Chem 224 Exp.# 2 Caffeine Extraction Part 1 FS2017

volumes and amounts are approximate

TEA BAGS: Heat 40-50 ml distilled water to boiling on a hotplate in a 250 ml beaker. Weigh 3 dry tea bags, (remove paper tags, if present). Record mass to the nearest mg. Then add bags to boiling water. Hold tea bags under the water with a stir rod and cover the beaker with a watch glass. Hold temperature at boiling for 5-10 minutes, replacing any water that boils off, then remove bags and squeeze out excess water between 2 watch glasses into the beaker.

Save the bags. Place them in a labeled watch glass in the grey oven to dry. Allow to dry in oven overnight or until next week. Reweigh to determine the amount of material extracted.

Cool the water extract solution to room temp. An ice bath or running water over the outside will speed this up. This will turn light brown and cloudy on cooling. Add about 2 grams of K2CO3 to the aqueous extract and mix to dissolve. The solution should darken and become more clear as the tannins redissolve. This will prevent tannins from extracting into the CH2Cl2

Fill a 125 ml separatory funnel with water and check for stopcock leaks. Pour out the water. Obtain 25 ml of CH2Cl2 (dichloromethane) in a small erlenmeyer. Transfer the aqueous extract to the 125 ml separatory funnel and extract the aqueous solution with a 6-8 ml portion of CH2Cl2, (dichloromethane), under a hood, by gently rocking for 30 seconds. Allow the layers to separate. The bottom CH2Cl2 layer should be nearly clear. Drain off the bottom layer into a 50 ml beaker. If an emulsion is present, drain it off with the bottom layer. You will deal with the emulsion in a later step. Repeat the extraction twice more with 6-8 ml portions of CH2Cl2 each time, combining the CH2Cl2 extracts in the 50 ml beaker. The brown aqueous layer remaining in the sep funnel will be discarded in the halogenated waste.

Pour the CH2Cl2 extract through phase separation paper in a funnel into a 50 ml erlenmeyer flask. Cover the funnel with a watch glass to reduce evaporation of CH2Cl2. The paper will retain any brown aqueous material allowing only the CH2Cl2 to go through. Rinse down the paper with another 1-2 pipets of CH2Cl2 to remove any caffeine crystallized on the paper due to evaporation of CH2Cl2 during the filtration.

Dry the CH2Cl2 extract with 3-4 spatula-fulls of anhydrous Na2SO4. Adequate drying agent has been added when it no longer clumps together. Pour off or pipet the dried CH2Cl2 into a small tared (weighed) beaker. Rinse the residual Na2SO4 with 2-3 ml of CH2Cl2 and add this to the tared beaker.

Evaporate the CH2Cl2 using a hairdryer or hotplate into a hood. Monitor this <u>carefully</u>. IMPORTANT: Do NOT continue to heat the beaker when the last of the solvent boils off or you may sublime off some of the caffeine. Also be careful the hairdryer does not knock over the beaker.

Reweigh the beaker to determine the amount of crude caffeine extracted. Record the mass to the nearest mg. Run a MP on a portion of the material. Record the MP temperature range and the brand name & number of the MP apparatus used.

Store your caffeine in this labeled beaker until next week, when it will be transferred to the conical vial for sublimation. Both 224A & B sections are sharing the same drawer, so it is important NOT to disturb the caffeine or tie up the 5ml vial for the other lab section. The beaker label should include your full name & section #.

Purification of caffeine by sublimation will be done next week.