EXPERIMENT 7A: EXTRACTION AND PURIFICATION OF CAFFEINE FROM TEALEAVES

Experiment 7A IS AN INDIVIDUAL WORK & REPORT

IMPORTANT: Make sure that you follow the proper laboratory safety protocol (refer to the course syllabus) BEFORE going to the lab. You will be dismissed from the laboratory if you do not follow the proper safety protocol.

There is ONLY ONE pre-lab report for this experiment even though this is a 6-hour lab (two lab periods).

On-line Resources AND TUTORIALS For This Experiment: Please click on the following links to view the following:

Theory of Extraction
Extraction Tutorial
Thin Layer Chromatography (TLC) Tutorial

PRE-LAB REPORT GUIDELINES (PRE-LAB REPORT MUST BE WRITTEN INSIDE YOUR LAB NOTEBOOK)

NOTE that ONLY parts 1, 2 and 3 are DUE on Tuesday, April 12 & Wednesday, April 13

& Part 4 (study questions) is DUE on Thursday, April 14 & Friday, April 15

1. *Introduction* - list the goal(s) of the experiment as well as any experimental techniques that will be used during the experiment for purification and/or identification. Include references and the changes to the procedure noted below:

Changes in the lab procedure

On page 79, make the following changes to the LAST SENTENCE in the first paragraph:
Instead of **"Add the 1-propanol from the first two extractions to the funnel"**

Replace it with **"Add the 1-propanol from the last two extractions to the same beaker used previously to store the other 1-propanol layers."**

Also on page 79, in the second paragraph, the drying agent will be **anhydrous sodium sulfate and NOT** anhydrous sodium sulfite.

On page 79, in the middle of the third paragraph replace

"Repeat the extraction 2 more times with an additional 10 mL of acetone each time...."
with
"Rinse the NaCl precipitate in the beaker twice with an additional 10 mL of acetone each time...."

2. *Flowchart* - Prepare a flowchart of procedures **AND** show the location of the caffeine throughout each step of the experiment in the flow chart. Make sure you also **REFERENCE** the procedures.

3. Set up BLANK DATA tables for recording any numerical values AND in-lab observations. (Use a *NEW* page for the tables).

IMPORTANT: You MUST record ALL the observations during the lab period for this experiment in your notebook. Failure to do so will result in no credit for your lab technique as well as part of your post-lab report.

NOTE: Make sure that you look for organization of ideas and not just a copy of the procedure in boxes.

IMPORTANT: Make sure that you follow the proper laboratory safety protocol (refer to the course syllabus) BEFORE going to the lab. You will be dismissed from the laboratory if you do not follow the proper safety protocol.

4. *Study Questions*

Complete the following questions. ***(MUST SHOW ALL WORK FOR FULL CREDIT)***.
Note: K_p is equivalent to α (refer to lecture guide for definitions and the necessary equations to complete the study questions).

(a) Which three precautions have to be taken when removing solvents like propanol and acetone?

(b) A compound distributes between water and diethyl ether with K_p=6. If 1.0 g of the compound were dissolved in 40 mL of water, how much compound could be extracted by **TWO** 20-mL portions of diethyl ether? How much could be extracted by **FOUR** 10-mL extractions?

(c) If the value of K_p is 0.5 for the distribution of a compound between hexane and water, and equal volumes of the two solvents were used, how many extractions of the aqueous layer will be required to recover at least 97 % of the compound?

(d) Calculate the percentage of a compound that can be removed from liquid phase 1 (being water) by using **ONE** to **FOUR** extractions with a liquid phase 2 (being an organic solvent). Assume that K_p = 5 and the volume of phase 2 equals to 20 % that of phase 1.

(e) Warfarin has a pK_a=5.3 and a log K_{ow}=2.7. How do you expect the log K_{ow}-value to change if the pH-value is (i) decreased to pH=2 (ii) increased to pH=10?

NOTE: You MUST turn in this pre-lab report to your TA at the BEGINNING of the lab on the dates listed above even if you finished the entire experiment in ONE lab period. Pre-lab reports will not be accepted at other times or locations.

Report Guidelines for EXTRACTION AND PURIFICATION OF CAFFEINE FROM TEA LEAVES EXPERIMENT

POST-LAB REPORT GUIDELINES - THIS IS AN INDIVIDUAL REPORT

Post-lab report must be written inside your lab notebook.

1. Abstract

Outline the objective of the experiment as well as any experimental results that obtained. Include any technique that you used in the lab to isolate, purify and analyze the experimental products.

2. Data, Observations and Data Analysis

a. Recopy all observations as well as numerical data in an organize manner.

b. Use equations to help explain any chemical reactions that occur during the experiment *and* explain the phenomenon (physical changes) behind each step of the observation

c. Calculate the percentage caffeine that you recovered from tea.

d. Mass spectrum of caffeine:

I. Identify the base peak
II. Identify the molecular ion peak
III. Does the molecular ion peak confirm the present of caffeine? Explain.

e. UV-Vis Spectrum of Caffeine: Calculate the molar extinction coefficient (ε) for caffeine. Show all your work. Note: Assume the diameter of the cell holder to be 1 cm.

Note: We will discuss mass spectroscopy later on in the course. At this point, use the following definitions to answer the questions related to mass spectrum of caffeine.

Base peak is the signal on the mass spectrum that has the highest intensity with respect to the y-axis.

Molecular ion peak refers to the signal on the mass spectrum that gives you the molecular mass of the sample. Simply look at the x-axis (which is the mass) and look for a signal that gives you the molecular weight of caffeine.

f. Calculate the R_f values of **BOTH** the pure caffeine and the crude caffeine. Show all your work.

Note: Read the Thin Layer Chromatography chapter in Mohrig (Techniques on Organic Chemistry, 3rd or 4th edition). Pay special attention to page on how to find R_f value and what it means. Concepts behind TLC will be discussed later on in the course.

3. Conclusion

a. Compare your percentage caffeine value to the reported value of about 2-4 % caffeine content in tealeaves. Explain what may cause the percentage to be differed (i.e. where the loss of caffeine may come from during the experiments).

b. Compare experimental infrared spectrum to literature infrared spectrum (provided with the experiment 7B worksheets). What can you conclude about the purity of your product?

Note: If you do not have the literature caffeine infrared spectrum, go to the following Web site and download the infrared spectrum

http://sdbs.riodb.aist.go.jp/sdbs/cgi-bin/cre_index.cgi?lang=eng

Do a **NAME** search on caffeine and there should be a hyperlink for the infrared spectrum of caffeine. Use the spectrum that was obtained in "KBr" (see upper right corner of the spectrum!).

c. Interpretation of caffeine product composition and purity from TLC results. **Explain** all your reasoning.

NOTE: Before answering part 3., refer to the notes on TLC listed on the previous page of this guideline.

Attach ALL the spectra & the experimental TLC plate with your post-lab report.