



Standard Practice for Vacuum Distillation of Solvents From Solvent-Reducible Paints For Analysis¹

This standard is issued under the fixed designation D 3272; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 This practice covers a vacuum distillation procedure that separates the solvents from the nonvolatile portion of paints so they may be analyzed.

1.2 An alternative procedure for the analysis of solvents in paints is described in Practice D 3271.

1.3 *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 3271 Practice for Direct Injection of Solvent-Reducible Paints into a Gas Chromatograph for Solvent Analysis²

3. Summary of Practice

3.1 The sample of whole paint with added tricresyl phosphate is heated by means of a silicone oil bath. The distillation is allowed to proceed for a period of time and then vacuum is applied. Finally dry air is passed through the system to facilitate the removal of any high-boiling solvents present.

4. Apparatus

4.1 Apparatus³ is shown in Fig. 1. It is essential in assembling this apparatus, that the volume of the portion preceding the flask be kept to a minimum by using diameters of connections and distances as small as possible. Include a gage in the assembly for measuring pressure.

4.2 *Vacuum Pump*, capable of pulling 2 mm Hg or less of vacuum pressure.

4.3 *Septum Sample Vial*, PTFE-fluorocarbon-faced.⁴

4.4 *Safety Shield*.

5. Reagents

5.1 *Methylene Chloride or Acetone.*

5.2 *Silicone Oil.*⁵

5.3 *Tricresyl Phosphate.*

6. Procedure

6.1 Immerse the receiver in a bath containing dry ice with methylene chloride or acetone of sufficient depth that the liquid reaches to the bottom level of the rubber stopper.

6.2 Pour approximately 15 g of the whole paint sample into the 50-mL distillation flask. Immediately follow with 10 mL of tricresyl phosphate and without delay mix the contents of the flask.

6.3 Assemble the apparatus and close the needle valve and stopcock attached to the capillary tubing. Immerse the distillation flask by raising a silicone oil bath preheated to 155 to 160°C (**Warning**—see Note 1) until it reaches the side arm (Note 2). Allow the distillation to proceed for 15 min without vacuum.

NOTE 1—**Warning:** Care should be taken to prevent the temperature of the silicone oil exceeding 160°C because of possible formation of explosive mixtures of air and solvents from the sample being analyzed.

NOTE 2—The liquid level in the oil bath should be as high as is practical.

6.4 After 15 min apply vacuum slowly and carefully to avoid bumping or rapid distillation until 2 mm Hg is reached. This step should take approximately 10 to 15 min. Next, carefully open the stopcock. After bubbling subsides, adjust the needle valve so as to regulate the flow of air at a rate of 60 to 80 bubbles per minute (at the bubble counter). Pass air through the system for a total of 30 min while keeping the dry ice bath at a minimum low temperature.

6.5 At the end of the 30-min period release the vacuum very slowly with air flowing to prevent back-up of the paint. Remove the receiver containing the solvents and warm the contents only to the temperature of cold running tap water. Transfer the isolated solvents promptly to an airtight septum

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² *Annual Book of ASTM Standards*, Vol 06.01.

³ Obtainable from SGA Scientific, Inc., 735 Broad St., Bloomfield, NJ 07005.

⁴ Septum vials available from Precision Sampling Corp., Baton Rouge, LA 70315, have been found satisfactory for this purpose.

⁵ The sole source of supply of silicone oil 710 known to the committee at this time is Dow Corning Corp., Midland, MI. 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.

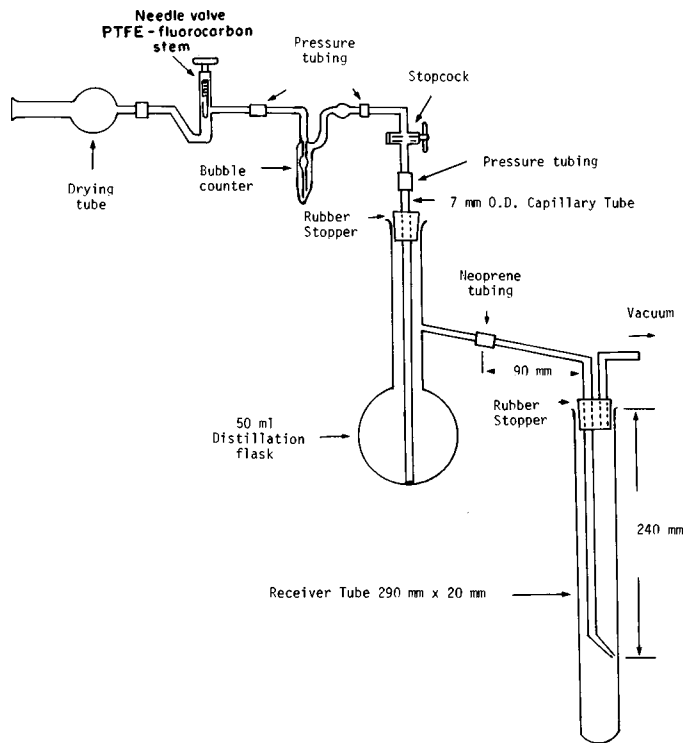


FIG. 1 Distillation Apparatus

faced vial for subsequent analysis.

7. Keywords

7.1 solvents in paints; vacuum distillation

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