# Weigh a sample of benzoic acid and a sample of 3-aminoacetophenone; each sample should be approximately 0.5 g. Dissolve in approximately 25 mL of ether or CH2Cl2. Transfer the solution to a separatory funnel and extract with two *separate* 25-mL portions of 5% sodium hydroxide.

Collect the aqueous extracts in one flask and slowly add 1M HCl until the pH is 2 or lower. Add approximately 3-4 mL HCl dropwise. Cool the mixture in an ice bath, collect the solid by vacuum filtration, wash with cold water and dry to constant mass.

Transfer the ether solution to a tared filter flask and gently remove the ether solvent by evaporation. Gently warm flask on hot plate and swirl to prevent “bumping”. Collect the solid from your filter flask.



Using the original mass of each component, calculate your percent recovery for each component.

**Notes:**

Once the NaOH solution is added to the separatory funnel, two layers will form since water and organic solvents such as ether and CH2Cl2are immiscible. The more dense liquid (water or organic solvent) will be the bottom layer.

* You must know which layer is on top and which is on the bottom.
* Also, you need to know what substance is dissolved in each layer.

With this information, you can keep track of the two dissolved components once the layers are separated.

Often the base used to extract a carboxylic acid is aqueous sodium bicarbonate (NaHCO3 aq). When bicarbonate reacts with the carboxylic acid, CO2 gas is produced. Take care not to stopper the separatory funnel until the fizzing subsides. Use a stir rod to promote de-gassing, then stopper and shake gently. A safe operation is to stopper the funnel, **invert once then open the stopcock to release pressure.** Do this several times until most of the gas is dispelled from the funnel. Then you can vigorously shake the funnel to mix layers thoroughly and separate the layers.