SCAQMD METHOD 302-91

DISTILLATION OF SOLVENTS FROM PAINTS, COATINGS AND INKS

1. Principle

1.1 A weighed sample is placed in a vacuum apparatus and distilled by using a vacuum pump while being heated in a silicone oil bath. The pressure and heating are adjusted to control the rate of distillation in order to prevent bumping, clogging of tubes, and loss of low-boiling compounds. The distilled vapor is condensed and retained in a receiver immersed in a liquid nitrogen bath.

2. Apparatus

2.1 Apparatus as illustrated in Figure 1. It is essential to assemble the apparatus so that the system is free of leaks and the glassware is not mechanically stressed.

2.2 The assembly and distillation should occur in a fume hood with safety shield large enough to accommodate the entire apparatus.

2.3 Vacuum pump capable of reducing the pressure to at least 2 mm Hg

2.4 Sample vial, with PTFE-fluorocarbon-faced silicone septum, 15 mL recommended

2.5 Variable transformer for control of immersion heater

2.6 Graduated cylinders, Class A, 50, 25, and 10-mL capacity, glass stoppered

2.7 Funnels, glass

2.8 Balance, top loading, capable of reading to 0.01 g

3. Reagents

3.1 Liquid nitrogen

3.2 Silicone Oil[1]

4. Procedure

Note: All weights are obtained using a top loading balance and are recorded to the nearest 0.01 g.

4.1 Weigh a clean, dry, empty receiver. Record weight in grams, Wr1.

4.2 Connect the receiver to the vacuum distillation apparatus at points A and B of Figure 1. The distillation flask is not connected at this time. With the inlet stopcock closed, evacuate the receiver. Once the receiver is fully evacuated, close the outlet stopcock. Immerse the receiver in the liquid nitrogen cold trap so that the level of the liquid nitrogen is maintained well above the end of the inlet tube during distillation.

4.3 Weigh a clean, dry, empty 1-liter distillation flask with stopper. Record weight in grams, Wf1.

4.4 Pour approximately 50 g of the well-stirred whole paint into the weighed distillation flask, stopper, and weigh. Record weight in grams, Wf2. The difference in weights (Wf2 - Wf1) is the weight of the sample, Wsamp.

Note 1: For water-based paints, it may be necessary to use a smaller sample size to prevent foaming or to facilitate the control of distillation process. Lithographic inks and muck samples may require a larger sample size because of the relatively small amount of distillate many of these samples contain.

4.5 Turn on the heating tape to prevent condensation of the distillate in the glassware leading to the cold trap. Attach the distillation flask as in Figure 1. Partially evacuate the assembly by opening the inlet stopcock.

[1]Silicone oil 710 available from Dow Corning Corp., Midland, Michigan, has been found satisfactory for this purpose.

4.6 Begin rotation and open the inlet and outlet carefully to prevent bumping and escape of sample vapors to the vacuum pump. Allow the system to reach 2 mm Hg before raising the silicon bath to just above the level of the sample in the distillation flask. The vacuum and heat are adjusted to control the distillation rate.

4.7 Distillation is continued until the residue appears dry, or the oil bath reaches 200^{o}C. If charring or rapid evolution of gases occurs, note the temperature and discontinue distillation. Repeat from Section 4.1 and avoid exceeding the noted temperature.

4.8 Unplug the oil bath heating element and allow to cool to 10 degrees below maximum temperature attained by the oil bath.

4.9 Close inlet and outlet of the receiver and lower oil bath.

4.10 Allow the distillation flask to cool, wipe the oil off the distillation flask, and carefully disengage the flask once the distillation flask is cool enough to handle by hand. Stopper and allow to reach room temperature.

4.11 Record weight of distillation flask and contents in grams, Wf3. The difference in weights (Wf3 - Wf1) is the weight of the residue, Wres.

4.12 Lower the liquid nitrogen cold trap and allow to come to room temperature before disconnecting the receiver from distillation apparatus at points A and B (See Figure 1).

4.13 Open the outlet to the receiver to allow contents to come to ambient pressure, then close to prevent evaporation.

4.14 Record final weight of receiver and contents in grams, Wr2. The difference in weight (Wr2 - Wr1) is the weight of the distillate, Wdis.

4.15 Compare the sum of Wres and Wdis to Wsamp. The percent difference should not be greater than 0.5%. If it is greater than 0.5%, the distillation procedure should be repeated. For samples where condensation occurs inside the rotovap sleeve, the percent difference should not exceed 0.8%.

4.16 Record the weight of an appropriate graduated cylinder and its stopper in grams, Wc1.

4.17 Transfer the distillate completely to the weighed graduated cylinder with the aid of a glass funnel.

4.18 Record the volume to the nearest 0.1 mL, Vdis. This is the uncorrected distillate volume.

4.19 Record the weight of the graduated cylinder and contents in grams, Wc2.

5. Calculations

5.1 Residue, % (w/w) = $\frac{Wres}{Wsamp} \times 100$

Where, Wres = Weight of residue, in grams (Sec. 4.11)
Wsamp = Weight of sample that was distilled, in grams (Sec 4.4)

5.2 Density, g/mL = $\frac{Wc2 - Wc1}{Vdis}$

Where, Wc2 = Weight of graduated cylinder and contents, in grams (Sec. 4.19)
Wc1 = Weight of graduated cylinder, in grams (Sec. 4.16)
Vdis = Volume of distillate, in mL (Sec. 4.18)

5.3 Corrected distillate volume = $\frac{Wdis}{D}$

Where, Wdis = Weight of distillate, in grams (Sec. 4.14)
D = Density of distillate, in g/mL (Sec 5.2)

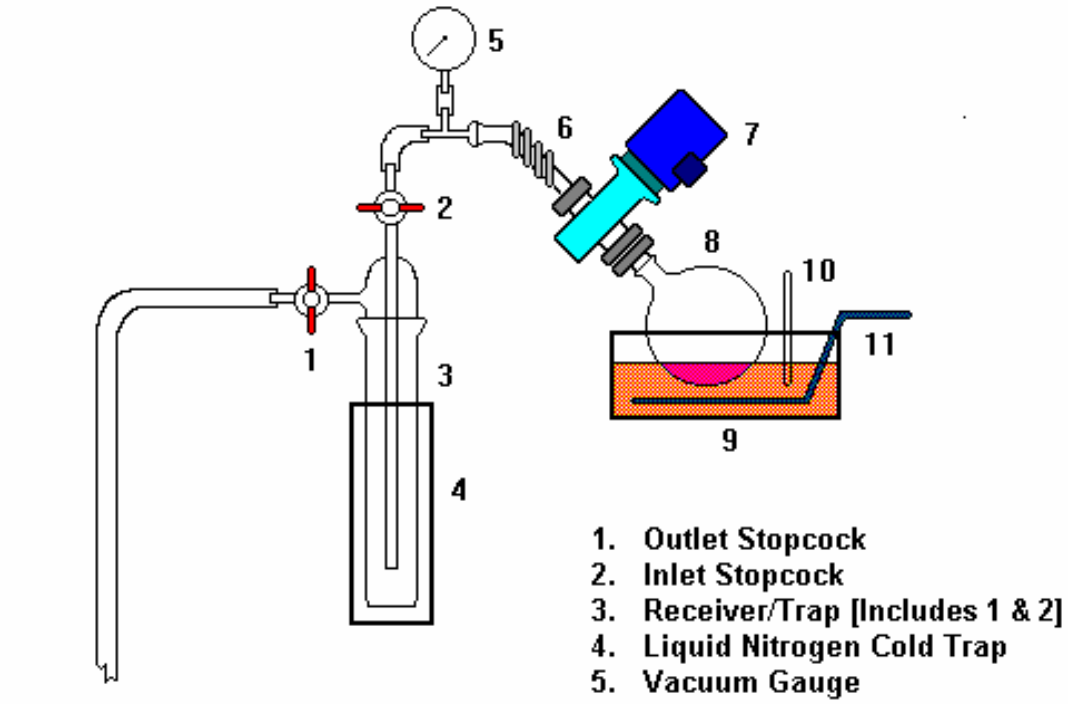
6. References

2.1 ASTM Standard D 3272 Vacuum Distillation of Solvents from Solvent-Based Paints for Analysis[2].

[2]Annual Book of ASTM Standards, Vol. 06.01.

Figure 1

Vacuum Distillation Apparatus



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This method describes a vacuum distillation procedure to separate solvents from the nonvolatile portion of paints, coatings and inks. The resulting distillate is available for the analysis of exempt compounds. This method may be used for other types of samples with minor modifications.

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