Preparation of acetanilide

Synthesis of Acetanilide Reaction O NH2 + H3C C O O C CH3 O N C CH3 H + H3C O C OH Aniline Acetic anhydride Acetanilide Acetic acid Purpose: Acetanilide is a useful precursor to many pharmaceuticals such as acetaminophen and penicillin. Experimental Procedure. (Estimated time: 1. 5 h.) Unless otherwise noted, all manipulations should be done in the chemical fume hood. Place 100 µL of aniline into a tared 10 X 75-mm test tube (standing in a small beaker or Erlenmeyer flask). Now add 0. -mL of distilled water; with swirling, followed by 3 drops of concentrated hydrochloric acid. Add 10 mg of powdered decolorizing charcoal, or the pelletized form (Norit) to the resulting solution. Fit the test tube with a cork stopper and take it back to your hood. Gravity filter this suspension (25-mm funnel fitted with fluted fast-grade filter paper - see instructor) into a 3. 0-mL conical vial containing a magnetic spin vane. Wet the filter paper in advance with distilled water and blot the excess water from the stem of the funnel.

Use an additional 0. 5 mL of distilled water to rinse the test tube and pass that through the filter paper into your vial. Your aniline hydrochloride solution is ready for reaction. Assemble the apparatus as shown below: CONICAL VIAL W/ MAGNETIC SPIN VANE AND AIR CONDENSER Dissolve 150 mg (1. 10 mmol) of sodium acetate trihydrate in 0. 5 mL of distilled water in a 10 X 17-mm test tube. Cap the tube and set the solution aside for use in the next step.

Add, with stirring, 150 µL of acetic anhydride to the solution of aniline hydrochloride, followed quickly by addition (Pasteur pipet) of the previously prepared solution of sodium acetate. Stir to thoroughly mix the reagents (~5 min.). You should see the formation of a white precipitate. Allow the reaction

mixture to stand at room temperature for approximately 5 min and then place it in an ice bath for an additional 5-10 min to complete the crystallization process.

Collect the acetanilide product by filtration under reduced pressure using a Buchner funnel. Rinse the conical vial with two 0. 5-mL portions of distilled water and use the rinse to wash the collected filter cake. Characterization. Weigh and determine the percent yield of the product. Obtain an IR spectrum using the total internal reflectance attachment. Take a 1H NMR of your product - if there is not enough we will pool samples together. Take the melting point of the product and compare it to the literature value. BUCHNER FUNNEL