SAMPLE LAB NOTEBOOK ENTRY FOR CHEM 267/268

Use this as a very general guide to follow in keeping your notebook. A copy of the notebook entries along with a typewritten report, will be submitted for each experiment.

**Experiment:** Diels-Alder Synthesis

**Date:** 1/19/03

**Student:** Pati Samal

**Diels-Alder Synthesis**

\[
\text{cis-Norbornene-5,6-endodicarboxylic anhydride}
\]

\[
\begin{align*}
\text{cyclopentadiene} & \quad \text{maleic anhydride} \\
\quad \quad & \quad \text{cis-Norbornene-5,6-endodicarboxylic anhydride} \\
\text{Diels-Alder product} & \quad \text{(Diels-Alder Adduct)}
\end{align*}
\]

**Reagents**

<table>
<thead>
<tr>
<th></th>
<th>MW</th>
<th>Dens.</th>
<th>B.P.</th>
<th>M.P.</th>
</tr>
</thead>
<tbody>
<tr>
<td>cyclopentadiene</td>
<td>66.1</td>
<td>0.80</td>
<td>81</td>
<td>-</td>
</tr>
<tr>
<td>maleic anhydride</td>
<td>98.1</td>
<td>-</td>
<td>53</td>
<td>-</td>
</tr>
<tr>
<td>Diels-Alder product</td>
<td>164.2</td>
<td>-</td>
<td>165</td>
<td>-</td>
</tr>
</tbody>
</table>

**Quantities**

<table>
<thead>
<tr>
<th></th>
<th>mL</th>
<th>g</th>
<th>moles</th>
</tr>
</thead>
<tbody>
<tr>
<td>cyclopentadiene</td>
<td>0.20</td>
<td>0.16</td>
<td>0.0024</td>
</tr>
<tr>
<td>maleic anhydride</td>
<td>0.20</td>
<td>0.0020</td>
<td></td>
</tr>
</tbody>
</table>

Maleic anhydride is limiting reagent. Cyclopentadiene is in excess.

**Theoretical amount of Diels-Alder product**

\[= 0.0020 \text{ mol} \times 164.29/\text{mol} = 0.3289\]

**Note:** Insert periodic table under copy sheet before writing. *The Hayden-Mauler Student Lab Notebook*
PRELAB OUTLINE

REF: WILLIAMSON TEXT PPs. 316, 317 AND CNE 269 HANDOUT.

- MEASURE 0.20g MALEC ANHYDRODE INTO REALT. TUBE
- DISSOLVE IN 1mL ETHYL ACETATE THEN ADD 1mL LIGROIN (60-80° B.P.)
- ADD 0.20 mL DRY CYCLOPENTADIENE, MIX WELL.
- ALLOW TUBE TO COOL TO RT. TO CRYSTALLIZE
- (IF CRYSTALS DO NOT FORM, SCRATCH INSIDE OF TUBE JUST BELOW SURFACE WITH GLASS STIRRING ROD, IF CRYSTALS ARE TOO FINE, REHEAT TO DISSOLVE & ALLOW TO COOL SLOWLY, ADD 1G. CRYSTAL IF NECESSARY.)

- REMOVE SOLVENT BY AIR DRY METHOD, RINSE CRYSTALS WITH COLD LIGROIN, REMOVE SOLVENT.
- SCRAPE CRYSTALS ONTO FILTER PAPER TO DRY
- WEIGHT, TAKE M.P.
(ALL MATERIAL TO HERE IS DONE BEFORE LAB)

PROCEDURE, OBSERVATIONS, DATA

- Maleic Anhydride

  Gross: 0.0379 g
  Tare: 0.0129 g
  Net: 0.0250 g

Add 0.020 g Maleic Anhydride to react tube.

Add 1 mL Ethyl Acetate to dissolve then add
1 mL Cigroin (80:60), Sol'n is slightly
yellow but clear (no precipitates). Use
syringe to transfer 0.20 mL Cyclopentadiene (Dry)
to reaction tube and mix well with stirring
rod. Reaction tube gets warm, yellow
color disappears. Place warm tube into
beaker containing paper towel as insulation
so tube cools slowly.

AFTER 10 MIN, LARGE COLORLESS
PLATE-LIKE CRYSTALS APPEARED.
TUBE WAS ALLOWED TO
COOL TO ROOM TEMP FOR COMPLETE
CRYSTALLIZATION, SOLVENT WAS REMOVED BY THE PAPER METHOD AND THE CRYSTALS WERE RINSED WITH 1.5 ML COLD LIGROIN. THE LIGROIN WAS REMOVED, THE CRYSTALS WERE SCREWED OUT OF THE TUBE AND ALLOWED TO DRY IN THE AIR, TO CONSTANT WEIGHT.

1st weight crystals + filter paper

\[ \frac{0.5857}{0.7529} = 0.775 \]

After 10 min

\[ \begin{align*}
0.5659 & \quad 0.7529 \\
0.3139 & \quad 0.3139 \\
\end{align*} \]

After 5 min

\[ \begin{align*}
0.5659 & \quad 0.7521 \\
0.3139 & \quad 0.3139 \\
\end{align*} \]

\[ \text{Yield} = \frac{0.3139}{0.3289} \times 100 = 95\% \]

m.p. 163.5 - 164.5 °C.
RESULTS AND DISCUSSION

The Diels-Alder reaction of cyclopentadiene and maleic anhydride produces a product in excellent yield and purity, as judged by the m.p. Additional analysis to confirm the identity and purity could have been done by TLC, IR, and NMR, (etc., etc., etc., etc.)

ANSWERS TO ASSIGNED QUESTIONS

(1.)

\[
\text{C}_6\text{H}_{14} + \text{C}_2\text{H}_2 \rightarrow \text{C}_8\text{H}_{16}
\]

(etc., etc., etc.)