**Identification of Unknowns**

**Data Presentation**

* Melting Point of Alcohol 🡪 87-90oC
* Melting Point of Ketone 🡪 78-80oC

1. Rf 95:5 (Hexanes: Ethyl Acetate) = Rf(A) =0.414; Rf(B) 0.12 ; **Δ**Rf = 0.294
2. Rf 90:10(Hexanes: Ethyl Acetate) = Rf(A) =0.509; Rf(B) 0.11 ; **Δ**Rf = 0.399
3. Rf 85:15 (Hexanes: Ethyl Acetate) = Rf(A) =0.610; Rf(B) 0.20 ; **Δ**Rf = 0.410
4. Rf 80:20 (Hexanes: Ethyl Acetate) = Rf(A) =0.880; Rf(B) 0.440 ; **Δ**Rf = 0.440

**Discussion and Results**

This particular lab, it was concluded that the Unknown 3279 was a mixture of a ketone and alcohol, 9-fluorenone and 9-fluorenmethanol (respectively). This lab required the student to isolate the ketone and alcohol, for which the column chromatography was conducted, where as samples were collected in glass vials. The first part of the lab was to determine the solvent system to be used in the column chromatography, which resulted to be the 95:5 (hexanes: ethyl acetate), since it met the criteria of being between 0.25 and 0.35 (**Δ**Rf = 0.294), and in addition to that it had a good enough separation between the spots (see diagram on the Observations’ sheet). The Spot A happened to be the ketone, as it would climb higher in the solvent system than the alcohol, because the more polar alcohol would attract the most to the TLC plate and move the slowest, thus Spot B being the alcohol. Hence, the 95:5 solvent system turned out to be the optimum for the column chromatography.

While performing the column chromatography, every 1mL was collected in different vials to run a TLC analysis in order to determine the presence of the ketone and alcohol. Theoretically, the ketone should be the first one to travel through the silica gel in the column chromatography, which after looking at the TLC analysis it most certainly did because of the high Spot (A) on the TLC analysis for vials 4-19. After the ketone disappeared, the only compound left to go through was the alcohol, for which a 50:50 (hexanes: ethyl acetate) was used, due to the fact that it was a lot more polar allowing the polar alcohol to move out of the column and into the vials. The spots from vials 23-27 were lower than the ones of ketone, similar to the Spot B. The two different sets of vials were then combined in two different vials, one of ketone and the other for the alcohol, then they were dried into solid alcohol and ketone crystals.

The Ketone’s melting point range was 78-80 oC. The closest literature melting point range of a ketone (from the lab manual) was 4-phenylcyclohexanone (78-80 oC). The alcohol had a melting point range of 87-90oC, the closest one from the lab manual was 4-biphenylmethanol (99-101oC). The melting point range of the ketone was extremely close to the literature value, however, the alcohol wasn’t. Due to lack of time, two melt temps were used instead of one. After concluding the melting point of the ketone, no more time could’ve been wasted thus another melt temp in the weighing room was used (it looked extremely old), and the alcohol melting point range was measured by the this one, resulting in an inaccurate measurement, of around 12 degrees Celsius. Although, the melting point data is what suggests that the compounds were 4-phenylcyclohexanone and 4-biphenylmethanol. However, further analysis, said otherwise.

An IR was taken for both the ketone and the alcohol in order to determine their presence and whether or not they were separated. IR Spectra Ketone: shows that there is a very sharp peak around 1700s, indicating that there is a carbonyl group in the compound. IR Spectra Alcohol: shows that there is a broad peak between 3550-3200, concluding that there is an alcohol group in that compound. However, the alcohol peaks are usually huge in length as well as wide. This indicates that there could be impurity in the substance. In addition to that error, the IR spectrum in the lab didn’t function perfectly, as the background base line was completely off, allowing room for error. Although, neither show signs of the other in their spectrums, signifying that they were separated efficiently.

The NMR, for the ketone, suggests that there are 8H in one proton (aromatic) between 8-7 instead of 4H (aromatic) in one proton (which was 4-phenylcyclohexanone). Hence, the other compound that was close to the melting point range of the ketone was 9-fluorenone (82-85oC), close enough to 78-80oC. 9-fluorenone also correlates with the NMR data perfectly. In the NMR for ketone it states that there is a scrambled peak with 8 chemically equivalent hydrogens, so presumably they are in a connected benzene ring structures. Which the compound has 2 rings, in which in between there is a cyclo-pentane like structure, thus making the rings chemically equivalent and adding up to a total of 8H. The NMR also suggests that there are only 8 hydrogens in the whole compound that correlates to the 9-fluorenone, as there are only 8 hydrogens in total. The NMR confirms the unknown alcohol to be 9-fluorenone

The NMR for the Alcohol does not support the assumed compound, which was based on the melting point range. It also disagrees with the alcohol presumed, 4-biphenylmethanol, because the NMR suggests that there are 8H in on proton (aromatic) while the alcohol presumed has 9H in one proton. Since, the old melt temp was used, presumed highly inaccurate, it was bound to give the wrong compound. Thus the other closest alcohol happened to be 9-fluorenemethanol, which happens to have a similar structure to our confirmed ketone. The NMR suggests that there are 8 chemically equivalent hydrogens combined into one or two aromatic benzene rings. Furthermore, it suggests that there are 2 protons with 1 hydrogen and 2 hydrogens very close to each other that they could be considered as one proton. And finally, it suggests that there is a one proton with one hydrogen that doesn’t see passed an oxygen molecule. This correlates perfectly with the new presumed alcohol group, 9-fluorenemethanol. This is because there re two aromatic benzene rings, which are separated by a cyclo-pentane like structure that is bonded to CH2OH group. This shows that there are two benzene rings, chemically equivalent, and the cyclo-pentane like structure attached to 1 hydrogen and the CH2OH group (which has 2H and 1H that doesn’t see pass the O). Hence, having 8H due to the chemically equivalent aromatic benzene rings, two protons with 1H and 2H right next to each other, and 1H in an OH group that doesn’t see pass the H. The NMR confirms the unknown alcohol to be 9-fluorenemethanol.

In conclusion, the ketone and alcohol were separated through the column chromatography and TLC analysis/ IR spectroscopy were used to confirm that they were efficiently separated. The melting point ranges were taken to get an assumption of what each of the compounds might be and the NMR was used to confirm the identities of the ketone and alcohol, which resulted into 9-fluorenone and 9-fluorenemethanol.