VIII. Preparation of Cyclopentadiene

A. Reaction

![Chemical Structure](image)

B. Procedure

The apparatus for the cracking of dicyclopentadiene to cyclopentadiene will be set up in the hood and will be used to prepare all of the cyclopentadiene needed for the Diels-Alder reaction and for the preparation of ferrocene. The equipment consists of a 100 ml round bottom flask fitted with a Claisen head, a condenser take-off, a thermometer, a condenser, a receiver adapter, and a receiver cooled in an ice bath. The round bottom flask is filled about one third full of paraffin oil and a thermometer is run down through the Claisen Head into the oil. The oil is then heated with a mantle to about 250°C. The thermometer in the oil is removed and a syringe containing dicyclopentadiene is positioned by means of a septum directly above the oil. The dicyclopentadiene is added cautiously to the hot oil. The cyclopentadiene distills at 42°C, but the temperature may go a few degrees higher due to super-heating. The dicyclopentadiene is added at a rate sufficient to maintain a continuous collection of material in the receiver. One should be able to collect 10-20 mL per hour without difficulty.
A. Reaction

\[ \text{Cyclopentadiene} + \text{Maleic Anhydride} \rightarrow \text{Product} \]

B. Procedure

In a 2 mL Craig tube place 0.7 mL of ethyl acetate and 150 mg (1.53 mmoles) of freshly ground maleic anhydride. Stir to dissolve. Then add 0.7 mL of hexane followed by 150 μL (120 mg, 1.82 mmoles) of cyclopentadiene. Observe the heat of the reaction. After a few minutes scratch the walls of the tube in order to induce crystallization. Cool the tube in an ice bath for a few minutes and collect the crystals by centrifugation. Remove the crystals, allow them to dry, and determine the yield, mp, and IR spectrum.
C. Report: Diels Alder Reaction

Name __________________ Date __________________ Exp. No. ______

Chemical Reaction:

Reagents:

<table>
<thead>
<tr>
<th>Compound</th>
<th>Mol Wt.</th>
<th>Density</th>
<th>Wt., mg</th>
<th>Vol. µL</th>
<th>mmole</th>
</tr>
</thead>
</table>

Products:

<table>
<thead>
<tr>
<th>Compound</th>
<th>Mol Wt.</th>
<th>Density</th>
<th>Wt., mg</th>
<th>Vol. µL</th>
<th>mmol Theoretical</th>
</tr>
</thead>
</table>

Attach IR spectrum, Gas Chromatograms, etc. I.R. bands and assignments

Yield ______ mg ______ mmol

Yield ______ %

mp or bp range ______, Lit. ______

Evidence for the confirmation of structure.