Experiment No. 1

**Purification of a benzaldehyde by distillation and TLC experiment**

**Aim**: To purify Benzaldehyde by distillation method

**Chemical**: Crude benzaldehyde

**Apparatus**: 100 mL round bottom flask, distillation apparatus, glass stopper, stir bar,   
 hot plate, heating mantle, thermometer.

**General procedure**:

(1) **Collect the necessary glassware** - short path distillation head, thermometer and adapter, receiver flasks (at least two), Vigreux column.

(2) **Preheat oil bath or heating mantle** - if the boiling point is unknown, this step should be omitted. Keep in mind that for most distillations the heating apparatus must be 20-30 °C higher than the boiling point of the distillate. Note: Due to thermal breakdown and possible ignition, oil baths are only useful for temperatures below 200 °C.

(3) Record weight of labeled receiving flasks.

(4) Put compound to be distilled in a round bottom flask with stir bar. (The stir bar will prevent bumping.) The size of the round bottom flask is very important. It should be roughly half to two-thirds full; any higher and it may boil over prematurely, any less and it may take too long to distill.

(5) **Assemble glassware making sure to grease all of the joints**- Be sparing with the vacuum grease—it's expensive and you don't want it getting into your compound.

(6) **Insulate the column**- When using a Vigreux column it should be wrapped using glass wool and aluminum foil. Without insulation, these set-ups tend to take a very long time

(7) Connect the condenser to the water lines, turn on the water, and check for leaks.

(8) SLOWLY open the distillation apparatus to vacuum. You should see the liquid begin to bubble - DON'T WORRY - this is normal. Excess solvent or low boiling impurities will often boil away under vacuum at room temperature (This is a good example of why you need to keep your trap full of liquid nitrogen, otherwise these compounds will go directly into your pump oil!).

(9) Once the bubbling subsides, or slows almost to a stop, then you can start heating the flask.

(10) **Lower your hood sash** - this is always a good practice in case of accident, but it also keeps the distillation apparatus away from the air conditioning of the lab. This will cool your set-up and make your distillation take longer.

(11) **DO NOT HEAT TOO QUICKLY**!!! Patience is the key to distillation.

(12) Slowly increase the temperature of the oil bath until the solution is refluxing.

(13) **Wait to see the distillation thermometer respond**. If nothing happens after about 10 minutes then raise the temperature slightly.

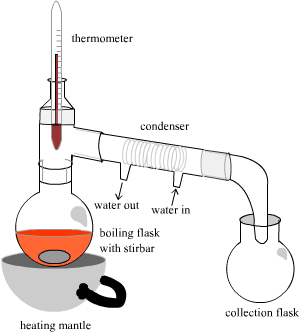
(14) Repeat step #13 until you see the distillation thermometer respond. Once this happens, prepare to collect.

(15) **Try to keep the apparatus at a constant temperature**—at least within 5 degrees of the apparatus temperature when the distillation thermometer registered.

(16) Collect until a dramatic change in temperature occurs. Usually the temperature of the distillation thermometer will drop when one fraction is done distilling. At this point you should change receiver flasks or stop the distillation entirely.

(17) **Release the Vacuum**. When you are done collecting, it is not quite time to cool the apparatus. First, you must release the vacuum. Before you do this, however, make sure that all of your collection flasks are secured to the apparatus by clamps, joint clips, your hand, etc. You do not want to release the vacuum then see your product flask shatter on the bottom of the hood! Once everything is secure, vent the apparatus to nitrogen and then remove the oil bath and let the set-up cool to room temperature.

(18) Once everything has cooled, record the weight of your tared collection flask(s) and calculate the weight of your product(s).



Results: The product obtained was:

1.

2.

3.

4.

5.

Yield (%):

Purity: