Second Part- Organic Synthesis

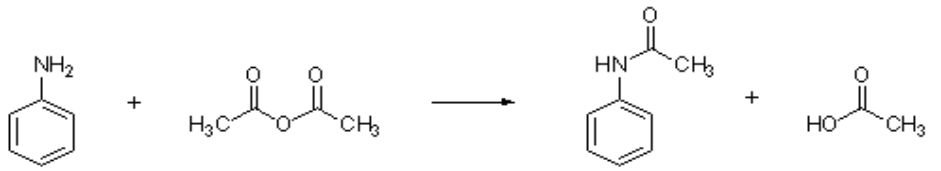
Exp.05: preparation of Acetanilide

Objectives:

- To synthesis acetanilide by reaction of aniline and acetic anhydride (amine to amide).
- To purify acetanilide by crystallization method using water.
- Purity check by melting range and TLC.
- Acetanilide characterization using IR spectrum.

Discussion:

This experiment involves four functional groups common in organic chemistry. The substrate (reactants) are both liquids and one of the products is solid. The reaction of aniline with acetic anhydride is a transformation in which products, acetanilide and acetic acid, are obtained.



anline acetic anhydride acetanilide acetic acid

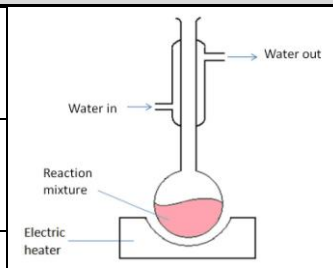
The substrate (reactants):

Compound	MP (BP)	Density	Hazards
Aniline	(184 ^{0}C)	1.022 g/mL	Irritant (eyes/skin). Harmful if inhaled/ingested. Possible carcinogen.
Acetic Anhydride	(138 ^{0}C)	1.082 g/mL	Irritant (eyes/skin). Toxic by inhilation, Flammable (fp 49 ^{0}C).

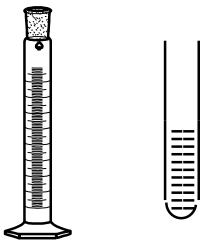
A solid product is often desirable since it may be recrystallized and a melting point determined.Recrystallization is a widely used technique to purify a solid mixture. The desired product is isolated from its impurities by differences in solubility. Insoluble impurities and colored impurities can be removed from hot solvent through the use of activated carbon and filtration. Soluble impurities remain in the cold solvent after recrystallization. The desired product should be as soluble as possible in hot solvent and as insoluble as possible in cold solvent. The selection of solvent is, therefore, critical to the successful recrystallization.

Experimental Procedures:

First Method:

Step	Procedures	
1	Place 0.1 molof aniline (d=1.022 g/mL)in a spherical flask(100mL).	
	Add 20 mL of glacial acetic acid and 20 mL of acetic anhydride.	
	Heat the mixture under reflux for 10-15 min.	
2	Cool the reaction mixture and transfer it into beaker contain 50 mL of NaOH 5% with stirring in ice bath.	
3	Collect the product by vacuum filtration usingBüchner funnel.	
4	Purify acetanilide by crystallization method using water.	
5	Allow the sample to dry completely. Weigh the dry product, calculate the percentage yield and determine its melting point. Collect to product in a paper and write your name and submit it with the report.	
	% Yield acetanilide = mass acetanilide recovered x100 Theoretical mass of acetanilide	

Second Method:

Step	Procedures	
1	Using a dropper, place 0.15 to 0.20 g of aniline (about 10 drops) (d = 1.02 g/ml) in a large tared test tube and determine the weight to the nearest mg.	
	Add 5 ml of distilled water to the test tube and then add 20 drops of acetic anhydride again using a dropper	
	Stir, the mixture using stirring rod for 5 minutes until solid forms.	
2	The product crystallized in the same test tube. Add 5 ml of water and heat the test tube in a hot water bath with occasional stirring until the entire solid dissolved.	
3	Set the test tube aside to cool for 3-5 minutes and then chill it in an ice bath. When crystallization is complete, collect the product by vacuum filtration using Büchner funnel.	

4	Allow the sample to dry completely. Weigh the dry product, calculate the percentage yield and determine its melting point. Collect to product in a paper and write your name and submit it with the report.
	% Yield acetanilide = $\frac{\text{mass acetanilide recovered}}{\text{Theoretical mass of acetanilide}}$ x100

Characterization:

After you make sure that your sample is completely dry. Take a small amount to the IR lab in building 17 for IR analysis.

Discuss the spectrum in the report.

Laboratory Report

Name: ---------- Date: ----------

Experiment Subject: ----------

- Reaction:

Calculations:

Compound	Mol. Formula	Mol. Wight	Moles	Wight, mg	Density	Volume	Limiting reagent

Purification:

Recrystallization solvent: ----------

Purity check by melting range: ----------

TLC: ----------

Physical Data (Product):

State: ---------- Melting Point: ----------

Color: ---------- Solubility: ----------

Yield:

Characterization:
