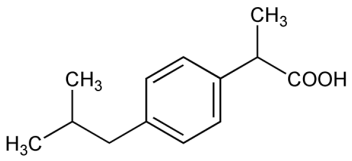
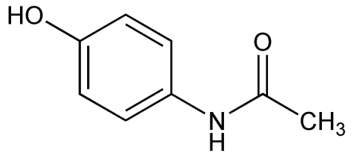
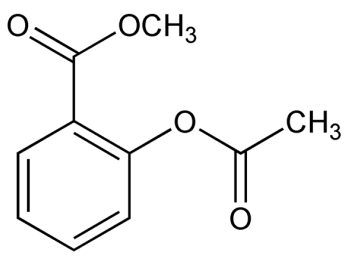
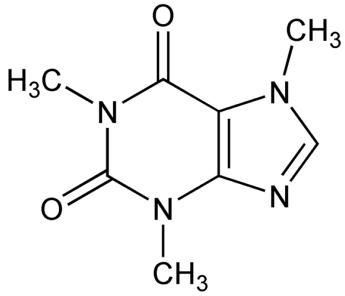
Experiment 4 Laboratory Report (35 pts)

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. 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.

Question 1: Complete the table below using one of the reference handbooks. (4 pts)

Name	**Structure**	**Melting Point (°C)**
ibuprofen		75-77 C*
acetaminophen		167.85-168.05 *C

acetylsalicylic acid		118 – 140 *C
caffeine		234 – 239 *C

Citation: (1 pt)

Top is temperature

Bottom is picture

15687-27-1. Basic Physical Properties of Chemical Compound, In ***Knovel Critical Tables*** **[Online], 2nd ed.; Knovel Corporation, 2008.; from https://app.knovel.com/hotlink/itble/rcid:kpKCTE000X/id:kt002VLXT1/knovel-critical-tables/basic-physical-properties (accessed April 11, 2024)**

15687-27-1. The Merck Index Online; Royal Society of Chemistry, 2024; M11390. https://merckindex-rsc-org.revproxy.brown.edu/monographs/m11390(accessed April 11, 2024)

103-90-2. Basic Physical Properties of Chemical Compound, In ***Knovel Critical Tables*** **[Online], 2nd ed.; Knovel Corporation, 2008.; from https://app.knovel.com/hotlink/itble/rcid:kpKCTE000X/id:kt002VLXT1/knovel-critical-tables/basic-physical-properties (accessed April 11, 2024)**

103-90-2. The Merck Index Online; Royal Society of Chemistry, 2024; M11390.

https://merckindex-rsc-org.revproxy.brown.edu/monographs/m11390(accessed April 11, 2024)

580-02-9. Basic Physical Properties of Chemical Compound, In *Knovel Critical Tables* [Online], 2nd ed.; Knovel Corporation, 2008.; from https://app.knovel.com/hotlink/itble/rcid:kpKCTE000X/id:kt002VLXT1/knovel-critical-tables/basic-physical-properties (accessed April 11, 2024)

580-02-9. The Merck Index Online; Royal Society of Chemistry, 2024; M11390. https://merckindex-rsc-org.revproxy.brown.edu/monographs/m11390(accessed April 11, 2024)

58-08-2. Basic Physical Properties of Chemical Compound, In *Knovel Critical Tables* [Online], 2nd ed.; Knovel Corporation, 2008.; from https://app.knovel.com/hotlink/itble/rcid:kpKCTE000X/id:kt002VLXT1/knovel-critical-tables/basic-physical-properties (accessed April 11, 2024)

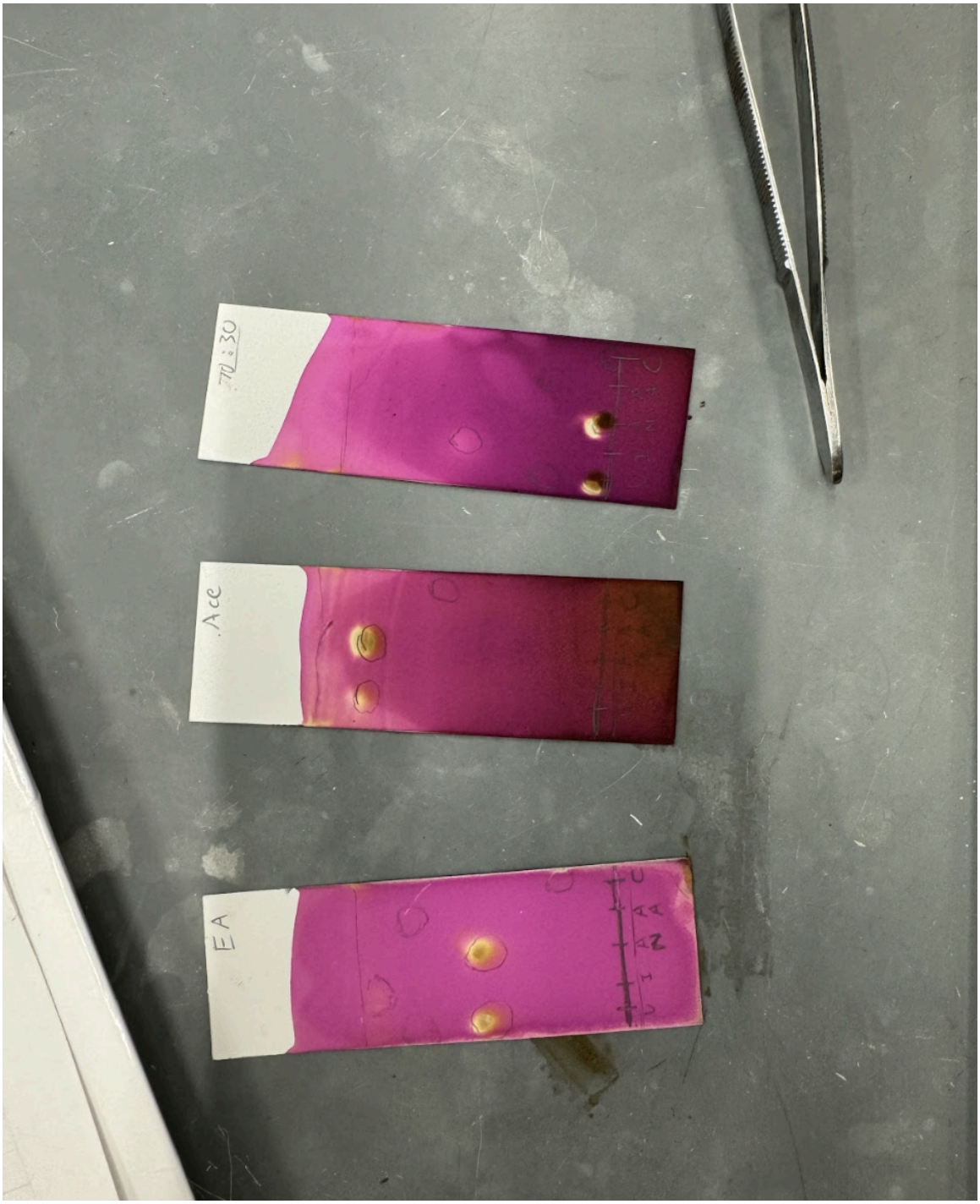
58-08-2. The Merck Index Online; Royal Society of Chemistry, 2024; M11390. https://merckindex-rsc-org.revproxy.brown.edu/monographs/m11390(accessed April 11, 2024)

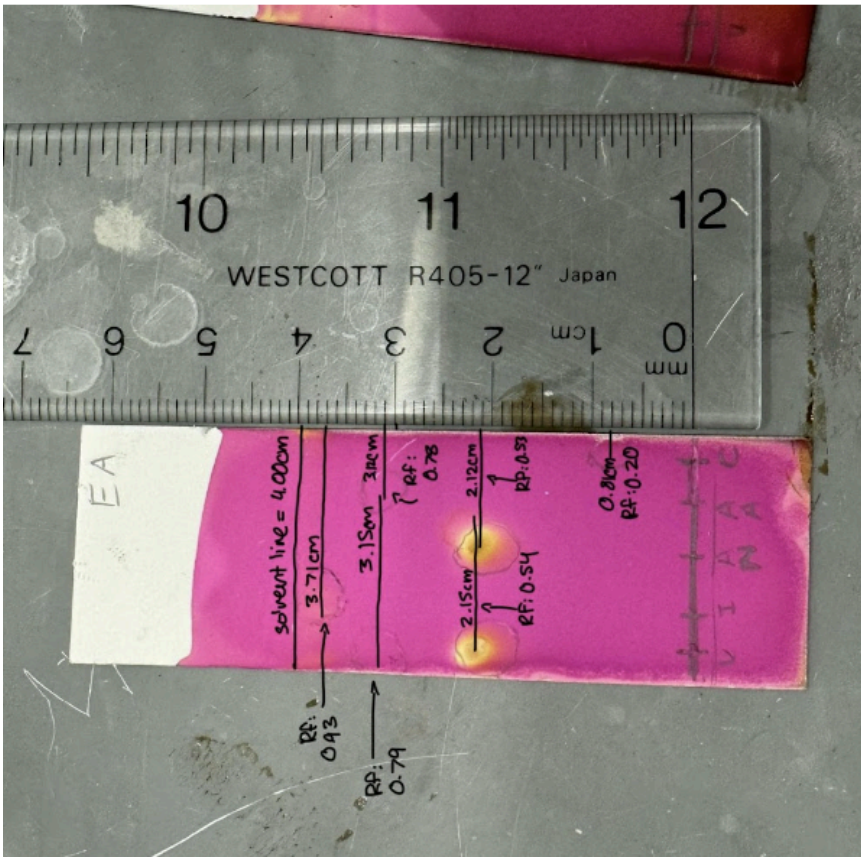
Question 2: Write your Week 1 procedure in a numbered list in the box below. You should have a similar level of detail as is in the procedures for experiments 1-3. (3 pts)

1. Take a TLC plate and label with a pencil line the start and the ticks indicating the position of spotting our unknown, ibuprofen, acetaminophen, acetylsalicylic acid, and caffeine.
2. Collect 50 mg of unknown and store in a test tube, make sure to keep for 2 weeks.
3. Spot the unknowns on the TLC plate in their appropriate position
4. Develop TLC with ethyl acetate within the TLC-developing chamber
5. Review via UV light after it is finished developing,
6. Calculate RF and note distance travelled by solvent and spots
7. Repeat 1-4 via 70:30 hexane and acetone as your developing agents. Use TLC stain if necessary to better visualize the TLC plate.
8. Record results as appropriate, as well as which solvent is best for developing the stain.

Question 3: Week 1 TLC Results

Insert pictures of each of your TLC plates (images or recreated drawings). (3 pts)


70:30
Ace
EA



Which solvent gave you the best separation of your mixture? Justify this based on your TLC plates above and solvent polarities. (2 pts)

The Ethyl Acetate gave the best separation of the mixtures. As seen on the TLC the ethyl acetate separated the different unknowns and the such the best based on their polarities. The acetone was too polar which everything moving way too much and thus there being little visible separate. On the other hand the 70:30 hexane: ethyl acetate didn't move at all and thus there was a lack of separation. However there was a possibility it wasn't the best as shown by our 2nd week's separation which may indicate that it is too polar a solvent.

Complete the table below for the solvent that gave you the best separation on your TLC plate. (2 pts)

Name	**R_f value**
ibuprofen	3.8/4 = 0.95
acetaminophen	2/4 = 0.5
acetylsalicylic acid	3.2/4 = 0.8
caffeine	0.8/4 = 0.2

Based on your TLC results, which components are in your mixture? Justify this based on your data in the table above. (2 pts)

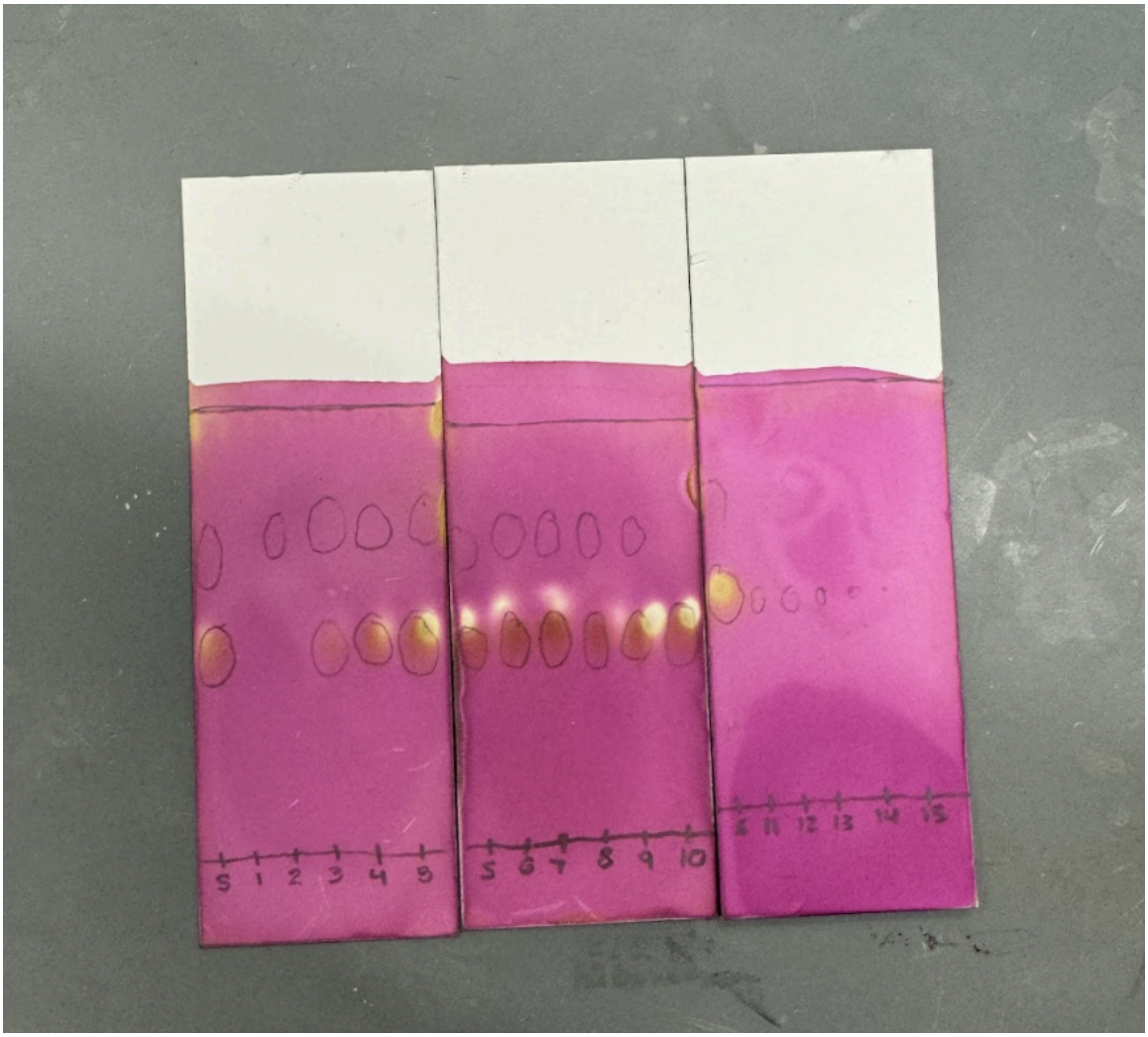
Based on the TLC results, I am quite confident that our mixture is a combination of acetaminophen and acetylsalicylic acid. The Rf values for acetaminophen and acetylsalicic acid line up very well with our unknown's RF values of about 0.5 and 0.8 respectively. At least based on polarity, the results seems to line up. The acetaminophem was the unpolar one which matches its chemical makeup, and the acetylsalicyclic acid was much more polar which matches its structure.

Question 4: Column Chromatography TLC Results

Complete the table below (0.5 pt)

Mass of mixture you started your column with (mg)	48 mg
Solvent used for column	Ethyl acetate.

Insert images of your TLC plates from your column below. (1.5 pts)



Discuss which fractions contained each component, and which you combined to recover each component based on your TLC (3 pts).

Most likely, fraction 2 contained the more polar acetylsalicylic acid while fraction 10-13 and onwards had the less polar acetaminophen. These were combined appropriately to extract each part individually via evaporation through nitrogen gas stream.

Question 5: Column Chromatography Recovery Results
Complete the table below: (2 pts)

Component	**Fractions Combined (#s)**	**Mass of empty test tube (g)**	**Mass of test tube + product (g)**	**Mass of product (g)**
acetylsalicylic acid	2	3.531g	3.542g	0.002g
acetaminophen	10-13	3.528g	3.547g	0.004g

Calculate the percent recovery of each component assuming that the mixture you started with was a 50:50 mix of each component. (% recovery = mass recovered/mass used * 100) (2 pts)

Component 1: 0.002/ 0.024 * 100 = 8.3%

Component 2: 0.004/0.024 * 100 = 16.7%

Discuss your % recovery for each compound. Was it good? Are there any explanations for why your recovery was low if this is the case? (2 pts)

Our % recovery for each compound was incredibly low. This makes sense cause with only spots 2 and 10 available for any recovery we were bound to get a very low yield out. This is rather unfortunate, considering we believed Ethyl acetate was the best option we had as our solvent. From the TLC, it appears that ethyl acetate wasn't non-polar enough for the effect we wanted.

Question 6: Column chromatography melting point results (2 pt)

Component	**Standard Melting Point (°C)**	**Recovered Compound Melting Point (°C)**
acetaminophen	167.85-168.05 *C	140-148*C
acetylsalicylic acid	118 – 140 *C	125-131*C

Discuss the purity of your recovered components based on their melting points. (2 pts)

Our melting points where quite similar with the standard melting points as reported in the literature. This would indicate that our analysis was performed rather well in terms of gaining the maximal purity possible. Looking more into it, our recovered melting point was off by about 20 degrees celsius for the acetaminophen and for acetylsalicyclic acid it was quite spot on. This makes sense for the acetaminophen since that was our 10-13 which likely had some contamination, particularly since we got so little of the compound itself from the extraction/recovery. However while not too pure, it was pure enough.

Question 7: Reflect on how your lab experience has changed over the semester. How have you grown? What areas remain for improvement in future lab work in classes or in conducting research? Did your perception of working in a lab or your ability to do so change over the semester? (*At least 3 sentences; 3 pts*)

Overall I feel that the lab experience has been a positive one. I feel like I grown more adept with the use of lab techniques and working with my colleagues in order to conduct good science. However I do see myself becoming more organized as an individual so I can do better research and lab work. Overall, I have a much more positive idea of working in a lab and I am very excited to continue to do so as the semester progresses and in next years ORGO class.

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: Jason Lin

Banner ID: B01881807