

Designation: D6989 - 03 (Reapproved 2020)

# Standard Practices for Preparation of Solvent and Water Based Ink Resin Solutions<sup>1</sup>

This standard is issued under the fixed designation D6989; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

#### 1. Scope

1.1 These practices describe laboratory procedures for preparing a solvent or water based ink resin solution in low boiling solvent or alkaline water using two types of lab equipment; (1) an industrial blender (Sections 3 - 7), and (2) a laboratory roller mill (Sections 8 - 12).

Note 1—ASTM Subcommittee D01.37 recommends using the industrial blender where possible.

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

1.3 These procedures are for use with ink resins intended mainly for liquid (for example, flexographic and rotogravure) inks. The type of resins is typically, but not limited to, acrylic and styrene/acrylic copolymers, polyamides, polyesters, polyvinylbutyral, and maleated/fumarated rosin esters.

1.4 The typical low boiling solvents to be used include ethanol, isopropanol, *n*-propanol, ethyl acetate, isopropyl acetate, and *n*-propyl acetate. For water based ink resin solutions, water is used in combination with ammonium hydroxide or amines such as dimethylethanolamine, monoethanolamine, and triethylamine.

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

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

1.7 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. Terminology

#### 2.1 Definitions:

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

2.1.2 *compatibility, n*—the ability of two or more differing substances to be mixed together without resultant kick-out or haziness.

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

2.1.4 *incompatibility*, *n*—when a mixture of two or more differing substances results in precipitation, kick-out, or haziness.

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

2.1.5.1 *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.

#### **BLENDER**

# 3. Summary of Blender Practice m-d6989-032020

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

3.2 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.

3.3 The resulting resin solution can be used to measure parameters such as viscosity and solubility or compatibility of a printing ink resin.

#### 4. Significance and Use of Blender Practice

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

#### 5. Apparatus

5.1 Balance or Scale, weighing to  $\pm 0.1$  g accuracy.

5.2 *Blender*, with one quart vessel and cover, 115 alternating current volts (VAC), 60 Hz, 840 W (preferably explosion proof).

<sup>&</sup>lt;sup>1</sup> 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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5.3 Filter Media (such as organdy cloth).

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

## 6. Sampling

6.1 Solid resin should be flaked or crushed (typically to a size no larger than  $6 \text{ mm}^2$ ).

6.2 The total mass of resin solids and solvent should be approximately 800 g. The exact amount (separately) of resin solids and solvent will vary depending on the target percent solids to be achieved.

# 7. Procedure

7.1 Weigh (separately) solid resin and desired solvent blend (which may consist of a mixture of organic solvents or a combination of water and ammonia/amine) to the nearest 0.1 g to meet concentration requirements. Add the solvent blend to the blender vessel.

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

7.3 Add solid resin slowly into blender vessel and increase mixing speed, as mixture viscosity thickens, until all resin is added. Place cover on blender vessel.

7.4 Insulate blender vessel with paper towels wrapped in aluminum foil (optional).

7.5 Continue mixing for 15 min after all resin is added.

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

7.5.2 If the mixture does not become clear, the mixture is incompatible.

7.6 After all resin has dissolved, remove the vessel from the blender and pour the solution through organdy cloth or other suitable filter media into a container for storage.

7.7 Cover sample and save for future testing.

# LABORATORY ROLLER MILL

#### 8. Summary of Laboratory Roller Mill Practice

8.1 Small samples of ink resin and solvents or alkaline water are cut into solution by rolling a jar or other cylindrical container on an automated laboratory roller mill, typically overnight.

8.2 The resulting resin solution can be used to measure parameters such as viscosity and solubility or compatibility of a printing ink resin.

# 9. Significance and Use of Laboratory Roller Mill Practice

9.1 This practice provides a means of preparing resin solutions without the use of heat or high shear, and will only provide satisfactory results with resins of a suitable solubility. This practice is typically used when the resin solution is not

required for immediate use (that is, the material is weighed and placed on the laboratory roller mill overnight for use the next day).

# **10.** Apparatus

10.1 Balance or Scale, weighing to  $\pm 0.1$  g accuracy.

10.2 Glass Jars, 1 pint or 1 quart size, with metal lids.

10.3 Laboratory Roller Mill.

10.4 Filter Media (such as organdy cloth).

## 11. Sampling

11.1 Solid resin should be flaked or crushed (typically to a size no larger than 6  $\text{mm}^2$ ).

11.2 The total mass of resin solids and solvent should be approximately 400 g for a pint jar and 800 g for a quart jar. The exact amount (separately) of resin solids and solvent will vary depending on the target percent solids to be achieved.

## 12. Procedure

12.1 Weigh required mass of solvent blend (which may consist of a mixture of organic solvents or a combination of water and ammonia/amine) into jar.

12.2 Weigh solid to the nearest 0.1 g to meet concentration requirements into the glass jar.

12.3 Place the lid on the jar and close securely to ensure a tight seal.

12.4 Seal the gap between the glass jar and metal lid by affixing a strip of electrical tape at the lid/jar interface, to further ensure a leak-free operation.

12.5 Label the jar to indicate the contents. Preferably, the jar should be labeled on the lid, as information recorded on the side wall of the jar may be rubbed off during the rolling process.

12.6 Agitate the contents of the jar by shaking vigorously, in order to wet the resin thoroughly with the solvent blend.

12.7 Place the jar on the roller mill.

12.8 Activate the roller mill motor.

12.9 Allow the jar to rotate on the mill until all resin is visibly dissolved (typically for 16 hours or overnight).

12.10 If all ink resin is not dispersed, repeat 12.7 - 12.9.

12.11 After all resin has dissolved, remove the glass jar from the roller mill and pour the solution through organdy cloth or other suitable filter media into a container for storage.

12.12 Cover sample and save for future testing.

# 13. Keywords

13.1 blenders; cold cuts; ink resins; laboratory roller mills; resin solutions