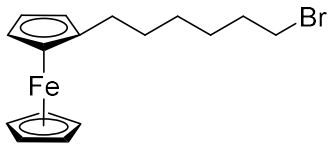
Experiment 17

Microscale Column Chromatography: Separation of Compounds

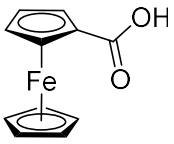
Study Questions

1) Consider the compounds ferrocene, acetylferrocene, and diacetylferrocene.
   a. Which is the most polar and which is the least polar? **Answer:** Diacetylferrocene is most polar; ferrocene is least polar.
   b. Which will have the lowest R_f in TLC? **Answer:** Diacetylferrocene.
   c. Which will elute first in column chromatography? **Answer:** Ferrocene.

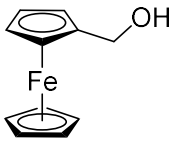
2) Shown below are some other ferrocene derivatives.



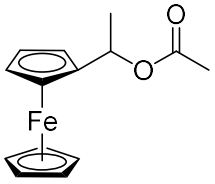
(6-Bromohexyl)ferrocene



Ferrocenecarboxylic acid



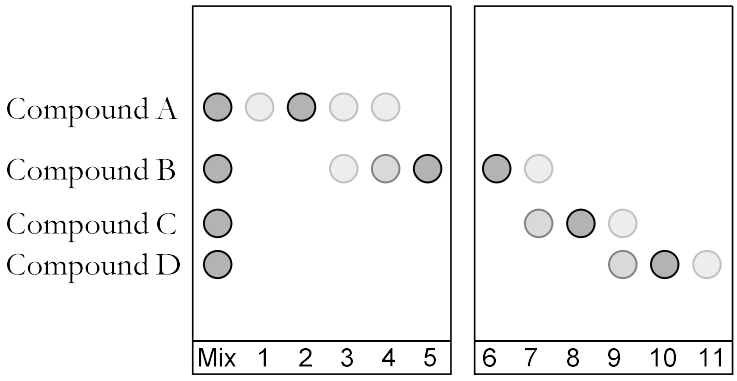
Ferrocenemethanol



(1-Acetoxyethyl)ferrocene

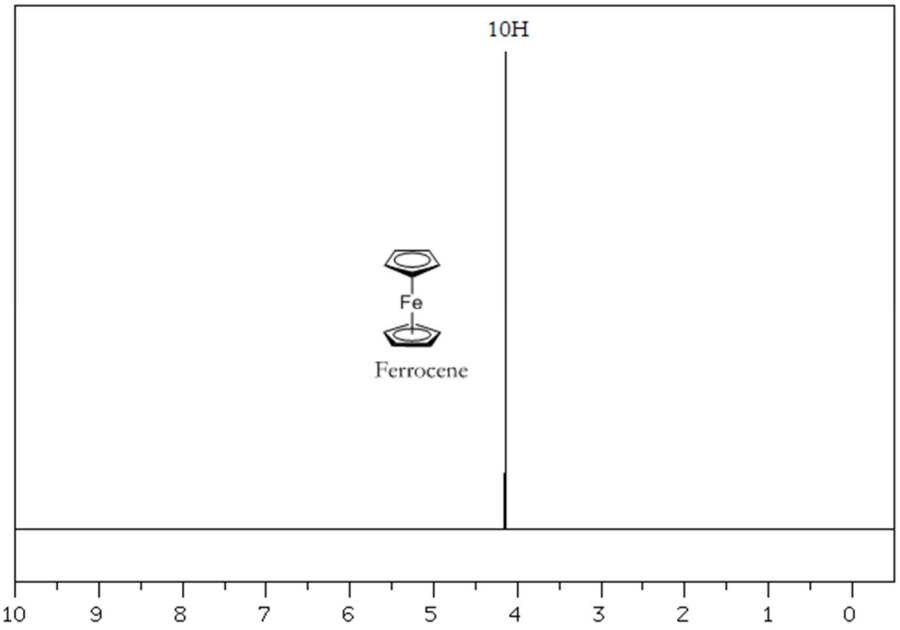
   a. Rank these compounds from most to least polar. **Answer:** Ferrocenecarboxylic acid, ferrocenemethanol, (1-acetoxyethyl)ferrocene, **(**6-bromohexyl)ferrocene.
   b. Which will have the lowest R_f in TLC? **Answer:** Ferrocenecarboxylic acid.
   c. Which will elute first in column chromatography? **Answer: (**6-Bromohexyl)ferrocene.

3) A chemist found that a mixture of four components (Compounds A–D) could be separated on a silica gel TLC plate using 10% diethyl ether in hexanes as the eluting solvent (see "original mixture" far left plate in the figure below). The mixture was then chromatographed on a silica gel column eluted with this same solvent mixture and 11 fractions of 15 mL each were collected. Thin-layer chromatographic analysis of the various fractions (1-11) under the conditions stated above gave the results shown in the figure below:



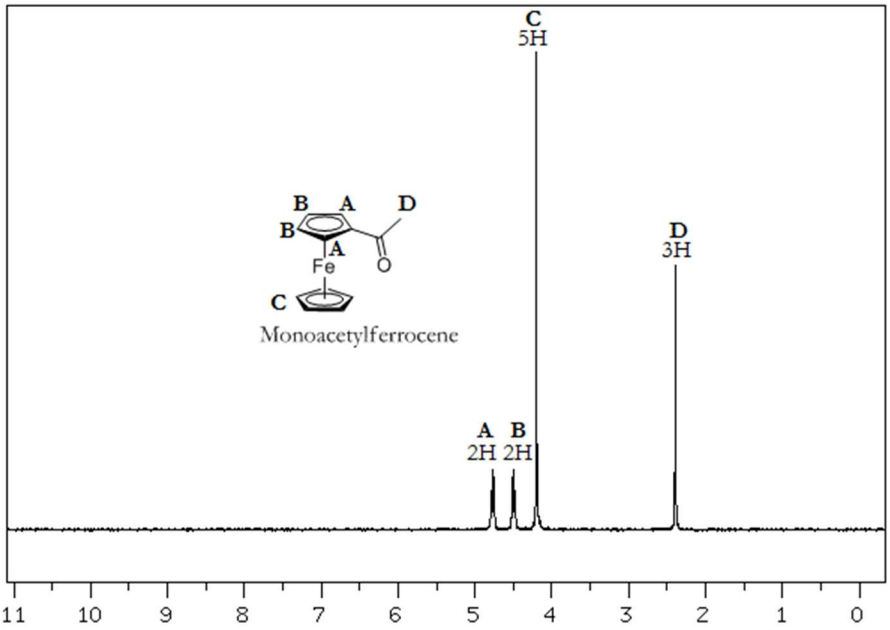
   a. According to these results which fractions should be combined to give pure samples of A, B, C, and D? **Answer:** Combine 1 & 2 for A; 5 & 6 for B; 8 for C; and 10 & 11 for D.
   b. Which fractions contain more than one component? Indicate for these 'mixed' fraction numbers what components of the original mixture are present. **Answer:** 3 & 4 contain both A and B; 7 contains both B and C; 9 contains both C and D.

4) The NMR spectrum of ferrocene is shown below (taken from SDBS). Explain why the aromatic protons show a single band at 4.2 ppm.



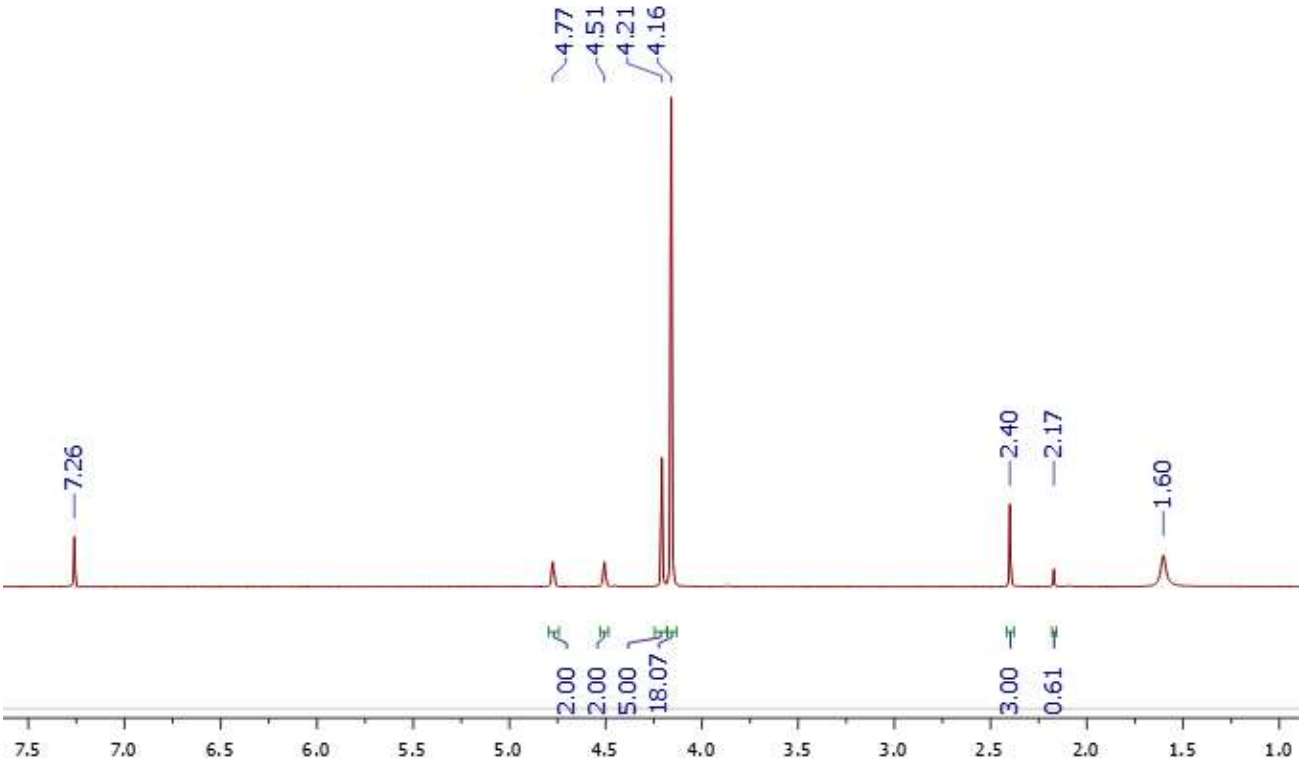
Answer: Because they are all equivalent.

5) The NMR spectrum of monoacetylferrocene is shown below (taken from SDBS). What would the NMR of diacetylferrocene look like?



Answer: The "c" peak would disappear and the others would double in intensity.

6) A student performed this experiment and obtained the NMR spectrum shown below. What other impurities are present? What is the molar ratio of ferrocene to monoacetylferrocene in the crude product? What percent of the crude product is ferrocene vs. monoacetylferrocene?



Answer: Impurities present are $CDCl_3$ (7.26 ppm), acetone (2.17 ppm), and water (1.60 ppm). For product ratios, the two easiest peaks to use are at 4.21 (5H in monoacetylferrocene) and 4.16 (10H in ferrocene).

$$\frac{\text{Moles of } \textbf{ferrocene}}{\text{Moles of } \textbf{monoacetylfer.}} = \left(\frac{18.07}{5.00}\right)\left(\frac{5\text{H}}{10\text{H}}\right) = 1.807$$

$$\text{Molar \% } \textbf{ferrocene} = \frac{1.807}{1.807 + 1} = 0.644 \text{ or } 64.4\ \%$$

$$\text{Molar \% } \textbf{monoacetylfer.} = \frac{1}{1.807 + 1} = 0.356 \text{ or } 35.6\%$$