30 min, typical), can regulate the maximum temperature, can be done under an inert atmosphere, and can prevent the random solvent loss during preparation.

1.4 These procedures are for use with ink resins intended mainly for oil-based offset and letterpress inks. The type of resins are typically, but not limited to C₉ 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.

1.5 The typical high boiling solvents to be used include C₁₂ to C₁₆ petroleum distillates, 2,2,4 trimethyl 1,3-pentanediol di-isobutyrate,² alkali refined linseed oil, tridecyl alcohol, or combinations of the above.

1.6 To avoid fire or injury, or both, to the operator, these practices 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.

¹ These practices are 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 The sole source of supply of the plasticizer TXIB known to the committee at this time is Eastman Chemical Company, / Texas E. M. Division, P.O. Box 7444, Longview, TX 75607-7444. 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.

NOTE 1—**Precaution:** Users of this practice 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. Take safety precautions since there is the potential for vapor ignition. Do the methods outlined in a shielded exhaust hood, where there is access to a fire extinguisher if needed.

1.7 *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 statement see Note 6.

2. Referenced Documents

2.1 ASTM Standards:

D 56 Test Method for Flash Point by Tag Closed Cup Tester³

D 1310 Test Method for Flash Point and Fire Point of Liquids by the Tag Open-Cup Apparatus⁴

D 1725 Test Method for Viscosity of Resin Solutions⁵

D 5062 Test Method for Resin Solution Dilutability by Volumetric/Gravimetric Determination⁵

D 5597 Practice for Preparation of Oil-Based Ink Resin Solutions Using a Hot Oil Bath⁶

E 1 Specification for ASTM Thermometers⁷

E 230 Specification for Temperature-Electromotive Force (EMF) Tables for Standardized Thermocouples⁷

3. Terminology

3.1 Definitions:

3.1.1 *cold cut*—dispersion of resin into solvent using high shear dispersion without external heating.

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

3.1.3 *dissolution*—the point at which all resin completely dissolves in the solvent.

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

³ *Annual Book of ASTM Standards*, Vol 05.01.

⁴ *Annual Book of ASTM Standards*, Vol 06.01.

⁵ *Annual Book of ASTM Standards*, Vol 06.03.

⁶ Discontinued; see 1998 *Annual Book of ASTM Standards*, Vol 06.03.

⁷ *Annual Book of ASTM Standards*, Vol 14.03.

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

3.1.5 *oil bath*—non-volatile, silicone fluid contained in a large heat resistant crystallizing dish heated by a temperature controlled stirrer hot-plate.

3.1.6 *solution*—resin and solvent form a clear, compatible, and homogeneous mixture.

3.1.6.1 *Discussion*—Industrial practice may use the term “solution” loosely to describe what may actually be a clear “dispersion”. For the lack of simplification, the terms solution and dispersion have been used interchangeably in this practice.

HOT OIL BATH

4. Summary of Hot Oil Bath Practice

4.1 Place the required amount of resin and solvent in a 250-mL Erlenmeyer flask.

4.2 A hot oil bath is heated to the required dissolution temperature (150 to 200°C, typically about 180°C or slightly higher for high softening point or poorly solvated resins).

4.3 The Erlenmeyer flask containing the mixture of resin and solvent is placed into the hot oil bath with inert gas purge and a cold water condenser.

4.4 Allow the mixture to mix at the desired temperature until all of the resin is completely dissolved.

4.5 Remove the flask from the hot oil bath and allow it to cool while still under an inert atmosphere for 10 to 15 min. Save the sample for future testing.

5. Significance and Use

5.1 These practices provide means of preparing small quantities of resin solution (in some procedures in an inert gas atmosphere using uniform, controlled heating).

5.2 This practice provides quick ways to prepare a resin solution for quality control testing during the manufacture of resin solutions and vehicles. Samples can usually be prepared in approximately 30 to 45 minutes or less.

5.3 These practices can be used to prepare commonly specified ink test solutions such as 33.3 % resin in alkali refined linseed oil, and 50 % resin in heat-set ink solvent (that is, C₁₂ to C₁₆ hydrocarbon petroleum distillate with initial boiling point (IBP) about 470°F).

6. Apparatus

6.1 *Balance*, capable of weighing to ±0.01 g accuracy.

6.2 *Sieve*, 16-mesh.

6.3 *Thermometer* (see Specification E 1) or *Thermocouple* (see Specification E 230), AP style with a range of 0 to 250°C.

6.4 *Heat Resistant Crystallizing Dish*, 150 by 75 mm in size.

6.5 *Stirrer/Hot Plate*, with a range of 38 to 371°C.

6.6 *Condenser*, with ground glass joints.

6.7 *Erlenmeyer Flask*, 250-mL with 24/40 joint top and side arm.

6.8 *Silicone Oil*.

6.9 *Auxiliary Equipment*, (that is, a 76-mm stir bar, lab jack, lab stand, flask clamp, glass bubbler filled with mineral oil, inert gas source, etc.).

6.10 *Assembly of Hot Oil Bath Set-Up*— Place a stirrer/hot plate in an aluminum tray on a lab jack. Put the crystallization dish filled approximately 2/3 with silicone oil on top of the hot

plate. Arrange the condenser above the center of the bath. Clamp the Erlenmeyer flask containing the solution ingredients on to the condenser. Adjust the flow of nitrogen to flow down the condenser into the Erlenmeyer flask. Lower the flask into the oil bath.

7. Reagents

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

8. Reagents and Materials

8.1 *Nonvolatile Resins*, (for example, hydrocarbon resins, rosin ester resins).

8.2 *Solvents*, used in this procedure will be those most often used in the manufacture of lithographic ink vehicles, for example, alkali refined linseed oil (ARLO), hydrocarbon petroleum distillate C₁₂ to C₁₆.

8.3 The resins and solvents agreed upon between producer and user.

8.4 *Standard Ink Oils*.⁸

9. Procedure

9.1 Set the hot oil bath to heat at the specified temperature. Set the temperature, if possible, at 10°C above the softening point of the resin, but below the initial boiling point of the solvent. (180°C is a common starting temperature for many high-melting-point ink resins.)

9.2 Crush large size pieces of resin sample and pass the crushed resin through a 16-mesh sieve.

9.3 Weigh to the nearest 0.02 g, an appropriate amount of the screened resin into a 250-mL Erlenmeyer flask to meet the concentration requirements for preparation of a 30 to 100-g sample. Typically 100 g of solution is prepared.

9.3.1 —Examples of common ink resin solutions are as follows:

Solution No. 1	Percent	Solution No. 2	Percent
resin	33.3	resin	50
alkali refined linseed oil	<u>66.7</u>	470°F IBP ink oil	<u>50</u>
	100.0		100

9.3.2 High viscosity, high molecular weight, (“structured” or “self gelling”) resins may require a stronger solvent system. Possible resin solutions for use with these resins are as follows:

Solution No. 3	Percent
resin	45
TXIB ²	30
243°C (470°F) IBP ink oil	<u>25</u>
	100.0
Solution No. 4	Percent
resin	50
TXIB	<u>50</u>
	100.0

⁸ Suitable standard ink oils are available from several suppliers. Please call the National Association of Printing Ink Manufacturers, Inc., (NAPIM) at Heights Plaza: 777 Terrace Ave., Hasbrouck Heights, NJ 07604; Phone: (201) 288-9454 for assistance.

9.4 Weigh concentration of solvent needed to the nearest 0.1 g.

9.5 Place flask containing resin mixture into ground glass fitting on water-cooled condenser, secure flask with clamp, jack up hot oil bath under flask until the bottom of the flask is close enough to the bottom of the bath (but not touching the bottom) for the stir bar to mix efficiently. Maintain inert gas flow over the resin-solvent mixture at approximately 1 bubble per 5 s through the outlet mineral oil bubbler. If lab jack not available, lower flask manually.

9.6 Allow the mixture to continue mixing until all resin is dissolved.

9.7 Check to see that all resin is dissolved.

9.8 After all the resin is in solution, and if the solution is clear, lower the hot oil bath and allow the solution to cool under the inert gas atmosphere.

10. Evaluation

10.1 During solution preparation, observe the dissolution of resin and, if desired, record the time and temperature at which dissolution occurred or the maximum temperature at which the mixture was heated if the resin did not dissolve.

10.2 Upon cooling, samples can be tested for viscosity following Test Method D 1725, dilutability following Test Method D 5062, color, etc.

11. Report

11.1 Report on solution preparation the following information:

- 11.1.1 Dissolution time and temperature,
- 11.1.2 Solution clarity,
- 11.1.3 Failure of resin dissolution, if necessary, and
- 11.1.4 Maximum temperature at which resin failed to dissolve.

STIRRER—HOT PLATE

12. Summary of Stirrer/Hot Plate Practice

12.1 Small samples of ink resin and aliphatic ink oil or ink resin and alkali refined linseed oil (ARLO) are cut into dispersion in an Erlenmeyer flask to a specific temperature, at a specified rate, with stirring.

12.2 The resulting fluid dispersion can be used to measure parameters such as viscosity and aliphatic solubility or compatibility of a printing ink resin.

13. Apparatus

13.1 *Erlenmeyer Flask*, 125-mL, fitting the following description: a height of 114 mL, an outside base diameter of 67 mL, and an opening of 27 mL.

13.2 *Magnetic Stirring Bar*, polytetrafluoroethylene-coated, and 25 mm in length.

13.3 *Thermometer*, No. 42°C, conforming to Specification E 1, or other accurate temperature measuring device capable of measuring to 220°C or greater, in 1°C increments.

13.4 *Cork Stopper*, high quality, designed to fit the flask used. This cork is then bored out appropriately to receive the thermometer in 13.3 in a snug fashion. The hole should be drilled at an angle of approximately 25° so the tip of the

thermometer comes to rest at the inside edge of the flask. Place a small groove on the side of the cork to prevent pressure build-up.

13.5 *Hot Plate Stirrer*, capable of a surface temperature of 300°C.

13.6 *Stop Watch*.

14. Calibration and Standardization

14.1 The setting of the hot plate surface temperature must be calibrated by making a blank run in the following manner.

14.2 Determine the total mass of the intended solution described in 12.1 (Note: the mass should be between 30 and 45 g). Weigh into the 125-mL Erlenmeyer flask a quantity of ARLO equal to the intended solution mass described in 12.2. Next, add the stirring bar and affix the thermometer/cork assembly described in 13.4 to the Erlenmeyer flask.

14.3 Turn on the hot plate temperature controller to a setting that will give a surface temperature of approximately 300°C. Allow the hot plate 10 min to heat up and equilibrate.

14.4 Set the flask on the preheated hot plate stirrer and begin stirring.

14.5 Start the stop watch.

14.6 Measure the time required for the ARLO to reach a temperature of 215°C.

14.7 The hot plate surface temperature is correct when the ARLO heats from room temperature to 215°C in 11 min \pm 15 s. On a hot plate, this is usually at a setting between 5 and 6 on the temperature controller dial.

15. Procedure

15.1 Crush large size pieces of resin sample and pass the crushed resin through a 16-mesh sieve.

15.2 Weigh to ± 0.02 g into the Erlenmeyer flask, the ink resin and solvent at the ratio agreed upon between producer and user. Typical resin solutions are noted in 9.3.1. The total mass of ink resin solids and solvent should be between 30 and 45 g.

15.3 Carefully place the stirring bar into the flask to avoid splashing the solvent.

NOTE 2—It is not recommended that the stirring bar be added to the tared flask while on an electronic balance. The magnetic field associated with the stirring bar can affect weighing accuracies.

15.4 Affix the thermometer/cork assembly in the mouth of the flask. Adjust the thermometer tip so it is just off the bottom surface of the flask (1 mm).

15.5 Place the flask on the hot plate stirrer that has been heated 10 min to the calibrated setting derived in Section 14.

15.6 Without stirring, let the ink resin-solvent slurry heat to 100°C.

15.7 When the temperature reaches 100°C, begin stirring the mixture. Keep the stir rate slow at first (so that resin is moving in the solvent but does not splash resin up above the solvent level). For more efficient dissolution, mixing speed is gradually increased as the resin softens. Avoid over-stirring that causes ink resin solids to splash up on the sides of the flask.

15.8 As the ink resin melts, increase the stir rate until a definite vortex is established.