

Chemistry 136

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Vacuum Distillation Notes

1. Establishing Reduced Pressure.

Please refer to the attached photocopies of the apparatus. Vacuum distillation is a difficult and important technique. Make sure you understand the effect of vacuum on boiling point (see pressure-temperature nomograph). You should also understand the difference between the water aspirator (~25 torr in Los Angeles), the mechanical pump (~ 1 torr if poorly maintained, as most pumps are likely to be, ~0.01 torr if well maintained), and the diffusion pump (10^{-3} - 10^{-6} torr if properly set up). The most important part of your vacuum pump is the cold trap, which is designed to keep solvent vapors out of the oil. The trap must always be cooled in a Dry Ice/ acetone bath (-78°C) before the pump is operated. On occasion you may wish to use liquid nitrogen to cool the trap. If liquid nitrogen is used, *never leave the trap open to the air*, as oxygen will condense, and can cause explosions with organic condensates in the trap. **IF YOU FIND A BLUE LIQUID IN THE TRAP, IT IS LIQUID OXYGEN. ASK FOR HELP!** As recently as 1990, two German graduate students died in an explosion caused by liquid oxygen condensed in a trap. If acid or noncondensable vapors are being pulled off your vacuum flask, additional chemical traps before the liquid nitrogen trap are required. If you suspect that this is the case, see your TA. Always clean the trap before starting a distillation and before leaving the pump.

2. Achieving Controlled Boiling

In general, boiling chips are not effective for controlling bumping during a vacuum distillation. As an alternative to boiling chips, many researchers use magnetic stirrers to prevent bumping. For solutions of low viscosity, this method is quite effective.

If the material is at all viscous the best method is to use a capillary tube, which introduces tiny bubbles into the system that act as nucleation sites. There are two techniques for making these, depending on whether you are using an aspirator or a vacuum below 1 torr.

a. Aspirator. Use 5-6 mm standard tubing, and pull it into the finest capillary you can. The tube should be rotated in a medium torch flame until it is evenly soft all around for a length of about 1/2 cm., removed from the flame and allowed to cool for about a slow count of three, then pulled steadily and smoothly until a very thin and flexible capillary is formed. Check that the capillary is open by blowing through it into a small test tube with a little ether in it. The bubbles should be very small.

b. Higher vacuum. Use 1 mm i. d., 6-7 mm o.d. thick-wall capillary, and pull as above. You should just barely be able to see the bubbles when you blow through this capillary into ether.

Install the capillary by using a 10/30 standard taper joint which fits into the joint on the vertical arm of the Claisen head of a vacuum distillation apparatus. (Thermometer adaptors with o-rings are an alternative). Put a short length of heavy rubber vacuum hose over the joint, and moisten the inside with a small drop of glycerol. The capillary should be measured visually from outside, and broken to the right length to reach just to the bottom of the flask (pear shaped distillation flasks should be used). The capillary can now be slipped down inside the apparatus, carefully, until it just touches the bottom of the flask. Be prepared to apply the vacuum instantly, because the liquid will enter the capillary, and if it is viscous, may plug it, especially if it is a small one. You will break a few capillaries before you get the hang of this. If the vacuum has to be interrupted for any reason, pull the capillary out of the liquid at once. Use a cow or other fraction cutter to avoid breaking the vacuum.

Those who are really compulsive about avoiding air can put a balloon filled with nitrogen attached to a short length of vacuum tubing over the end of the capillary. If the capillary is well drawn, you won't notice the amount of nitrogen drawn into the distillation flask.