PRELAB FOR EXPERIMENT #1: RECRYSTALLIZING A SOLID

Your name TA's name
Your Partner's name Lab Section

PURPOSE:
To learn how to 1) select the appropriate solvent for recrystallizing an impure material and 2) perform a hot recrystallization.

MATERIALS:
REAGENTS

Name of Reagent	M.W.	M.P./B.P	density	hazards
Acetanilide	135	114	1.219	toxic
Water	18.02	100	1.00	Yeah right!
Acetone	58.08	56.2	0.7857	flammable
Ethanol	46.08	78.3	0.789	flammable
Petroleum ether	~ 80	30-60	0.656	flammable
Carbon black	12	N/A	N/A	Dust hazard

[Reagents located using http://chemfinder.cambridgesoft.com/]

A. Selecting a Recrystallization Solvent
1) Place 100-200 mg acetanilide in each of four test tubes labeled: 'water', 'acetone', 'ethanol' and 'pet ether'.
2) Add 2 ml of the indicated solvent to the test tubes and mix. Record cold solubility observations.
3) Select the test tubes with solvents which did not dissolve acetanilide and place them in the hot-water beaker and warm them up to a gentle boil on a hot plate.
Record solubility observations in hot solvents.
4) Allow the test tubes to slowly cool down to room temperature undisturbed. Prepare an ice-water bath in a 250 ml beaker. Place the test tubes in the ice water bath to effect further crystallization. Record your observations of any crystal formation.
5) Select the solvent most appropriate for recrystallization of acetanilide.
Check your choice with your TA, and proceed with the experiment.

B. Decolorizing a Solution of Impure Acetanilide and Hot Filtration
1) Set up a hot filtration apparatus using a stemless funnel, a fluted filter paper (the TA will demonstrate), and a 125 ml Erlenmeyer flask.

2) Weigh out 1.5 g of impure acetanilide. Reserve enough for a melting point (about the volume of two match heads) and place the rest in a 125 ml Erlenmeyer flask.
3) Place about 50 ml of the chosen recrystallizing solvent in a 150 ml beaker, add a boiling chip and bring to a gentle boil on a hot plate. Add about 15 ml of hot recrystallizing solvent to the Erlenmeyer flask that contains the acetanilide and bring the mixture to boiling.
4) Add small portions of solvent successively, while stirring and gently boiling the mixture, until the entire compound has dissolved.
5) Add about 10% more solvent, and let the mixture cool for about 20 s.
6) Cautiously add about one-quarter teaspoon (a spatula-tip full) of decolorizing carbon and bring the mixture to a gentle boil.
6) Pre-steam the hot filtration apparatus, and begin filtration by pouring a small portion of the mixture onto the funnel. When most of this has gone through the filter, add some more. Continue until all of the dissolved material has been filtered
7) Rinse the dissolving flask with 5-6 ml of hot solvent (10% of the volume used so far) should be added to the charcoal flask and reheat to a gentle boil.
8) Reduce the total solvent volume by gentle boiling to 2/3 initial volume. Set flask aside to slowly cool.

C. Isolating the Recrystallized Product with Vacuum Filtration
1) Set up the vacuum filtration apparatus.
2) When the crystallizing mixture has reached room temperature, further chill in an ice water bath. Chill some recrystallizing solvent for washing the crystals.
3) Turn on the vacuum knob and add a little solvent to the funnel. Swirl the crystallizing flask a few times to suspend the crystals, and pour them out onto the funnel.
4) Rinse with chilled solvent and press the filter cake dry with a spatula.
5) Draw air through them for 5 minutes. When the crystals are dry, place them onto a tared watch glass. Place the watch glass with the sample in the drying oven at 60°C for 10 minutes to remove the last traces of solvent. Record the mass of the purified acetanilide, and determine the percent recovery based on the amount of crude material used.

D. Determining the Melting Range

1) Using a Melt-Temp, determine the melting temperature of both the impure and the purified acetanilide.