**Exp 8. Synthesis of Acetophenetidin**

**Introduction:**

Acetophenetidin ( p-ethoxyacetanilide) is known as phenacetin. It was used as an analgesic and antipyretic in 1887. This experiment is to synthesize acetophenetidin by reacting p-phenetidine and acetic anhydride.



 Procedure:

First week

1. Weigh out 2 (± 0.1) g of p-phenetidine in a 125 mL Erlenmeyer flask
2. Add 38 mL of deionized water and 1.3 mL of concentrated hydrochloric acid (add acid to water) to the 125 mL Erlenmeyer flask.
3. Add 1 scoopula of charcoal and a couple of boiling stones to the reaction mixture.
4. Boil the reaction mixture for 5 minutes.
5. Setup hot gravity filtration (<http://www.youtube.com/watch?v=u-O2VDM9n5E> or search online on hot gravity filtration) while doing step 4.
6. Perform hot gravity filtration.
7. Rinse the filter and the 125 mL Erlenmeyer flask with 2 X 5 mL of hot water.
8. Remove the reaction mixture from the hot plate and place a thermometer in the reaction vessel.
9. Prepare a sodium acetate solution by dissolving 2.1 g sodium acetate trihydrate in 9 mL of deionized water (use all in step 10).
10. When the filtrate cooled to 70 0C, add sodium acetate solution and 2.5 mL of acetic anhydride (corrosive reagent) simultaneously to the reaction mixture, then use a stirring rod (not thermometer!) to stir the solution.
11. Allow the solution to slowly cool down to room temperature while stirring periodically.
12. Cool the reaction mixture in an ice-bath for 15 min.
13. Collect crystals by vacuum filtration and rinse crystals with deionized water.
14. Allow a week for the crystals to dry.

Second week

1. Weigh your product.
2. Use 1 g of your product to conduct recrystallization.
3. Put 1 g of product into a 125 mL Erlenmeyer flask and place the flask on a hot plate.
4. Add 30 mL of deionized water to the flask and apply heat.
5. Use minimum amount of hot water to dissolve sample (30 mL of deionized water may not be enough)
6. Once more than 95 % of sample dissolves in the hot water, solution, remove the flask from the hotplate and allow it to slowly cool down to room temperature.
7. Cool the solution in an ice bath for 15 minutes.
8. Collect crystals by vacuum filtration and rinse crystal with deionized water.
9. Allow crystal to dry for a week before weighting and melting point determination (one for crude product and one for recrystallized product).