Week 7-9
Carboxylic Acids

Preparation of Methyl 3-Nitrobenzoate

Methyl benzoate

Methyl 3-nitrobenzoate

Preparation of 3-Nitrobenzoic Acid

Methyl 3-nitrobenzoate

3-Nitrobenzoic acid

Preparation of 3-Nitrobenzamide

3-Nitrobenzoic acid

3-Nitrobenzamide
Preparation of Methyl 3-Nitrobenzoate

- We are using concentrated acids. Be careful!!!

- All glassware must be dry! Excess of water will stop the reaction.

Figure 8.1. The synthesis of methyl 3-nitrobenzoate.
Preparation of Methyl 3-Nitrobenzoate
Mechanism

\[
\begin{align*}
\text{H}_2\text{O} \cdots \text{NO}_2 + \text{H} \cdots \text{OSO}_3\text{H} & \leftrightarrow \text{H}_2\text{O} \cdots \text{NO}_2 + \text{HSO}_4^- \\
\text{H} & \quad \text{O} \quad \text{NO}_2 \leftrightarrow \text{H}_2\text{O} + \text{O} = \text{N} = \text{O}
\end{align*}
\]

\[
\begin{align*}
\text{CO}_2\text{CH}_3 & \quad \leftrightarrow \quad \text{CO}_2\text{CH}_3 \\
\text{O}_2\text{N} & \quad \leftrightarrow \quad \text{O}_2\text{N} \\
\text{I} & \quad \leftrightarrow \quad \text{II} \quad \leftrightarrow \quad \text{III} + \text{H} \cdots \text{O} \cdots \text{H}
\end{align*}
\]

*Figure 8.2. Mechanism of the nitration of methyl benzoate.*
Dropwise addition of the HNO$_3$ solution.
- When the addition of the HNO$_3$ solution is over remove from ice bath and allow to warm to RT.
Isolation of Methyl 3-Nitrobenzoate

- Add reaction mixture into 25 g of ice

50 mL ice = 25 g ice
Trituration of Methyl 3-Nitrobenzoate

1. Place in beaker
2. Redissolve benzoate with water into beaker
3. Suction
4. Filtration
5. Dry filter cake with filter paper
Recrystallization of Methyl 3-Nitrobenzoate

- Steam In
- Hot Water Out

Recrystallize product with minimum amount of methanol

- Cool down slowly to get crystals of your product

- Pre-weigh flask or beaker
- Transfer your product, weigh the flask again
- Based on your isolated product calculate % yield

- Air condenser
- Suction
- Filtration

Steam In
Hot Water Out
Calculation of % Yield

Calculate the percent yield for the formation of ammonia in the following reaction.

\[
\begin{align*}
N_2 + 3H_2 & \rightarrow 2NH_3 \\
2.2 \text{ g} & \quad 1 \text{ g} & \quad 1.2 \text{ g}
\end{align*}
\]

\[
\text{Percent Yield} = \frac{\text{Actual Yield}}{\text{Theoretical Yield}} \times 100
\]

\[
\begin{align*}
2.2 \text{ g N}_2 & \times \frac{1 \text{ mol N}_2}{28 \text{ g N}_2} \times \frac{2 \text{ mol NH}_3}{1 \text{ mol N}_2} \times \frac{17 \text{ g NH}_3}{1 \text{ mol NH}_3} = 2.7 \text{ g}
\end{align*}
\]

\[
\begin{align*}
1 \text{ g H}_2 & \times \frac{1 \text{ mol H}_2}{2 \text{ g H}_2} \times \frac{2 \text{ mol NH}_3}{3 \text{ mol H}_2} \times \frac{17 \text{ g NH}_3}{1 \text{ mol NH}_3} = 5.7 \text{ g}
\end{align*}
\]

Limiting reagent is the one you run out. In this case \( N_2 \)

\[
\text{Percent Yield} = \frac{1.2}{2.7} \times 100 = 44 \%
\]