

Sublimation of Caffeine

You may want to test the procedure using a sample of pure caffeine first, in order to observe the sublimation, before attempting the sublimation on your extracted caffeine sample.

Preheat the hotplate/Al block to about 200 ± 5 °C. Put some ice and rock salt into a beaker. Assemble the sublimation apparatus shown below with your crude caffeine in the 5 ml conical vial. Install a trap between the apparatus and the aspirator. Turn the water on full and make sure you are getting a vacuum and all joints have good tight seals. Place some cold water from the ice/salt mixture into the thistle tube condenser with a long pipet so that the small stem is filled with fluid.

To begin sublimation, lower the sublimation apparatus into the preheated hot Al block and look for deposition of caffeine on the tip of the cold finger condenser. Sublimation should be complete in 1-5 min. Circulate the cooling water by pumping the pipet bulb. You may want to add fresh cold ice/salt to the stem of the thistle tube condenser as the apparatus warms.

When sublimation is complete, remove from the Al block and allow to cool to room temperature. Remove the water/ice/salt from the thistle tube with the long pipet. Disconnect the vacuum carefully so that the inrush of air does not blow away the sublimed caffeine. Finally, shut off the water to the aspirator.

Carefully remove the cold finger condenser assembly and scrape off the caffeine onto a piece of preweighed (tared) weighing paper. Weigh the caffeine and record the mass to the nearest mg. Calculate the percentage recovery of sublimed caffeine from the crude caffeine. Determine the melting point of the purified caffeine. Record the MP range & the apparatus used. Calculate the % error for the MPs of the crude and sublimed caffeine. Calculate your overall percent recovery based on the masses of the tea bags recovered from the oven. Tabulate results for both steps in your lab book. Show calculations. Comment on the outcome.

