Recrystallization of an Impure Sample of Naphthalene

Objectives:

1. To purify a sample of naphthalene using the technique recrystallization. Methanol will be the solvent used for the recrystallization.
2. To evaluate the inherent trade-offs of the technique of recrystallization (high percent recovery versus high purity of the final naphthalene sample).

Procedure:

Add 1 g of the impure naphthalene to a 25 mL Erlenmeyer flask. Add methanol and a boiling stone to a second Erlenmeyer flask. The exact volume of methanol that will be needed for the recrystallization is an unknown quantity. It is best not to boil too much methanol (it takes a long time for it to boil) and not to boil too little methanol (running out of solvent during the recrystallization also costs time). Place the Erlenmeyer flask with the methanol on the hot plate and heat it to boiling. Once the solvent is boiling, use a pipet to transfer small portions to the Erlenmeyer flask containing the naphthalene. At this point, both Erlenmeyer flasks should be placed on the hot plate. Continue adding hot methanol to the naphthalene sample until all of the naphthalene has dissolved. To encourage dissolution of the naphthalene, swirl the Erlenmeyer flask periodically. Remove the hot saturated solution of naphthalene in methanol and place on an insulated surface, like a cork ring. Allow the solution to cool to room temperature. Further cool the solution using an ice bath. During the cooling process, ensure that the remaining solvent (aka mother liquor) does not evaporate as the crystals of naphthalene form. If crystallization has not started, several strategies can be used: (1) scratch the inside of the Erlenmeyer flask with a glass rod or (2) add a seed crystal. After crystallization is complete, vacuum filter the mixture using a Büchner funnel to separate the naphthalene crystals from the mother liquor. The crystals may be washed with cold methanol. Solids may form in the mother liquor in the filter flask; record this observation, but do not attempt to isolate these crystals. Allow the collected naphthalene crystals to dry. Record a final mass of the recrystallized naphthalene. Analyze the purity of the recrystallized naphthalene and the contents of the filtrate (mother liquor) by TLC, using a mixture of hexanes and ethyl acetate to develop the plate. Use the final mass of naphthalene and the TLC data to evaluate whether the naphthalene has been successfully purified and how much naphthalene was lost in the process of recrystallization.

Continuation of Ch 8, Exp 1. (You may want to add a couple of notes to your procedure from Ch 8, Exp 1. Most of the information below will already be in your procedure from week 4).

After completing the naphthalene recrystallization, use the same basic procedure to recrystallize the crude benzoic acid and crude dimethoxybenzene obtained in the Week 4 lab. Before carrying out each recrystallization, set aside a small amount of each crude product to be saved for analysis next week. Benzoic acid is recrystallized from water, dimethoxybenzene is recrystallized from methanol. (Note: dimethoxybenzene has a low melting point, so make sure that during the recrystallization, dimethoxybenzene is actually dissolving in the methanol, not just melting.) In these cases, if you do notice the formation of crystals in the filtrate in the filter flask, you will want to refilter and collect these crystals, allowing you to maximize percent recovery.