CH 241  Preparation of Caffeine Salicylate

Note! This is your first preparative lab. Be sure to include the balanced equation of the reaction and show the mechanism of the reaction. Calculate the moles of reagents used, the theoretical yield of the product, and after the lab, the % yield of the product.

Last week you extracted caffeine from tea. This week you will use the purified caffeine you collected and prepare a derivative to confirm the identity of the organic compound you extracted.

Caffeine melts and sublimes at 238 °C and for that reason is difficult to identify with a melting point determination. However, since caffeine is a base and can accept a proton, it can react with an acid to form a salt. If the acid used is salicylic acid, the salt formed, caffeine salicylate, has a sharp melting point and can thus be used to help characterize caffeine.

A. Procedure

Based on the fact that the balanced equation shows that 1 mole of caffeine will react with 1 mole of salicylic acid, calculate the mass of salicylic acid required to react with the amount of caffeine you isolated last week. To do this reaction successfully, you probably should have at least 10 mg of caffeine. Add the caffeine and salicylic acid to a tared reaction tube. If you have a large quantity of caffeine, you may want to use a larger container for the reaction. (See your instructor). Add about 0.5 mL of dichloromethane for every 10 mg of caffeine. Heat the mixture to boiling and gentle boil for about 5 minutes. The product can be isolated (crystallized) by a mixed solvent technique. The ionic salt will readily dissolve in the polar dichloromethane, but not in petroleum ether. The addition of petroleum ether (a poor solvent for the product) will induce crystallization. Add petroleum ether dropwise until the mixture just turns cloudy. If too much petroleum ether is added, clarify the solution by adding a very small quantity of dichloromethane. Insulate the tube in order to allow it to cool slowly to room temperature, and then cool it in ice. Removing the solvent with a Pasteur pipette while the reaction tube is in the ice bath, isolates the needle-like crystals. Evaporate the last traces of solvent under vacuum, and determine the weight of the derivative and its melting point. Using a KBr pellet, obtain an IR of the derivative.

B. Analysis

Compare your experimental melting point of your product to the literature melting point of caffeine salicylate. Calculate the % yield of your reaction. Compare the IR spectrum you obtained with an IR from the literature.

C. Useful websites:

http://ull.chemistry.uakron.edu/organic_lab/cola/
Preparation of caffeine salicylate is demonstrated with close-up color photos.

http://www.forumsci.co.il/HPLC/FTIR_page.html

http://www.chem.orst.edu/ch361-464/ch362/irinterp.htm

Theory of FTIR

http://www.chem.orst.edu/ch361-464/ch362/IR_NMRuse/IR1.htm

Tutorials of the preparation of thin films and KBr pellets