

## The Rotary Evaporator

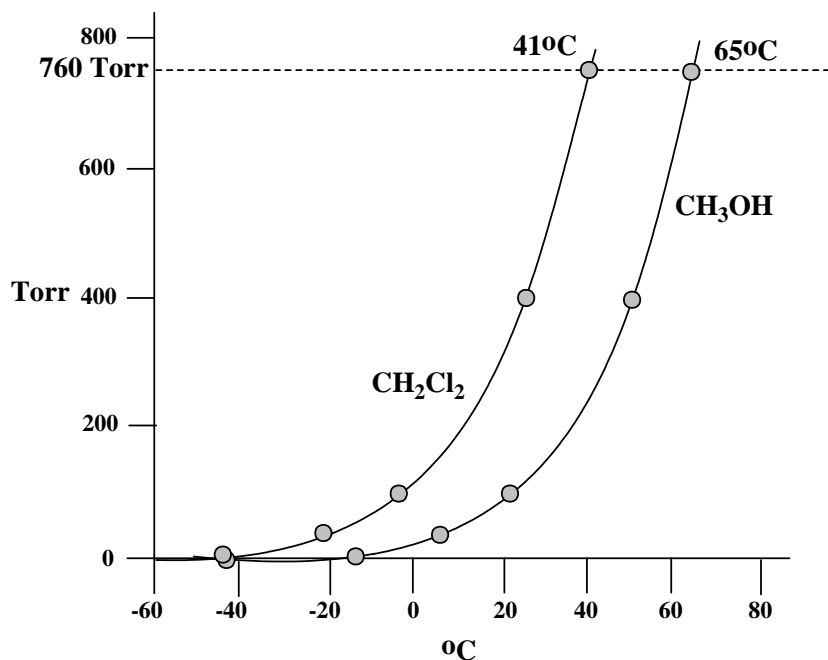
It is commonly taught in General Chemistry classes that the vapor pressure of a liquid or solid varies directly with temperature, i.e., the higher the temperature of a substance, the higher its vapor pressure. When a liquid is heated it begins to boil as the liquid turns to vapor forming bubbles that literally "push away" the ambient atmosphere above the liquid surface. "Boiling" has occurred. The vapor pressure of the liquid became **equal to** the ambient pressure, counteracting it or pushing it away. We say the liquid is **boiling**.



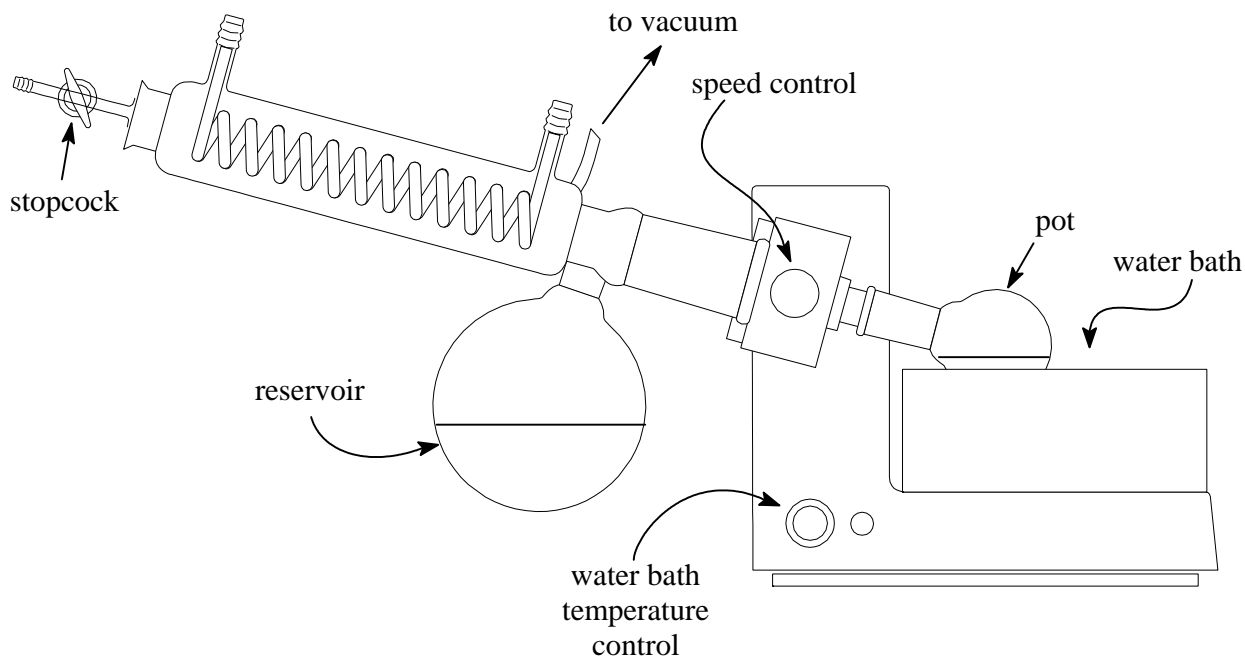
Precise measurements of the temperature when a liquid boils may include other ambient pressures. The **normal** boiling point is that temperature where the vapor pressure of the liquid equals a standard atmosphere or 760 torr. Of course, as the pressure above a liquid at room temperature is steadily decreased, by, say, an aspirator pump, the liquid boiling point will also decrease, so much so that it will "boil" at room temperature, since that is where its vapor pressure equals the ambient pressure. You can consult books like the CRC Handbook of Chemistry and Physics for lists of organic compounds with vapor pressures at various temperatures to predict where a compound will boil at each applied ambient pressure. The table and graph below for dichloromethane and methanol illustrate this phenomenon:

Vapor Pressure (torr)	1	10	40	100	400	760
CH <sub>2</sub> Cl <sub>2</sub> T°C	-70	-43	-22	-6	24	41
CH <sub>3</sub> OH T°C	-44	-16	5	21	50	65

(Source: CRC Handbook, p. D-192, 56<sup>th</sup> Ed., 1976)



We take advantage of this in our use of the **rotary evaporator** or **rotovap**. The diagram below is a generic rotary evaporator. The essential parts are shown. The rotary evaporator is used for



removing a solvent well below its boiling point, leaving behind the less-volatile material that was dissolved in that solvent. This is especially essential for a less-volatile solvent which, if removed at atmospheric pressure would have to be heated so high that the solute dissolved in it would decompose on prolonged heating. How the rotary evaporator works is summarized below.

1. First, the solution is poured into the "pot", typically a round bottom flask, using a funnel. Ideally the size of the flask should be **at least** twice the volume of the amount of solvent. Stated another way, the round bottom flask should be filled to **no more than** half its maximum volume. A blue plastic clip is snapped on to secure the flask to the splash protector 19/22 joint.



2. Next, cold water is circulated through the rotovap condenser to condense the volatile solvent on the condenser coils and to prevent, as much as possible, the volatile solvent from vaporizing into the aspirator. Ice and a small amount of water are placed in the water-circulating pump. The circulating pump is plugged in.



3. Next, the aspirator water should be turned on full force. Then the condenser stopcock is turned at a 90 degree angle to the glass tube in the stopcock to instate the vacuum. The system is now "closed". The aspirator lowers the pressure throughout the apparatus. The solvent in the pot will start to evaporate.



4. The flask should then be rotated by turning on the appropriate dial. Since evaporation cools down the pot, it is then immersed in a thermostated warm water bath. This keeps the solvent evaporating steadily. The green light indicates the heating coils are activated. The orange light signals that the bath is heating the water. This orange light goes off when the water temperature reaches the selected value. The temperature selection dial is located at the lower right on the water bath.

The warm vapors of the solvent now encounter a cold condenser coil and recondense, dripping off the coiled glass tube into the reservoir. As long as there is solvent in the pot, condensed solvent will collect in the reservoir. This ceases when the last bit of solvent evaporates from the pot.



5. The speed control rotates the pot in the warm water bath to spread a thin film of solution on the inner surface of the round bottom flask. This encourages more rapid evaporation and minimizes a phenomenon known as "bumping" - an intermittent, uncontrolled boiling. The on-off switch is located at the upper left on the rotovap motor and the speed control knob below it. You need not set the rotation speed of the flask at maximum; however, you must rotate the flask to prevent bumping at reduced pressure.



6. Lower the entire condenser-motor-flask assembly into the heated water bath using the handle, as shown at the right. Press the lever in the handle to disengage the handle lock and slowly lower the rotating flask into the water bath. Be certain there is enough water in the water bath to cover the round bottom flask to half-depth. Do not lower the flask so low that the ground glass joint is under water. This may encourage entry of water into the round bottom. Also, do not lower the flask so low into the bath that any rotating glass part is rubbing against the metal edges of the water bath. This will cut a groove into the splash protector, damaging it and perhaps causing it to implode under vacuum. Continue rotating the flask until all solvent is removed. You can usually tell when the great majority of solvent is removed because there is no more liquid dripping from the condenser.



7. You might ask at this point what prevents the solvent in the reservoir from evaporating, as well. This answer is "nothing". However, since evaporation is a cooling phenomenon, the liquid evaporating in the reservoir cools itself, minimizing its own evaporation. You could also immerse the reservoir in an ice bath to further curtail evaporation. In some cases, when you are removing a very volatile solvent such as diethyl ether or dichloromethane, this is essential.
8. The steps to follow are summarized below:
  - a. Add ice and water to the water-circulating pump start circulation through the condenser. (**Make sure the water level does not reach the pump-it is not a submersible pump!**) Start the aspirator and close the stopcock. Securely clip the partially-filled round bottom flask on the ground glass joint of the splash protector.
  - b. Rotate the flask slowly. This will minimize bumping. Wait about 30 seconds.
  - c. Lower the flask into the warm water bath.
9. When the solvent is removed, reverse the steps:
  - a. Raise the flask from the warm water bath. Stop the rotation and open the stopcock (you will hear air rush into the apparatus).
  - b. Turn off the water aspirator.
  - c. Remove the round bottom flask. A twisting motion is helpful.
  - d. Turn off the water-circulating pump and turn down the temperature control on the water bath unless someone else wants to use the rotary evaporator soon after you do.

If you carefully consider what each part of the rotary evaporator does, you ensure a safe, efficient removal of solvent.

Please learn how to use the rotary evaporator since you will be using it all year. It is not a good reflection on your lab proficiency if you have to ask how to use the rotary evaporator during the second semester.