Chapter 14: Solvent Removal

It is often necessary to remove solvent from a solution to recover either a solid or a high-boiling liquid. There are several ways to do this as outlined below.

14.1 Distillation

Simple distillation, explained in Chapter 7, can be used to remove solvent. Distillation works well if the solution is composed of a solid and a low-boiling solvent, or if the solution is composed of a high-boiling liquid and a low-boiling solvent (with boiling point differences greater than 100°). Advantages of distillation are that the solvent can be collected and recycled and that no vapors are released into the atmosphere. A disadvantage is that it can take a long time, and risks scorching your product.

14.2 Open-Dish Evaporation

Solvent can be evaporated by placing the solution in an open container (an Erlenmeyer, evaporating dish, beaker, vial, etc.). The container is set on a heat source (with boiling chips) and the solvent boiled off.

The problem with open-dish evaporation is that the solvent is released into the air. Open-dish evaporation should always be done in a hood if the solvent is anything other than water. Even in a hood, however, vapors are released into *somebody's* air. If the solvent is a hazardous compound (for instance, dichloromethane), it is probably better to choose another method of solvent removal.

If time is not critical, simply set the open container in a hood and allow evaporation at room temperature. If the solution is aqueous, you can leave it in your lab drawer until the next lab period.

In some facilities, compressed air is available. If so, you can use it to blow air across the surface of a solution. Air directed across the solution speeds up evaporation by blowing the vapors out and continually shifting the equilibrium in the direction of evaporation.

14.3 Rotary Evaporation

Rotary evaporators, or rotovaps (Figure 14-1), are standard equipment in most organic chemistry research labs. These evaporators are designed to remove solvent rapidly from solutions without constant monitoring. The motor in the rotovap spins a round-bottom flask rapidly, coating the walls of the flask with the liquid and providing a greater surface from which evaporation can occur. Cooling coils in the rotovap condense the vapors and drop them into a collection flask so that they can be recycled or properly disposed of. The rotovap is connected to a vacuum source, which lowers the ambient pressure and makes the solvent evaporate faster. Usually the flask containing the solution to be evaporated is warmed by a water bath, to further increase the rate of evaporation. The entire core assembly (everything but the bath and the main support) is mounted on a track, so that it can be moved up or down to position the round-bottom flask correctly in the bath, and so that you can safely attach your flask without getting bath water in it.

It is often the case that a sample being rotovapped will "bump", or send liquid upwards into the device. To prevent this, a bump trap is normally used. This blocks liquid from spouting directly upwards into the machine, but allows vapors to flow freely. Another necessary piece of glassware is an adapter, since all the

round-bottom flasks used in the organic labs have 14/20-sized joints, but the glass parts of the rotovap use larger 24/40 joints (see Chapter 5).

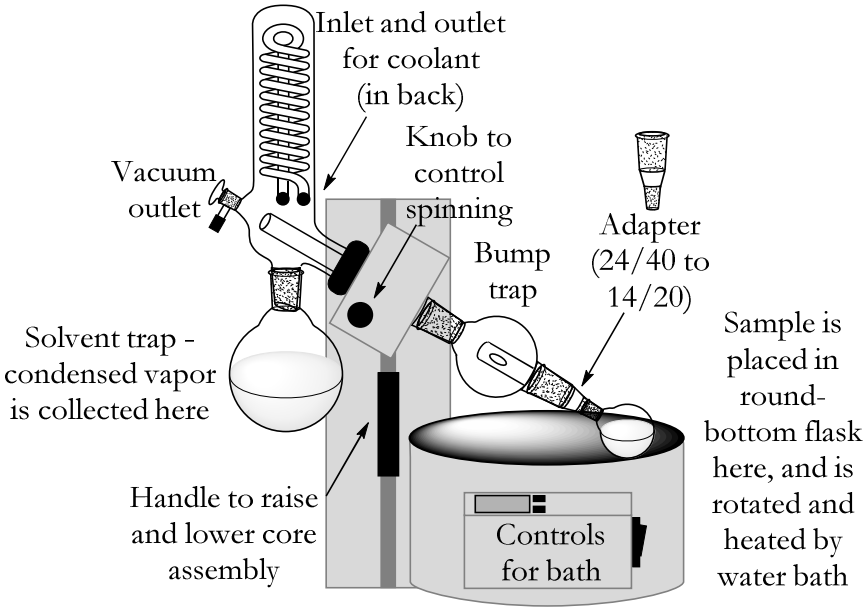

Figure 14-1: The general layout of a rotary evaporator, or rotovap.

A. Steps for Performing Rotary Evaporation

1. Prepare your sample.

Choose a round-bottom flask that holds at least twice as much volume as your sample. If you intend to weigh the sample after the solvent has been removed, you should record the weight of the empty flask first, and then weigh it again after you have finished rotovapping. Place the solution to be evaporated in the flask.

2. Prepare the rotovap.

Using the handle, raise up the core part of the rotovap assembly. If the solvent trap is more than half-full, detach it and empty it into the organic waste. Check that the rotovap's bump trap and adapter are clean.

If the sample will become a free-flowing powder after the solvent is removed (for instance, if you are rotovapping a mixture of compound and silica that you intend to put into a large-scale chromatography column), you should prevent this solid from being blown up into the bump trap. You can easily do this by stuffing a Kimwipe into the adapter and packing it in with a spatula.

Make sure that cooling fluid is flowing through the coils. You can check by feeling the hoses that connect to this part of the rotovap – they should feel cold to the touch.

Using the switch on the right side of the bath controls, turn on the bath heating. You can adjust the bath temperature up and down using the arrow buttons. A good default bath temperature for most solvents is 60°C.

3. Begin rotovapping.

Attach your flask to the adapter with a yellow Keck clip. Using the handle, lower the core assembly so that the round-bottom flask is halfway submerged in the water. None of the glass parts – the bump trap, adapter, or round-bottom flask – should be in contact with any of the solid parts of the bath. You may need to top up the water in the bath to achieve this.

Begin spinning the flask by adjusting the rotation control knob. Close the vacuum outlet slowly. If your solvent begins boiling too rapidly, you can partially open the vacuum outlet to control the pressure inside the rotovap.

If several minutes have elapsed and no solvent appears to be evaporating from your sample, you may need to increase the bath temperature. Perhaps the vacuum is not as strong as it should be – do any of the neighboring rotovaps have their vacuum outlet left open to atmosphere? If so, you will need to close them. Do you hear any hissing from your rotovap? It may not be sealed against atmosphere, and may need adjustments.

4. Remove your sample and clean up.

When all the solvent has been removed from your sample, perform the above steps in reverse order. First, close the vacuum outlet and stop spinning the flask. Raise up the assembly and remove your flask.

If any of your sample has bumped into the adapter or bump trap, give these items a quick rinse with acetone, then return them to the rotovap. Again check whether the solvent trap is more than half full, and if so, empty it into the organic waste. If nobody will use the rotovap after you, turn off the bath heating.

Frequently, if your compound is a solid, it will be coated onto the walls of the round-bottom flask in a thin layer. If you have trouble removing it from the flask, you may be able to bend one end of a spatula into a J-shape that can reach the upper areas of the flask.