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.

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Standard Practices for Preparation of Oil-Based Ink Resin Solutions[1]

This standard is issued under the fixed designation D5958; 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 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 D5597) 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 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.

1.5 The typical high boiling solvents to be used include C_{12} to C_{16} 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 D56. (**Warning—**Users of this practice 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. 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 The values stated in SI units are to be regarded as standard. The values given in parentheses are for information only.

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.* For specific hazard statement see 25.11.

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:*[3]

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
D1725 Practice for Preparing Resin Solutions for Viscosity Measurement by Bubble Time Method
D5062 Test Method for Resin Solution Dilutability by Volumetric/Gravimetric Determination
D5597 Practice for Preparation of Oil-Based Ink Resin Solutions Using a Hot Oil Bath (Withdrawn 1999)[4]
E1 Specification for ASTM Liquid-in-Glass Thermometers
E230 Specification for Temperature-Electromotive Force (emf) Tables for Standardized Thermocouples

[1] These practices 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.

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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 International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee,[1] which you may attend.

[3] 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.

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

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3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

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

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

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

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

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

3.1.6 *solution, n*—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 sake 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, C_{12} to C_{16} 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,* capable of reading 0 to 250°C and conforming to Specification E1. Alternately, temperature measuring devices such as liquid-in-glass thermometers, thermistors, thermocouples, or platinum resistance thermometers that provide equivalent or better accuracy and precision, that cover the temperature range specified, may be used.

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 ⅔ 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 C_{12} to C_{16} and 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 C_{12} to C_{16}.

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

8.4 *Standard Ink Oils.* [5]

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.

[5] The use of ink industry recognized standard test oils (petroleum distillates) is recommended for evaluating resins. The test oils are closely controlled from lot to lot to ensure consistent data. Sources and ordering information are available at www.napim.org/testmethods/standardstest.aspx.

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	66.7	470°F IBP ink oil	50
	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	25
	100.0

Solution No. 4	Percent
resin	50
TXIB	50
	100.0

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 D1725, dilutability following Test Method D5062, 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,* capable of reading 0 to 250°C and conforming to Specification E1. Alternately, temperature measuring devices such as liquid-in-glass thermometers, thermistors, thermocouples, or platinum resistance thermometers that provide equivalent or better accuracy and precision, that cover the temperature range specified, may be used.

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 ±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 1—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.

15.9 Heat the ink resin/solvent blend to the maximum temperature. The following maximum temperature guidelines are recommended.

If the solvent is:	Heat to (°C):
100 % ARLO	215
100 % hydrocarbon petroleum distillate	190
Aliphatic Ink oil/ARLO blends	190
ARLO/Aliphatic Ink oil/tridecyl alcohol (TDA) blends	190

NOTE 2—Maximum temperatures other than these may be agreed upon between producer and user.

15.10 As soon as the top temperature is reached, remove the flask and place it on the lab bench top.

15.10.1 After the flask has cooled below 150°C, lift carefully and inspect all inside edges to ensure the ink resin is totally dispersed into the solvent.

15.10.2 If all the ink resin is not dispersed, repeat 15.9 and 15.10 until all resin is dispersed.

15.10.3 Once all the ink resin has dispersed, allow the contents to cool below 100°C before opening the flask and performing any analysis on the dispersion agreed upon between producer and user.

NOTE 3—It is recommended that any quality control testing done on the dispersion should be performed just after making the solution. Long term storage of dispersions of this type can affect the rheological properties.

16. Precision and Bias

16.1 *Precision*—An interlaboratory study was conducted in which one operator, in each of seven laboratories, determined the viscosity of an aliphatically-soluble phenolic-modified rosin ester dispersed at 40 % solids in a hydrotreated C_{12} to C_{16} aliphatic petroleum distillate. Using this practice to make the solution, the operators then measured the viscosity of the dispersion in accordance with Test Method D1725.

16.2 Each operator ran duplicate samples on Day 1 and Day 2. The overall average viscosity of the solution was 15.89 s.

16.3 The pooled within-laboratory standard deviation was 0.20 s with 6 df. The between laboratory standard deviation was 1.35 s with 27 df.

16.4 Based on these results, the following criteria should be used for judging the acceptability of results at the 95 % confidence level.

16.4.1 *Repeatability*—Two results, each the mean of duplicate determinations obtained by the same operator, should be considered suspect if they differ more than 2.3 % of the measured value.

16.4.2 *Reproducibility*—Two results, each the mean of duplicate determinations obtained by operators in different laboratories, should be considered suspect if they differ more than 13.2 % of the value measured.

16.5 *Bias*—The information supplied is intended to reflect the accuracy of this practice under these specific circumstances only. Different resins dispersed in different solvents may give different precision values.

BLENDER

17. Summary of Blender Practice

17.1 Place required amount of resin and solvent in a blender jar.

17.2 Mix the resin-solvent mixture at high speed in a blender until heat is developed by the high shear and the resin is dissolved into solution.

17.2.1 Suggest mixing for 15 min at highest speed.

17.3 Remove blender jar from blender and pour the solution through a paint filter into a container for storage and future testing.

18. Significance and Use of Blender Practice

18.1 This practice provides a means of preparing resin solutions by the "cold cut" method, modelling high-shear production dispersion techniques.

19. Apparatus

19.1 *Balance* or *Scale*, weighing to ±0.1 g accuracy.

19.2 *Blender,* with one quart vessel, 115 alternating current volts (VAC), 60 Hz, 840 W.

19.3 *Dial Thermometer,* bi-metal 44 mm 0 to 220°C.

19.4 *Medium Mesh Paint Filter.*

19.5 *Auxiliary Equipment* (that is, aluminum foil, paper towels, lab filter stand, etc.).

20. Procedure

20.1 Weigh required mass of solution solvent into blender vessel.

20.2 Weigh flaked or crushed resin (typical size no larger than 0.6 cm^2 to the nearest 0.1 g to meet concentration requirements into some container (paper cups, aluminum pan, etc.).

20.3 Place the blender vessel on blender and start mixing action on low speed.

20.4 Add resin slowly into blender vessel and increase mixing speed, as mixture viscosity thickens, until all resin is added.

20.5 Insulate blender jar with paper towels wrapped in aluminum foil (optional).

20.6 Continue mixing for 15 min after all resin is added or for conditions agreed upon between producer and user. If possible, allow mixture temperature to rise to approximately 10°C above the melting point of the test resin (as long as this temperature is below the boiling point of the solvent).

20.6.1 If not all resin is dissolved or if the mixture is not clear, continue mixing until dissolution has occurred.

20.6.2 If the mixture does not become clear the mixture is incompatible.

20.7 After all resin has dissolved, remove the blender jar from the blender and pour the solution through the paint filter into a container for storage.

20.8 Cover sample and save for future testing.

21. Evaluation

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

21.2 Upon cooling, samples can be tested for viscosity, solvent tolerance or dilutability, color, etc.

22. Report

22.1 Report on solution preparation the following information:

22.1.1 Dissolution temperature,

22.1.2 Solution clarity,

22.1.3 Failure of resin dissolution, if necessary, and

22.1.4 Maximum temperature at which resin failed to dissolve.

HOT AIR GUN

23. Summary of Hot Air Gun Practice

23.1 Place required concentration of resin and solvent in a heat-resistant test tube.

23.2 Heat the test tube containing the mixture of resin and solvent with a hot air gun. Stir the mixture well during the heating process.

23.3 Remove heat when all resin is dissolved and allow sample to cool in the test tube. Cover and save sample for future testing.

24. Apparatus

24.1 *Balance or Scale,* weighing to ±0.01 g accuracy.

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

24.3 *Thermometer,* capable of reading 0 to 250°C and conforming to Specification E1. Alternately, temperature measuring devices such as liquid-in-glass thermometers, thermistors, thermocouples, or platinum resistance thermometers that provide equivalent or better accuracy and precision, that cover the temperature range specified, may be used.

24.4 *Master Heat Gun,* 260 to 399°C, 120 VAC, 60 Hz, 14 A.

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

25. Procedure

25.1 Place test tube in a beaker for support and tare on scale.

25.2 Weigh flaked or crushed resin (typical size no larger than 6 by 6 mm) to the nearest 0.1 g to meet concentration requirements to prepare a 30-g sample.

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

25.4 Place test tube in tube clamp with 0 to 250°C thermometer and mixing loop (optional) immersed into the mixture.

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

25.6 Mix resin and solvent slowly as heat rises.

25.7 Allow mixture temperature to rise to approximately 10°C above the melting point of the test resin (as long as this temperature is below the boiling point of the solvent).

25.8 Check to see that all resin is dissolved.

25.8.1 If the mixture is not clear, continue to heat until dissolution has occurred.

25.8.2 If the mixture does not become clear or exhibits significant precipitation, record it as incompatible at the temperature attained.

25.9 After all the resin is in solution turn off hot air gun and allow sample to cool.

25.10 Replace lost solvent if needed and stir until uniform.

25.11 Cover sample and save for future testing. (**Warning—**This method is safe when care is taken during stirring the resin/solvent mixture not to drop the thermometer or stirring loop. However, there is the potential, if the hot air gun is not positioned properly, that a break in the test tube could ignite the solution in the hot air gun.)

NOTE 4—To help prevent the loss of solvent while stirring, do not remove the stirring apparatus from the mixture during the heating cycle.

NOTE 5—A maximum heating time of 15 min at top temperature or a

maximum heating time as may be agreed upon between producer and user is recommended.

26. Evaluation

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

26.2 Upon cooling, samples can be tested for viscosity, solvent tolerance or dilutability, color, etc.

27. Report

27.1 Report on solution preparation the following information:

27.1.1 Dissolution temperature,

27.1.2 Solution clarity,

27.1.3 Failure of resin dissolution if necessary,

27.1.4 Maximum temperature at which resin failed to dissolve, and

27.1.5 Precipitation temperature upon cooling.

28. Keywords

28.1 cold cut; compatibility; dissolution; incompatible; precipitation temperature; solution

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