Experiment 23: Synthesis of Tetraphenyl-1,2-Dihydrophtalic Anhydride

In this experiment tetraphenyl-1,2-dihydrophtalic anhydride will be prepared via a Diels-Alder reaction followed by loss of carbon monoxide. The reaction and mechanism are outlined below.
The intermediate product, I, can exist in two possible stereochemistries, exo or endo shown below.

![exo and endo structures]

The endo configuration is favored in this reaction.

Procedure

1. Mix 7.0 gram of tetrphenylocyclopentadienone (prepared by student previously via aldol condensation reactions) and 1.9 g of maleic anhydride thoroughly in a 100 mL round bottom flask. Add 5 mL of bromobenzene to the round bottom. This will result in a thick mixture of solids. A reflux condenser is then attached to the flask.

2. Since CO will be produced in the reaction, it is necessary to set up the reaction in a fume hood or attach a CO trap. Please see your TA or instructor if for some reason you are not working in a fume hood.

3. When the apparatus is assembled, the mixture is refluxed for a total of three hours until the dark brown color of the solution disappears and a light, yellow solid mass appears. Agitate the mixture occasionally during the reflux period.

4. When the reaction appears to be complete, cool the flask to room temperature and add 50 mL of hexanes. Using a spatula, break up the solid in the ligroin and vacuum filter the
mixture. Wash the solid with two 10 mL portions of ligroin and allow it to dry. Take the melting point and then recrystallize the solid from an appropriate solvent system, followed by vacuum filtration and drying. Weigh the purified product and determine its melting point. Measure and IR spectrum for the product.