EXPERIMENT 6
Preparation of Acetanilide

College Of Science
Chemistry Department

Preparation and purification of Acetanilide

Purpose:

a) To synthesis acetanilide by reaction of aniline and acetic anhydride.
   b) To purify acetanilide by crystallization method from water

   c) Purity check by melting range

Equipment / Materials and Hazars:

hot plate beakers(250, 400 mL) ice stirring rod spatula

Büchner funnel aniline weighing paper digital scales rubber tubing (hose) acetic
anhydride filter paper Mel-temp apparatus
10- mL graduated cylinder large test tube medicine dropper

Compound	density	MP (BP)	FW (g/mol)	Hazards
Acetanilide	---			Irritant. Harmful if inhaled/ingested.
Aniline	1.022 g/mL	(184 ℃)	93.13	Irritant (eyes/skin). Harmful if inhaled/ingested. Possible carcinogen.
Acetic Anhydride	1.082 g/mL	(138 ℃)	102.09	Irritant (eyes/skin). Toxic by inhilation,

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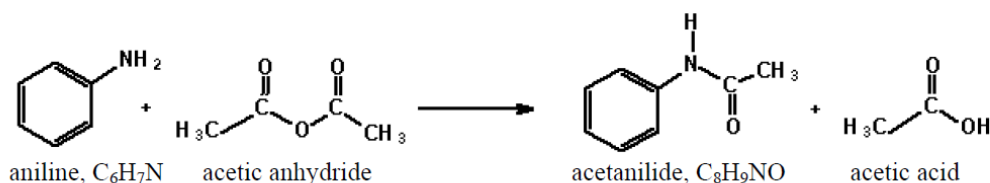## Preparation of Acetanilide

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Discussion:

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.

Recrystallization is a purification procedure, which requires solubility of the impure solid in a heated solution and crystallization of the solid upon cooling. Clearly, this operation depends upon solute-solvent in traction involving a number of parameters including concentration, polarity of solute and solvent (like dissolves like), etc. Choice of a solvent or solvent pair for recrystallization experiments generally involves preliminary tests using a small sample and various solvent systems. To determine the proper solvent or solvent system, the following steps are commonly performed.

I) The crude crystals should have low solubility in the chosen solvent at room temperature.
II) The crude crystals should have high solubility in the chosen solvent when heated to boiling.
III) The crude crystals should not react with the solvent
IV) The solvent should boil at temperature below the solid melting point.
V) The solvent should moderately be volatile so crystals dried readily.
VI) The solvent should be non-toxic, non-flammable, and inexpensive

aniline, C_6H_7N acetic anhydride   acetanilide, C_8H_9NO acetic acid

Experimental Procedures

Using a medicine 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 medicine dropper (Fig.1). stir, the mixture using stirring rod for 5 minutes until solid forms.

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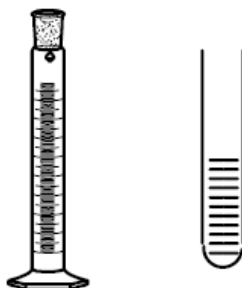
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Fig. 1 – 10 mL graduate cylinder and large test tube

The product crystallized in the same test tube. Add 5 ml of water and heat the test tube in a hot water bath (400 mL beaker) (Fig.2) with occasional stirring until the entire solid dissolved. 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 a small Büchner funnel (Fig.3). 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 to your instructor. The aqueous filtrate may be flushed down the drain.
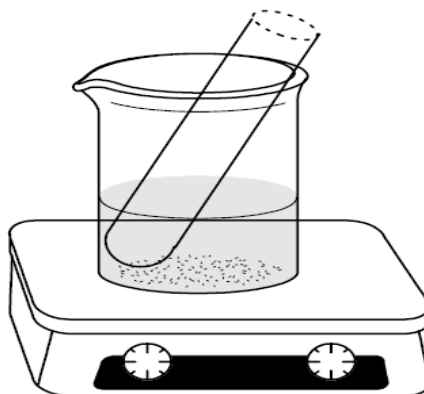

Fig. 2 – Hot water bath

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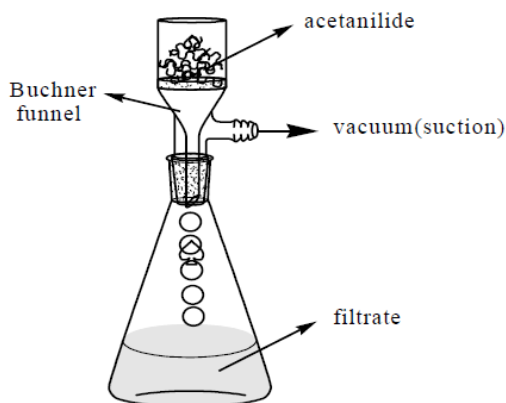**College Of Science**
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Fig. 3 – Büchner funnel and suction flask

$$\% \text{ Yield acetanilide } = \frac{\text{mass acetanilide recovered}}{\text{theoretical mass of acetanilide}} \times 100$$

Data and Results (Preparation and Purification of Acetanilide)

Date:_____ **Lab Report:** _____

1. Sample name _____

2. Data on the impure sample

a. Mass of the aniline + test tube + beaker _____ g

b. Mass of the aniline + test tube _____ g

c. Mass of aniline _____ g

d. Mole of aniline _____ mol

e. Theoretical moles of Acetanilide _____ mol

f. Theoretical mass of acetanilide _____ g

(show calculation)

3. Data for recrystallized acetanilide

a. Mass of recrystallized acetanilide + Weighing paper _____ g

b. Mass of recrystallized acetanilide _____ g

c. Calculation of percentage recovery

(show calculation)

_____%

d. Melting point of recrystallized acetanilide _____ ^{o}C

    e. Structural formula of the sample recrystallized