**Electrophilic Aromatic Substitution**

**Introduction**

The main purpose for this lab is to learn how to perform an Electrophilic aromatic substitution reaction, thus creating monoacetylferrocene from ferrocene reactant using phosphoric acid and acetic anhydride.

**Reaction**

**Yield**

**Physical Data**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Compound | Safety | Molar mass (g/mol) | Melting Point (degrees C) | Boiling Point (degrees C) | Solubility (g/L) | Density (g/mL) |
| Ferrocene | Toxic, irritant | 186.04 | 172.5 | 249 |  | 1.107 at 0 C |
| Monoacetylferrocene | Toxic | 228.07 | 81-83 | 163 |  |  |
| Diacetylferrocene | Toxic | 270.1 | 127 | - |  |  |
| Phosphoric Acid | Toxic, Corrosive | 97.995 | 42.35 | 158 | 0.00548 | 1.685 |
| Acetic Anhydride | Toxic, flammable | 102.09 | -73.1 | 139.8 | 0.000026 | 1.082 |
| Hexanes | flammable | 86.18 | -94 | 69 | 0.0095 | 0.6548 |
| Methylene chloride | Toxic | 84.93 | -96.7 | 39.6 | 13 | 1.33 |
| Sodium Hydroxide | Toxic, Corrosive | 39.9 | 318 | 1388 | 0.00111 | 2.13 |

*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.*

***Safety Precautions***

*Phosphoric acid, acetic anhydride, and sodium hydroxide are corrosive and will cause burns if have contact with skin. Hexanes are flammable. Ferrocene and methylene chloride are moderate health hazards and contact should be minimized.*

***Wastes***

*Aqueous Waste: Aqueous filtrates from vacuum filtration of crude product*

*Organic Waste: Methylene chloriode used to elute the TLC plates and all of the solvents used to elute the column.*

*Solid Chemical Waste: Used columns, melting point capillaries, coverslips and TLC plates*

**Procedure**

1. Place 1mmol of ferrocene in a 25mL round bottom flask.
2. Also add 2mL of acetic anhydride into round bottom flask.
   1. Swirl the flask to mix the contents
3. Set up the reaction with the versatile clamp
4. Place the heating mantle
5. Add 0.4mL of phosphoric acid to the round bottom flask
6. Time it as soon as you start the Variac (set at 40)
7. After 15 minutes, pour the mixture into a beaker containing 10mL of ice
   1. Will dilute the reaction mixture
8. Rinse the reaction flask with distilled water.
9. Place the beaker in an ice bath and add 6M NaOH dropwise to neutralize the reaction
10. Isolate the product by vacuum filtration.
11. Rinse the solid with cold water.
12. Take a small amount of product to dissolve into the methylene chloride for TLC analysis.
13. Allow the product to dry
14. Apply the Melting Point Test.
15. Weigh 50mg of the product, and mix the crude product with 150mg of silica gel and 1 ml of methylene chloride.
16. After the solvent has evaporated.
17. Conserve the mixture for next week’s lab.