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Standard Practice for Preparation of Oil-Based Ink Resin Solutions Using a Hot Oil Bath¹

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1. Scope

1.1 This practice covers a laboratory procedure for preparing an oil-based ink resin solution in a high boiling solvent using a hot oil bath.

1.2 This practice uses laboratory equipment generally available in a normal, well equipped laboratory.

1.3 This practice allows rapid resin solution preparation (typically 30 to 45 min) and regulates the maximum temperature under an inert atmosphere, preventing the random solvent loss during preparation.

1.4 The procedure is for use with ink resins intended mainly for oil-based offset and letterpress inks. The types of resins are typically, but not limited to, C₉ aromatic hydrocarbon resins, modified dicyclopentadiene resins, rosin pentaerythritol or glycerin 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–C₁₆ petroleum distillates, texanol isobutyrate, alkali-refined linseed oil, tridecyl alcohol, or combinations of the above.

1.6 To avoid fire or injury to the operator, or both, this procedure 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.

1.7 *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.*

2. Referenced Documents

2.1 ASTM Standards:

- D 56 Test Method for Flash Point by Tag Closed Tester²
- E 1 Specification for ASTM Thermometers³

3. Terminology

3.1 Definitions:

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

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

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

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

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

4. Summary of Practice

4.1 The required concentration of resin and solvent is placed in a 250-mL Erlenmeyer flask.

4.2 A hot oil bath is heated to the required dissolution temperature (150 to 200°C, typically approximately 180°C or slightly above 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 an inert gas purge and a cold water condenser.

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

4.5 The flask is removed from the hot oil bath and allowed to cool while still under an inert atmosphere for 10 to 15 min. The sample is saved for future testing.

5. Significance and Use

5.1 This practice provides a means of preparing small quantities of resin solution in an inert gas atmosphere using uniform, controlled heating.

5.2 This practice provides a rapid method of preparing 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 min.

5.3 This practice can be used for preparing 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–C₁₆ hydrocarbon petroleum distillate with initial boiling point (I.B.P.) of approximately 243°C (470°F)).

6. Apparatus

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

6.2 *Sieve*, 16 mesh.

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

6.4 *Crystallizing Dish*, heat resistant, 150 by 75 mm.

¹ This practice 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 14.03.