**Identification of unknowns: Isolation of an Alcohol and a Ketone**

**Introduction**

The main purpose for this particular lab is to identify two different unknown organic compounds, one being a ketone and the other being an aldehyde, by separating them through Column Chromatography and checking through TLC analysis.

**Physical Data**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Compound** | **Molar Mass (g/mol)** | **Melting Point (°C)** | **Boiling Point (°C)** | **Density (g/cm3)** | **Solubility**  **g/L** | **Hazards** |
| ***Hexanes*** | *86.18* | *-94.00* | *69.00* | *0.6548* | *0.0095* | *Flammable* |
| ***Ethyl acetate*** | *88.11* | *-83.60* | *77.10* | *0.8970* | *.8300* | *Flammable, Irritant* |
| ***Methylene Chloride*** | *84.93* | *-96.70* | *39.6* | *1.3300* | *13.00* | *Flammable, toxic* |

*\*Possible unknowns are given on pg.186 of the Experiments Book with MP and Structure.*

*Sources: Handbook for Organic Chemistry,* ***CRC Handbook of Chemistry and Physics*** *(especially Section C: "Physical Constants of Organic Compounds" ), available at the information desk in the Science Library (in Norlin) and in the Organic Chemistry Stockroom.*

**Procedure**

*Part 1:*

1. You would be given an unique unknown unkown mixture in a vial.
   1. It would contain an alcohol and ketone.
   2. Do not dispose of the vial and its contents for the remainder of the course.
2. Use TLC analysis to determine which solvent system should be used for the separation and to prepare for the slurry of the unknown mixture and silica gel.
3. Dissolve a tiny amount of your unknown in about 1mL of acetone (2/3 of a Pasteur pipet)
   1. Save it in a tight stoppered vial and label it “unknown standard”
      1. Use to determine the TLC solvent system and to run the TLC’s of the column fractions.
4. Spot 4 small TLC plates and run each plate in a different solvent system.
   1. Observe it under UV and mark it
   2. A single spot on the TLC plate means the mixture hasn’t been mixed thoroughly.
   3. Determine the Rf values
5. Looking at the Rf values, you can determine which one is your solvent system.
   1. This would also be used for the column chromatography
6. Check with the TA
7. For next part, prepare 50-80mg (make sure to be 100% in the range) and dissolve it with few drops of methylene chloride as possible.
8. Mix it with 75mg of silica and stir them together to create the slurry.
9. Store it in a vial, make sure its not covered and let it dry till the next lab period.

*Part 2:*

1. Prepare the column chromatography
   1. Add a small piece of cotton.
   2. Add silica gel until the near end of the pipet
      1. Make sure its 1.5 cm below the indentation in the pipet.
   3. Clamp it to the ring stand
   4. Add a collection flask under it
   5. Pre-elute the column with hexanes.
2. Load the column with the unknown compound
3. Fill a pipet with the solvent that was determined in part 1 of the lab and perform a column chromatography until 10 vials of 1mL of solvent each.
4. Perform the TLC analysis, spot from each of the 10 vials, which were collected earlier.
5. Remove the solvents from the pure compounds under the vacuum and run the IR spectrum of each of the compound.