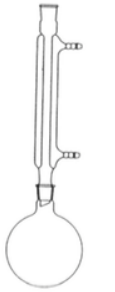
Chem 228 Synthesis Project: **Synthesis Solvatochromic Diazo Dye** FS2016

Step #2 Reduction of p-nitrobenzoic acid to p-aminobenzoic acid (PABA)

Download and print out the procedure for p-aminobenzoic acid in "Vogel's Textbook of Practical Organic Chemistry, 5th ed." (exp 6.52, p. 896)


Simple reflux

MSDS List for Step 2: Sn 12M HCl 18M NH4OH glacial acetic acid

Procedure:

Heat a steambath with ~1" of dist water to boiling on a hotplate set to ~250C.

In a 250 RB flask fitted with a condenser & stir bar, add 3.0 g of p-nitrobenzoic acid (0.018 mol), 7.0 g Sn metal (0.059 mol), and 15 ml conc (12M) HCl (0.18 mol) via powder funnel.

Immerse the flask in the hot water bath until a vigorous bubbling commences. Remove from the bath & continue stirring. Manually swirl the flask occasionally to wash down any reactant on the upper walls of the flask. Heat via water bath as needed to maintain a vigorous reaction rate. After 30-45 min, when most of the Sn has dissolved, cool and decant the liquid into a 250 ml beaker. Wash the flask to transfer any remaining organics into the beaker. Discard any remaining Sn metal.

Add conc. NH4OH to the beaker until pH~8 with pH paper. (I needed ~30 ml).
Filter white pasty hydrated tin oxide via 7 cm Buchner/500 ml filter flask. Rinse ppt with water. Discard ppt.

Transfer clear filtrate to 250 ml beaker with stir bar and evaporate to ~40 ml, or until the mixture becomes cloudy on hotplate set at 330C.

Cool beaker contents, add glacial acetic acid to pH 3-4 via pH paper. (I needed 1-2 ml). Cool beaker contents to RT, then in ice. Filter yellow product on 7 cm Buchner/500 ml filter flask. Wash ppt with cold water. Transfer ppt to tared weigh boat to dry. Discard filtrate.
Product will need to dry at least a week before weighing, MP, TLC, FTIR & NMR.

Prelab Question (step 2)

1. Write the reduction reaction of p-nitrobenzoic acid occurring in step #2 and calculate the theoretical yield based on your starting amount (~3g).
2. Write the reaction of Sn with HCl, and then that reaction product reacting with NH4OH to form a white precipitate.
3. Write the reaction of the organic product, (PABA) with HCl and its reaction with NaOH.