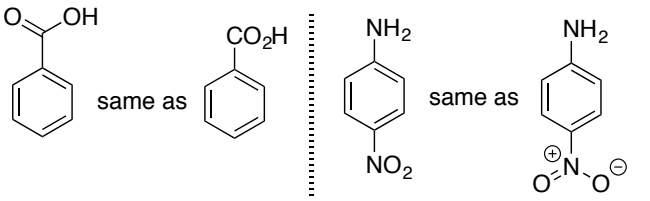
LAB 1 – EXTRACTION/RECRYSTALLIZATION

EXTRACTION – EXPERIMENTAL PROCEDURE



benzoic acid

para-nitroaniline
(p-nitroaniline; 4-nitroaniline)

Weigh about 1.0 g of a mixture that contains equal amounts of benzoic acid and p-nitroaniline using a weighing paper. Place the solid into a 100-ml beaker while at the balances. In your hood, add about 40 ml of dichloromethane into the beaker containing the mixture and stir it with a glass stirring rod; do not be concerned, if several small granules do not dissolve immediately. Transfer the homogeneous solution into the 250-ml separatory funnel.

Add 25 ml of 1M NaOH into the funnel, put the stopper, and shake; allow the layers to separate. A small dark brown "ring" at the interface may form. Drain the bottom layer into 250-ml Erlenmeyer flask labeled "**Organic Layer**." Drain the top layer (aqueous) into 125-mL Erlenmeyer flask labeled "**Basic Extract**." Repeat this procedure by transferring the organic layer into the separatory funnel and extracting with a second 25-mL portion of 1M NaOH. Separate the layers again by draining the organic layer into the same Erlenmeyer as before, *i. e.*, "**Organic Layer**," and then adding the top layer to the 125-ml Erlenmeyer flask labeled "**Basic Extract**" – the total volume will be 50 mL. The basic extract should be cooled in an ice bath for the neutralization step described below; *the cooling can be done by filling a half of a 400ml beaker with ice and water in approximately equal amounts, and putting the Erlenmeyer flask in the bath (make sure you do not spill the contents of the Erlenmeyer into the ice bath)*.

Next, transfer the organic layer to the separatory funnel and extract the organic solution with two portions of 3M HCl (25 ml each) using the same manipulations as described above. The HCl extracts should be combined in a 125-ml Erlenmeyer flask labeled "**Acidic Extract**." Cool the acidic extract on ice for the neutralization step described below.

Benzoic acid:

Benzoic acid is isolated by neutralization of the "**Basic extract**" with 3M HCl, which is added slowly to avoid excessive heat production. Add sufficient HCl to make the solution distinctly acidic to litmus or pH paper (about 20ml will be required). The resulting white precipitate of benzoic acid should be isolated by vacuum filtration; the benzoic acid should be washed with 20 ml of cold water. Keep the filter under vacuum for about 10-15min. Allow the benzoic acid to dry on the weighing paper.

***p*-Nitroaniline**:

The p-nitroaniline is isolated by neutralization of the "**Acidic extract**" with 3M NaOH, which should be added slowly to avoid excessive heating. Add sufficient NaOH to make the solution distinctly basic to litmus (45-55 ml will be required). The resulting yellow precipitate should be isolated by vacuum filtration (keep under vacuum for 10-15min), washed with 20 ml of cold water and allowed to dry on a watch glass.

The "**Organic layer**" after second extraction should be discarded **in the appropriate waste container** (halogenated waste in the hood).

RECRYSTALLIZATION – EXPERIMENTAL PROCEDURE

Use the samples you have obtained from the Extraction experiment above

1. Weigh and record the amount of each recovered sample from the extraction experiment. Make sure this is recorded in your notebook as well as in the report.

2. Do not use boiling stones while heating the samples, but make sure you swirl the flasks occasionally. Recrystallizations should be done in 25ml-Erlenmeyer flasks.

3. Recrystallization of p-nitroaniline (mantle setting ~4-5):

a) for 0.5 g of p-nitroaniline add approximately 5 ml of EtOH. Proportionally adjust the amount of EtOH for the actual amount of sample.

b) cool the flask on ice, then vacuum filter, and keep the crystals under vacuum on the filter paper for ~10min.

4. Recrystallization of benzoic acid (mantle setting ~8)

a) for 0.5 g of benzoic acid, first add ~10 ml of water into the Erlenmeyer flask. When the sample is dissolved, add sufficient excess water (about 4-5 ml) to prevent premature crystallization. Proportionally adjust the amounts of water for the actual amount of sample.

b) use decolorizing carbon if sample is colored, and a sufficient excess of water (~5 ml) to prevent crystallization on the funnel during the hot-filtration step (gravity filtration to be used here). Put the flask on ice. Once the crystals (solid) have formed, vacuum filter, and keep the solid on the filter under vacuum for at least 20-30 min before weighing it out.

5. Weigh each dried sample and calculate the % recovery.

6. Determine the melting point of both benzoic acid and p-nitroaniline.
You should use the thermal ruler for a quick measurement before using a Mel-Temp for an accurate measurement.

7. Dispose your sample into the appropriate containers. Please do not mix the sample, be very careful not to contaminate the sample. Lab reports will be due next week.