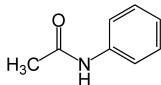


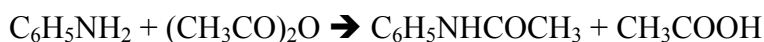
# Recrystallization of Acetanilide

## Background:

Acetanilide



Acetanilide can be produced by reacting acetic anhydride with aniline:



It is used as an inhibitor of hydrogen peroxide decomposition and is used to stabilize cellulose ester varnishes. It has also found uses in the intermediation in rubber accelerator synthesis, and camphor synthesis. Acetanilide is used as a key intermediate for the manufacture of the sulfa drugs, penicillin and other pharmaceuticals. It is also known as N-phenylacetamide, acetanil, or acetanilid.

## Reflux Apparatus

It is often necessary to heat a substance in a solvent for a long period without boiling away the solvent. This can be accomplished by attaching a vertical condenser to the flask containing the boiling solution (see the setup in the front of the lab). The solvent vapor condenses and returns as a liquid to the boiling solution. The continuous boiling, vaporization and back-flow of condensate in this manner is called refluxing. In recrystallization, we use a reflux apparatus to obtain a solution to be purified.

## Activated Charcoal

Activated charcoal (aka activated carbon) is a form of carbon processed to have small, low-volume pores that increase surface area for adsorption or chemical reactions. It has numerous uses in the areas of gas purification, decaffeination, gold purification, metal extraction, water purification, medicine, sewage treatment and air filters. Activated charcoal is produced from materials such as nutshells, coconut husks, peat, wood, coir, lignite, coal, and petroleum pitch. It is produced by one of the following methods: carbonization (pyrolysis) under inert atmosphere, Oxidative activation (pyrolysis in an oxygen or steam atmosphere), or chemical activation where the carbonaceous material is treated with a strong acid or base prior to carbonization.

## Recrystallization of Acetanilide

In this experiment a sample of impure acetanilide (a “dirty” mixture) will be recrystallized from water. The crude and pure products will be weighed and the melting point determined before and after recrystallization to illustrate the efficiency of the process. The purified product should appear as white flakes.

## Procedure

Weigh out a 2g sample of impure acetanilide and record its weight to 4 significant figures. Use a few milligrams of it to determine the melting point. Place the rest of the acetanilide in a 100-mL round-bottom flask, connect this to the reflux condenser and start a slow stream of water through the jacket of the condenser. Add 1 or 2 boiling stones and 35 mL of water to the flask through the top of the condenser and bring the water to a boil by heating with a heating mantle. Adjust the electrical current to the mantle so that the water refluxes steadily. Continue to heat until no more solid appears to dissolve. Then remove the mantle, allow the flask to cool a moment after refluxing stops. Remove the condenser, and add a small amount (about 0.2g) of decolorizing charcoal to the contents of the flask. Replace the condenser and allow the solution to reflux for an additional 5 minutes.

Meanwhile set up a vacuum filtration apparatus using a 125-mL filter flask (see setup in the front of the lab). Set a piece of filter paper in place, connect the flask to the vacuum jet on your bench top, and turn it on. Pour 15-20 mL of boiling water through the funnel to warm it and to wet the filter paper. Discard this water and reconnect the funnel to the filter flask.

Remove the heating mantle from the reflux assembly, disconnect the condenser, and using the clamp as a handle, filter the hot acetanilide solution, without delay, being sure the vacuum is turned on.

CAUTION: Pour the solution slowly at first to form a pad of charcoal on the filter paper.

If particles of charcoal pass through the filter paper, return the filtrate to the round bottom flask, heat the solution to boiling, and filter it again through the same piece of filter paper.

As the filtrate cools, crystals will begin to form immediately. Place the filtering flask in an ice bath to complete crystallization. Meanwhile discard the used filter paper, rinse the funnel, and refit it with clean filter paper.

When crystallization is complete, collect the crystals by vacuum filtration. (This can be done by using a second filter flask, if it is available, or by transferring the cold solution and crystals to a beaker before filtration.) Rinse the crystals (with the vacuum on) with a few milliliters of ice-cold water. Using a clean spatula, press the crystals as dry as possible on the funnel. Then transfer the crystals to a watch glass. Store them in your locker for drying until the next laboratory period.

Weigh the dried product and determine its melting point. Calculate the percentage recovery of pure material. Turn in the purified acetanilide in a sample vial to your instructor with your report. Don't forget to label the vial with your name and the name of its contents.

$$\% \text{ Recovery} = \frac{\text{wt. of dry purified crystals}}{\text{wt. of impure original sample}} * 100\%$$