**Chem 2229: Exp. #3 The Diels-Alder Reaction of Anthracene with Maleic Anhydride**

**In this experiment you will learn how to use the Diels-Alder reaction to form a bridged polycyclic anhydride. Two solids – anthracene and maleic anhydride – will be dissolved in xylene and refluxed. The product 9,10-dihydroanthracene-9,10-α,β-succinic anhydride will be recovered and isolated by vacuum filtration. The product will be characterized by its melting point. Theoretical and Percent Yield will be determined for the product.**

* The Diels-Alder reaction is an example of a pericyclic reaction. Pericyclic reactions are important because they are concerted reactions that take place in one step through a cyclic transition state in which symmetry characteristics of molecular orbitals control the course of the reaction. (Solomon's 599) The melting point (MP) is a physical property of a solid also used for the purpose of identification and purity determination.

**Reading Assignment:**

OCLT: p. 365 (reflux); p. 281 (Diels-Alder reaction); p. 366 (vacuum filtration); and pp. 309-315 (melting point).

Solomon's (12th ed): 13.10 The Diels Alder Reaction (pp. 599-613).

**Procedure**

1. Preheat the hot plate and aluminum block at a heat setting of ~180-200 °C while you assemble your glassware.

2. Put together a reflux set-up* as shown to the right.

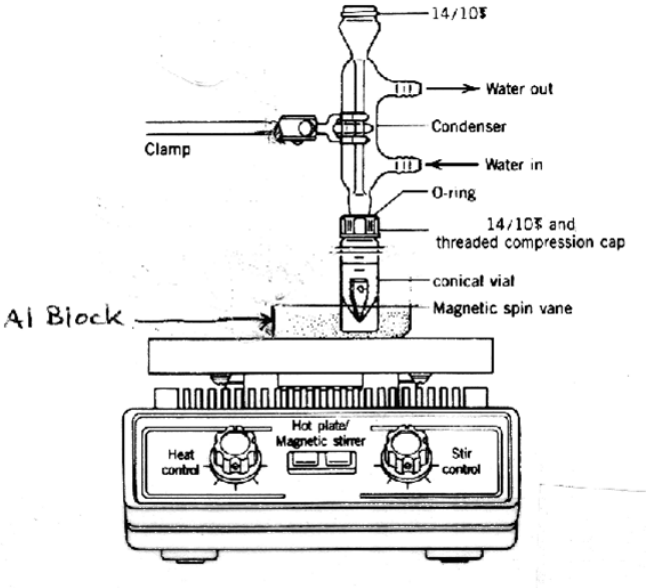


**Figure 1: Reflux Apparatus Set-up**

***Note:** Verify that there is an o-ring (and only one o-ring) in the black cap before attaching the conical vial to the jacketed water condenser. Do not lower empty glassware into the Al block; it will cause the glassware to crack. A thermometer is not necessary to monitor the reflux.

3. In a 5-ml conical vial, weigh out ~100 mg of anthracene and ~55 mg of maleic anhydride. Record the exact mass of each compound to the nearest mg (0.001g).* Record the physical properties of the compounds.

***Note:** It is important that the exact masses are recorded in order to determine which compound is the limiting reagent. The limiting reagent will be used to calculate the theoretical yield of the product.

4. To the 5 ml conical vial, add ~1 ml of xylene (*located in the hood*).

5. Add the large magnetic spin vane. Attach the jacketed water condenser. Lower the vial into the Al block. Clamp the jacketed water condenser to the ringstand. Start the stirring.

6. Attach the thin walled water hoses* to the condenser.

***Note:** Connect the hoses so that water travels against gravity (*cooling water comes into the bottom and drains out the top*). Turn the water on low. Water should go in the bottom of the condenser jacket slowly. (*If there is a lot of bubbling in the condenser, turn the water down.*) **Do not let the rubber tubing touch the top of the hotplate. It will melt.**

7. Heat the reaction mixture to boiling while stirring. Reflux for 30 min. (*Start timing only after you notice the condensation running down into the vial.*)

8. After the 30 minutes, remove the 5 ml conical vial from the aluminum block and the heat source. Turn the heat off on the hotplate. Leave the water condenser attached to the vial and the water running. Lower the clamp so that the vial is resting on the lab counter. Allow the vial to cool for 5 minutes.

9. Prepare an ice bath using a copper pot (*found in the common drawer*). The ice is located in a Styrofoam box near the door. Add ice to the copper pot and a small amount of water. If too much water is added the vial will tip over.

10. In a 3ml conical vial, acquire 2 ml of xylene. Add a cap with a septum to the vial and place it in the ice bath.

11. Remove the 5 ml conical flask from the reflux apparatus. Add a cap with a septum to the vial. Place the vial in the ice bath. Pack ice around the vial. Allow the solution in the vial to cool in the ice bath for 5+ minutes to minimize solubility of the product.

12. Disassemble the remainder of the reflux apparatus and put the equipment away. Put the hotplate away.

13. Assemble a vacuum filtration apparatus using the 25 ml filter flask, the 1.0 cm Hirsch funnel and the 1.0 cm filter paper. (*These items should be in your drawer. The filter paper will be in a disposable vial in your glassware kit and looks like large hole-punch holes*.) Test the vacuum. Attach the hose to the flask. Pour the contents of the vial over the filter paper quickly in order to seal the paper.

14. Vacuum filter the solution to isolate the white crystalline product. Rinse the crystals with 1-2 ml of cold xylene. Allow the crystals to dry over the vacuum for 10-15 minutes.

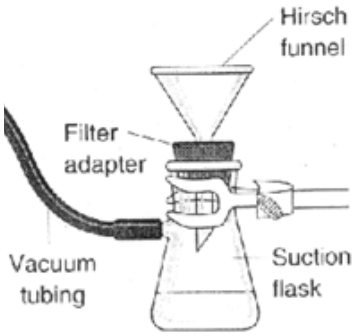


**Figure 2: Vacuum filtration system.**

15. Scrape the product off the filter paper onto a piece of tared weighing paper. Weigh the product. Record the exact mass to the nearest mg (0.001g).

16. Determine the melting point of the product. (If the determined MP range is >10 ºC, consult your instructor and redo the determination.) Record the name and number of the MP apparatus used. Consult handout for MP determination instructions.

**Clean Up:**

17. Dispose of the chemicals in the general waste container. Clean and return all glassware and equipment.

**Table 1: Molar Masses and Melting Points of Reactants and Products**

| Substance | Molar Mass (g/mole) | MP (°C) | BP (°C) |
|---|---|---|---|
| anthracene | 178.23 | 216-218 | |
| maleic anhydride | 98.06 | 54-56 | |
| xylene | 106.17 | | 137-144 |
| 9,10-dihydroanthracene -9,10-α,β-succinic anhydride | 276.29 | 262-264 | |

**Post Lab Calculations: (*Show all calculations in your lab notebook)***

1. Determine which of the reactants is the limiting reactant based on your recorded starting amounts of the reactants.
2. Determine the theoretical yield of the product based on the limiting reactant.
3. Determine the % yield of the product.
4. Determine % Error for the melting point of the product.
5. Tabulate your results in your lab book.

**References:**

Bone, Terry. "Diels-Alder Reaction" PowerPoint. Missouri S & T. November 2018.

Wade, L. G., Jr. "SYNT0717: The Diels Alder Reaction of Anthracene with Maleic Anhydride." Chemical Education Resources, Inc. Pennsylvania: 1998.