Tetraphenylcyclopentadienone. (revised 4/02)

This week you will prepare tetraphenylcyclopentadienone, recrystallize it and determine its mp. You will also collect the following data for the three previously synthesized compounds, benzoin, hydrobenzoin, and benzil: mp's (crude and recrystallized) and ir spectra (as mulls).

Synthesis of Tetraphenylcyclopentadienone. Using the benzil prepared during the previous lab period, prepare tetraphenylcyclopentadienone according to the procedure given in Williamson, Chapter 52 (Exp 1, Microscale). Note the following changes and suggestions.

Be very careful to not break the thermometer or poke a hole through the bottom of the reaction tube. USE CARE WHEN WORKING WITH TRITON B – SEE SAFETY SECTION BELOW. To measure the 0.20 mL of Triton B solution, use the syringe with the blunt-end needle from your kit. Draw the 0.2 mL into the syringe, then place the syringe into a 50 mL Erlenmeyer flask, needle tip down, until you are ready to use the Triton B. Reaction temperature is critical; when the sol'n has reached 100°, remove the tube from the sand bath and if necessary allow it to cool to 100°. Then add the Triton B and stir well. Maintain the temperature at 90-100° until crystals begin to appear, then continue heating at this temperature for a minute longer. In the hood, rinse the syringe with a little acetone and add the rinse to the Triton B waste container. Then rinse out the syringe with water at your wash sink.

If the crystals are too small to separate from the solution by the pipet method, collect them on the small Hirsch funnel. After rinsing the crystals with cold methanol, save a small amount of crude material for a mp (to be taken later), and recrystallize the remainder. CHANGE: use as a recrystallization solvent, a 1:1 mixture of 95% ethanol and toluene (approximately 1.2 mL/50 mg product), not triethylene glycol as stated in the book. The crystals dissolve slowly so let them heat for a minute between additions of solvent. Otherwise, too much solvent may be added resulting in a low or zero yield of crystals (you can always boil off excess solvent if you've added too much). Allow the crystals to dry at least overnight. Weigh the product, determine the % yield, and take the mp of crude and recrystallized material.

WASTE DISPOSAL: CHANGE: Place all filtrate and methanol rinsings from the initial reaction into the container labelled "TRITON B Waste". Solvent and rinsings from the recrystallization should be placed into the "NONHALOGENATED solvent waste" container. Eventually, when you are finished with your product and your TA no longer needs to see it, place it into the container labelled "tetraphenylcyclopentadienone" - do not include filter paper. We may actually put your product to use in a research lab, thus recycling it and eliminating the need for disposal.

SAFETY. Triton B is toxic and is as caustic as sodium hydroxide. Keep it off of your skin. If you become accidentally contaminated, wash the affected area immediately with water. Clean up all spills immediately or others may become contaminated.

Postlab Questions for Weeks 1 and 2 of the Multistep Synthesis

1.) Write mechanisms for all of the reactions in the sequence, except for the oxidation.

2.) A compound that contains a highly conjugated system of pi electrons absorbs energy in the visible region of the electromagnetic spectrum, thus making the compound appear colored. Using resonance structures discuss why tetraphenylcyclopentadienone is highly colored.

3.) Why is Triton B rather than sodium hydroxide used as a base?

4.) Interpret the ir spectra of benzoin, hydrobenzoin, and benzil and discuss how ir is especially helpful in following these transformations.

5.) In the oxidation reaction nitrogen gas is evolved. Show a mechanism by which nitrogen is formed. (Hint: note that in the reaction, nitrate is reduced to nitrite. Also, consider Wade, section 19-17.)