

Experiment 2 Report (40 pts)

This assignment is to be submitted individually and should be your own work.

Directions: Make a copy of this document and save it to your Google Drive. Type into the designated areas. Boxes can be expanded, but your answers must be in boxes. Answers in the tables can be words or phrases and do not have to be complete sentences. Handwritten reports will have a 1 pt deduction. This includes structures.

All answers for questions not in tables must be answered in complete sentences. Points will be deducted for excessively wordy answers or changing the format of the report, although table boxes can be made bigger if necessary. Avoid having tables or responses to questions going from one page to the other to facilitate grading.

Upload your report as a .pdf to Gradescope and make sure to carefully mark which questions are on each page. Please note that you can be asked for access to the Google Doc version of this assignment if there is suspicion of cheating or plagiarism.

1. What was the purpose of Experiment 2? You could write this all as one purpose or separate it by week. This should be related to the actual experiment, not the learning goals (for example - do not write something like “learn about liquid-liquid extraction and recrystallization). (3 pt)

The purpose of experiment 2 was to compare the techniques of liquid-liquid extraction and recrystallization to determine the efficiency of both methods and how effective one is compared to another. It is also to determine the difference in their application given their procedures as well as their pros and cons. Week 1 focused on the process of liquid liquid extraction while week 2 focused on recrystallization

2. Please complete the information below regarding the mixtures you used in this experiment. (2 pts)

Mixture	Major Component (Name)	Major Component Literature Melting Point (°C)	Impurity (Name)
B1	Phenanthrene	99 °C	Methyl Orange
B2	Phenanthrene	99 °C	Benzil

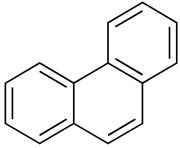
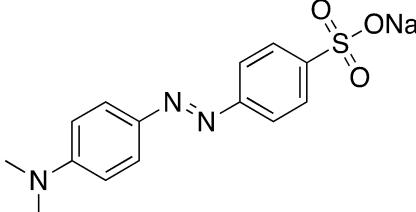
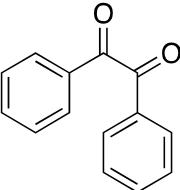
Cite the reference source (Merck, CRC or KCT) used for the literature value in the table. Do not use Reaxys for this assignment. (0.5 pts)

85-01-8 . Physical Constants of Organic Compounds. In *CRC Handbook of Chemistry and Physics* [Online], 104th ed; Haynes, W. M., Ed.; CRC Press, 2023. <https://hbcp.chemnetbase.com/contents/InteractiveTable.xhtml?dswid=-3722> (accessed February 28, 2024)

547-58-0. Physical Constants of Organic Compounds. In *CRC Handbook of Chemistry and Physics* [Online], 104th ed; Haynes, W. M., Ed.; CRC Press, 2023. <https://hbcp.chemnetbase.com/contents/InteractiveTable.xhtml?dswid=-3722> (accessed February 28, 2024)

134-81-6. Physical Constants of Organic Compounds. In *CRC Handbook of Chemistry and Physics* [Online], 104th ed; Haynes, W. M., Ed.; CRC Press, 2023. <https://hbcp.chemnetbase.com/contents/InteractiveTable.xhtml?dswid=-3722> (accessed February 28, 2024)

3. Mark the types of IMFs each compound below can participate in with **ethyl acetate** using an X. (6 pts)

Structure	ion-dipole	H-bonding	dipole-dipole	London Dispersion
 phenanthrene - major component in B1 and B2				X
 methyl orange - B1 impurity	X		X	X
 benzil - B2 impurity			X	X

4. Enter your UV-Vis data and observations for the B1 and B2 extractions in Week 1. (5 pts)

	B1 Extraction	B2 Extraction
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Observation of solution before water extraction	Orange Like coloration 2 separate layers with organics on top	Green like coloration, 2 separate layers with organics on top
Observation of 1 st aqueous layer	Mostly orange	Some what green
Absorbance of 1 st aqueous layer (include wavelength)	466 nm ~ 1.26 (we averaged 2 values, the wavelengths of 464.46 nm and 467.84 nm)	407 nm - 0.029
Observation of 2 nd aqueous layer	Less Orange, much more clear	Almost clear.
Absorbance of 2 nd aqueous layer (include wavelength)	466 nm ~ 0.48	407 nm - 0.08

5. Melting point analysis and % Recovery

Fill in the table blank cells in the table below. (Remember: melting points should be reported as ranges.) (4.5 pts)

Sample	Initial mass	Mass recovered	% Recovery	Experimental Melting point, °C
B1 - Extraction	0.14g	0.066g	47%	93 - 96°C
B2 - Extraction	0.04g	0.025g	62.5%	50 - 72°C
B1 - Recrystallization	0.1g	0.0671g	67.1%	81 - 87°C
B2 - Recrystallization	0.1g	0.050g	50.0%	85 - 92°C
85-01-8	---	---	---	88 - 97°C

Show **one** of the calculations for % recovery. Show all your work. (1 pts)

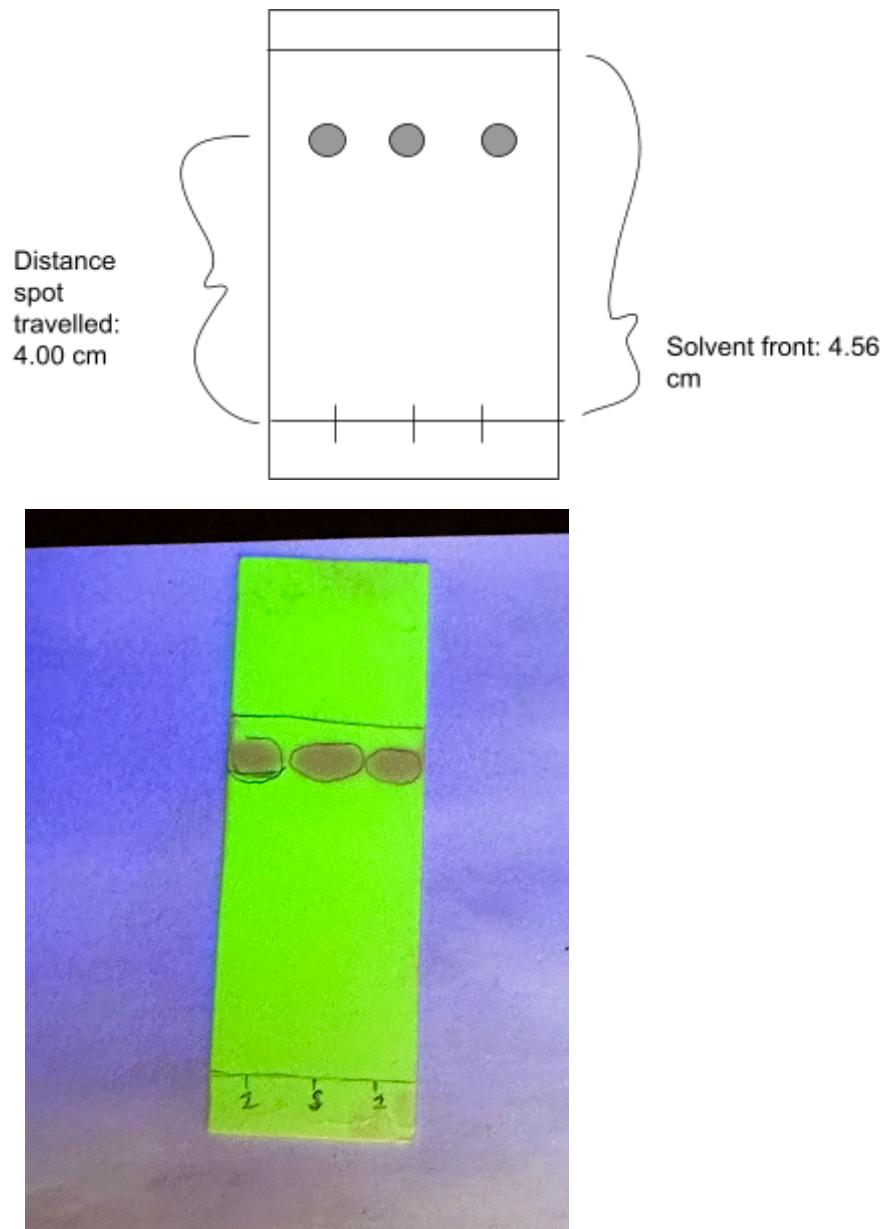
$$(\text{experimental yield} / \text{theoretical yield}) \times 100\%$$

So for Recrystallization B2: $(0.050\text{g} / 0.1\text{g}) * 100\% = 50\%$

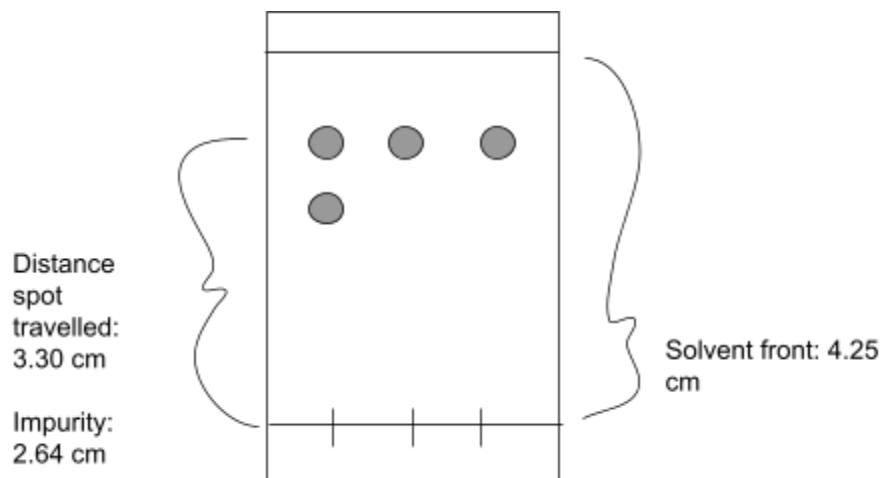
6. TLC Data

Insert or draw a picture of your TLC plate in week 2. Show the measurements and R_f values either on your drawing or near your picture. (3 pt)

$$B1: R_f = 4.00\text{cm}/4.56\text{cm} = 0.877$$



$$B2: R_f = 3.30\text{cm} / 4.25\text{cm} = 0.776$$



Explain what your TLC tells you about the purity of the isolated phenanthrene? This should be based on R_f values (which tell you about the identity of the spots) and the numbers of spots in each lane. (3 pts)

The TLC allows us to determine how pure our isolated phenanthrene sample based on Rf values and the number of spots. The Rf value allows us to confirm the polarity of our substance with the standard Phenanthrene and in our case confirm that our unknown has the same polarity level as Phenanthrene. In addition, impurities will separate out and form their own spots on the TLC as seen with the B2 and this tells us how pure of sample actually is. In this case, B1 is very pure with one spot while B2 has 2.

7. Summarize how well extraction worked to purify phenanthrene from mixtures B1 and B2 based on your data (UV-Vis, observations, melting point, TLC, and recovered mass). Be specific in terms of incorporating actual pieces of your data and how this makes sense based on the structures and IMFs mentioned in question 3. (4 pts)

For the B1 mixtures, it appears our extraction methods were quite successful. From the UV-Vis observations, we noted very high absorbance values of 1.26 and 0.48 for our unknown solid, which coincides with our observations of the orange coloration of our extractions. However, we had an error within our procedure regarding the amount of our initial mass, we did 0.14 grams, and thus had a recovered mass of only 47%. As this is human error rather than experimental, we ought to consider it but not weight this metric as heavily. However, this also makes sense based on the structure and IMF of Phenanthrene compared to Methyl Orange. Phenanthrene is non-polar while Methyl Orange is polar and can dipole-dipole with ethyl acetate meaning it can be extracted quite easily.

B2 on the other hand, while also successful was less so compared to B1. We had much lower absorbance values of our impurity of 0.029 and 0.08 as well as a lighter green color observed. We had a rather low extraction recovered mass of 67.1%. Interestingly, Benzil can also dipole-dipole with acetate, which means it ought to separate easily, but it appears our technique just may not have been as good as on our B2 as our B1.

8. Summarize how well recrystallization worked to purify phenanthrene from mixtures B1 and B2 based on your data (observations, melting point, TLC, and recovered mass). Be specific in terms of incorporating actual pieces of your data and how this makes sense based on the structures and IMFs mentioned in question 3. (4 pts)

For our B1, the TLC indicates that our recrystallization approach from the extraction in week 1 left us with a very pure sample because we only had 1 spot. Furthermore, our recovered mass between the 2 weeks was ok at about 67% respectively. This also makes sense based on the structure and IMF of Phenanthrene compared to Methyl Orange. Phenanthrene is non-polar, while Methyl Orange is polar and can dipole-dipole with ethyl acetate meaning it can be extracted quite easily. However, for recrystallization, it relies on Phenanthrene precipitating out of the solution of Ethyl acetate, which may not have happened fully given the time allotted for it.

As for our B2, our recovered mass was also lower at about 50% recovery, and our TLC had two dots which indicates impurity was left. Overall, this does mean our B2 was rather ineffective with regard to the use of recrystallization for purification. This doesn't add up with the difference in structures and IMF between Phenanthrene and Benzil so it is likely human error that caused such results.

9. Reflect on your experience in the lab this week. What went well and what might you do differently next time? This could be in terms of how you prepared, how you managed your time in lab, or something more technical. (4 pts)

Overall, I felt that lab this week was quite fun and went very well. Definitely, my communication with my teammates has improved and our ability to multitask and divide and conquer has improved significantly as well. Next time, I think I will be more organized with my notes since it was a pain to follow them this report. I will also have data tables prepared to facilitate this organization process.

By digitally signing below, I verify that all data collected, observations, and answers provided on this lab report are my own and are not duplications of another student's report either at Brown or at other institutions.



Digital Signature:

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