

Fluorenonol polarity



**ASSIGN
BUSTER**

1. In table format, provide the R_f values you recorded for each of the 3 compounds in Part I. List compound on one axis of your table and solvent system on the other. If multiple spots were present for a compound, give R_f value for each component and state if the component was major or minor. | 100% hexane| 25% EtOAc in hexane| 10% EtOAc in hexane| Fluorene| 0.34 cm| 0.94 cm| 0.67 cm| 9-fluoreneol| 0.00 cm| 0.49 cm| 0.05 cm| 9-fluorenone| 0.00 cm| 0.69 cm| 0.21 cm|

2. Explain how R_f values were generally affected by polarity of the solvent/eluent systems. As part of your answer, rank the solvent/eluent systems in order of decreasing polarity (this can be done according to structure of the individual solvents and % composition of the mixtures). Also rank the 3 compounds in order of decreasing polarity (this compound ranking can be based on structure as well as on these TLC results). Explain each of your rankings. Compounds: 9-fluorenone, 9-fluoreneol, Fluorene Solvent: Ethyl acetate 10% in hexane, Ethyl acetate 25% in hexane, 100% hexane Polarity of each compound reacted differently to each of the solvents used.

As we know, 'like' interacts with 'like'. Fluorene has the least amount of polarity based on structure and when it was mixed with non-polar and slightly polar solvents, its mobile phase moved the furthest. Such that when Fluorene was put in 25% EtOAc, which is 75% polar, its mobile phase was the furthest, followed by 90% polar in 10% EtOAc in hexane, and its most solid phase was in 100% hexane. 9-fluoreneol is the highest polar compound based on structure and when it was mixed with a polar solvent its mobile phase was the slowest, therefore, it did not travel very far up the silica gel plate.

When 9-fluorenone was mixed in slightly polar solvent, 10% EtOAc, its mobile phase also did not travel very far. The only time 9-fluorenone mobile phase travelled up the silica gel plate is when it was combined with 25% EtOAc in hexane because 75% of the solvent was polar, and the remaining 25% allowed its mobile phase to move up the silica gel plate. 9-fluorenone is the second most polar compound when mixed with the above solvents. Based on its structure, when mixed with 100% hexane there was no mobile phase because polar compounds stick with polar solvents.

When 9-fluorenone was combined with 10% EtOAc, given about 90% polar, there was a slight mobile phase, moving just slightly up the silica gel plate. Its mobile phase moved the furthest when it was combined in 25% EtOAc in hexane, given about 75% polar, as the remaining 25% was able to move the compound up the polar silica gel plate. Based on the compounds and the R_f values, the compounds with larger R_f values means that the mobile phase traveled the furthest on the polar silica gel plate. Also, the compounds with the larger R_f value indicates that it was a less polar structure, as it interacts less strongly with the polar adsorbent on the TLC plate.

3. Based on structure, which of the 3 compounds can hydrogen bond to the silica gel? Explain your answer. Do compounds that can hydrogen bond to the silica gel generally have lower or higher R_f's than compounds that can not? The 9-fluorenone is the only one out of the three compounds that can hydrogen bond to the silica gel plate. Based on R_f values shown in question one, it had a lower R_f value, generally meaning compounds that can hydrogen bond to the silica gel will have the lower R_f values.

4. If you attempted to separate a mixture of the 3 compounds using liquid chromatography (see Ch. 18, Lab Techniques book), which compound would theoretically elute from the column first if 25% EtOAc in hexanes was used as eluent? Briefly explain your answer. Fluorene would elute from 25% EtOAc in hexane, since polar compounds are least likely to have a mobile phase dislodge the fastest or furthest from the silica gel plate, which is polar. If the eluent were more non-polar, then there would be a mobile phase further from the solid phase, as non-polar compounds are able to move away from polar solvents because those compounds are not attracted to its polarity.

5. Based on TLC, is your dibromide pure and different from trans-stilbene? In your answer give R_f values for each compound (make sure to give solvent system as well!). Do your TLC results prove that your product is really trans-stilbene dibromide? Explain. Based on my results, my dibromide compound is different than trans-stilbene.

When both compounds were mixed in 100% hexane and 10% EtOAc in hexane, the R_f values were not so different. In 100% hexane, trans-stilbene's R_f value was 0.39 cm and the dibromide had a value of 0.17 cm. In 10% EtOAc in hexane, trans-stilbene's R_f value was 0.44 cm and the dibromide had a value of 0.65 cm. Since there was 'lag' the R_f value can be considered ambiguous and it is not very telling if the compounds are in fact different. When mixed in 25% EtOAc in hexane, the R_f value differed by 0.02 cm (trans-stilbene value of 0.96 cm and dibromide value of 0.4 cm). The trans-stilbene and the dibromide do not share identical R_f values, but we can assume since there is only a 0.02 cm difference, it is not enough to say that they are two distinct substances; rather there is some mixture of trans-

stilbene in the dibromide, because some of the characteristics may be similar.

6. Briefly discuss your TLC results for the “ headache relief” tablets. Do the tablets seem to contain caffeine? Explain. Does TLC prove your answer? If not, what other “ evidence” would you need to convict Wiley Coyote (chemical evidence that is)? Pure Caffeine| Unknown tablet| Acetaminophen| Acetylsalicylic Acid| EtOAc/Hexane/AcOH66: 33: 1| 0. 01 cm| 0. 02 cm| 0. 26 cm| 0. 26 cm| Based on the above results, one could assume that the tablet did indeed contain caffeine. When the pure caffeine was combined with EtOAc/Hexane/AcOH the Rf value was 0. 01 cm and the unknown tablet had an Rf value of 0. 02 cm. Since there was a difference of 0. 01 cm, it is not clear to say that there was no caffeine present. Also, since the difference was so minimal between the two compounds it can be suggested that there were significant amounts of caffeine in the unknown tablet. mBased on the TLC results, the Rf values can conclude there was caffeine in the unknown tablet since there was not a big enough difference in values to say that the unknown tablet contained any other substances.

7. Explain the key/simple difference between gas chromatography and liquid chromatography with regards to eluent (see Lab Techniques book). The mobile phase is an inert gas, generally He₂ or N₂ does not compete with the stationary phase like TLC or LC, where the mobile phase actively competes with the stationary phase. The mobile phase does not interact with the compounds during GC, while they do interact in liquid chromatography and TLC.

8. Later, we will cover mass spectrometry, and will likely also mention a combined analytical technique called GC-MS (gas chromatography-mass spectrometry). A good overview of GC-MS can be found on Wikipedia (http://en.wikipedia.org/wiki/Gas_chromatography-mass_spectrometry). Briefly, but clearly, explain how GC-MS would be better at solving the “caffeine crime” compared to simple TLC. GC-MS combines gas-liquid chromatography and mass spectrometry to identify different substances within a test sample.

It is used to perform specific tests that can positively identify the actual presence of a particular substance in a given sample. Although both GC-MS and TLC can identify substances in a given sample, the TLC is a more non-specific test that could lead to false positives identification. If the R_f values are identical, one can assume that the substances are the same and the GC-MS would be better to differentiate the R_f values for two separate substances in a given sample, giving a more accurate identification.