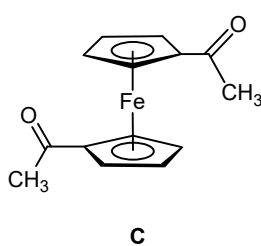
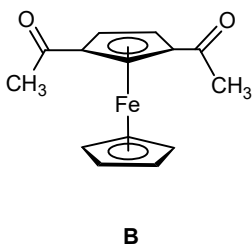
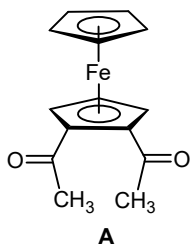


Questions to answer for the "Friedel-Crafts Acylation of Ferrocene" lab report

Name: _____

1. Shown below are three possible structures for diacetylferrocene. How could NMR (proton or C-13) be used to differentiate between isomers A, B and C? Note that the rings can freely rotate.



2. What does it mean to reflux a reaction? (i.e., why is it used, and how does it work?)
3. Why do bubbles form when bicarbonate is added? Write a balanced equation showing how a gas is produced.
4. The crude product is collected by vacuum filtration. Why so you think the product is not soluble? (What solvent is present?)
5. Why is H₂O solvent used to dissolve the crude product in a recrystallization process?
6. The polymeric byproducts are not soluble in hexane, and they are removed by gravity filtration. Why use fluted filter paper? Would it be okay to use vacuum filtration here? Why or why not?
7. After cooling, the pure acetylferrocene crystals can be collected and analyzed. At this point in the experiment, what could be done to improve the yield?

8. How can the melting point of a sample serve as an indication of its purity? (In this case, because it is a known compound, there are TWO things to look for.)
9. Consider the likely components present in the filtered reaction mixture: desired product (**acetylferrocene**), side product (**diacetylferrocene**), and unreacted starting material (**ferrocene**). Which compound is expected to have the **highest R_f** on a silica gel TLC plate? Explain.

Which compound is expected to have the **lowest R_f** on a silica gel TLC plate? Explain.

The TLC plate shows a mixture with many spots, and all three components are present! To separate the three components, we used column chromatography. All three compounds are colored, so it is easy to keep track of them as they move through the column (packed with silica gel).

10. First, a yellow band is eluted from the column with hexane. Which of the three compounds is expected to come off the column first? Why?
11. Why is the method being used called “flash” column chromatography?
12. To elute the next component, a different solvent is used (1:1 mixture of ether and hexane). Draw the structures of the two solvents, and explain how the addition of ether affects the overall polarity of the eluent.
13. How does the addition of ether affect the rate of travel through the silica gel for the remaining compounds? Explain your reasoning.

14. A second colored band is eluted with 1:1 ether/hexane. Of the three compounds known to be in the mixture, which one is expected to be collected second? Why?
15. Finally, a third colored band is collected by using ether as the eluent. Once again, explain how this change affects the polarity of the solvent and what effect it has on the compound's rate of travel.
16. Of the three compounds known to be in the mixture, which one is expected to be collected last?
17. Three spots have been circled in the "crude product" mixture **C**.
Provide a sketch of the TLC plate, identify each of the three spots, and briefly explain how you made the determination.
18. What component was isolated in fraction **1**? What evidence do you have to support your answer?
19. What components were isolated in fractions **2** and **3**?
20. Did the column successfully separate the mixture? Explain.