Chem 228 Synthesis Project: **Synthesis of a Solvatochromic Diazo Dye** FS2016

Step #3: *Diazotization and coupling of p-aminobenzoic acid (PABA) with 2-naphthol*.

Download the following reference papers:

Neha and Manisha Patni, "Der Chimica Sinica", 2016, 7(2): 93-100 (via http://www.imedpub.com/der-chemica-sinica/archive.php)

Kofie, William, et al. "International Journal of Pharmacy and Pharmaceutical Sciences", 2015, 7(4), 398-401. (via http://ijppsjournal.com/contents.htm)

MSDS List for Step #3: NaNO2 3M HCl 3M NaOH Na2SO4 2-naphthol

Procedure:

Set hotplate to ~200 ^{o}C while weighing up chemicals. Set up an ice bath or two. Place a small beaker with ~50 ml of distilled water in the ice bath to cool for later use.

Weigh 1.442 g (0.01 mol) of 2-naphthol and crush with a mortar & pestle. Transfer to a small labeled beaker. Add 3.4 ml of 3M NaOH (~0.01 mol). Heat to dissolve, add 5 ml of water then place in ice bath.

Weigh 1.371 g (0.01 mol) of PABA, transfer to another small labeled beaker. Add 7 ml (0.02 mol) of 3M HCl. Heat to dissolve, add 5 ml of water then place in ice bath. Some crystals may form, this is OK.

Weigh 0.695 g (0.01 mol) of NaNO2, add to 10 ml of water. Heat to dissolve then place in ice bath. When cold (0-5 ^{o}C) add NaNO2 solution to PABA solution in ice bath. The PABA crystals should dissolve and a clear pale yellow solution form.

Add the cold 2-naphthol solution, which should result in immediate precipitation of the brick red diazo dye. Stir, check that pH is 1-2 and allow to stand in ice bath for 20-30 min.

Vacuum filter the product using a 7 cm Buchner. Wash the dye 2x with 10 ml portions of ice cold water by disconnecting the vacuum, stirring the dye with the water, then reconnecting to remove the water.

Transfer to a labeled watch glass, place uncovered in desk. Product will need to dry at least a week before weighing, MP, TLC, FTIR & NMR.

Prelab Question (step 2)

1. Write the reaction of HCl with NaNO2 to form nitrous acid.
2. Write the coupling reaction of p-aminobenzoic acid occurring in step #3 and calculate the theoretical yield based on your starting amount (1.37 g, 0.01 mol).
3. Why is it necessary to maintain the reaction temperature below 5 ^{o}C?