

## **Exp.10: preparation of Anthracene-9,10-endo- $\alpha,\beta$ -Succinic Anhydride by Diels-Alder Reaction**

### **Objectives:**

- The purpose of this experiment is to form 9, 10-dihydroanthracene-9,10- $\alpha, \beta$ -succinic anhydride by way of a Diels Alder reaction.
- Purifying the Product To perform recrystallization.
- Characterize the product using melting points and the infrared spectrum.

### **Introduction:**

The Diels Alder reaction between anthracene and maleic anhydride to form 9, 10-dihydroanthracene-9,10- $\alpha, \beta$ -succinic anhydride was occurred via the Diels Alder mechanism. Anthracene served as the diene and maleic anhydride was the dienophile. Following the reaction

The crude product will be recrystallized. Melting points of both the crude product and recrystallized product will be taken and compared with the literature value. Also, an infrared spectrum of the recrystallized product will be taken. Both melting points and the infrared spectrum will be used to characterize the product.

### **Procedure:**

#### **Part 1- preparation:**

- Weigh 2.0 gm of anthracene and 1.1 gm maleic anhydride and place them in the reaction flask. Add 25.0 mL of xylene.
- Fit the vial with a condenser,
- Turn on the water to the condenser Use a 200°C sand bath to heat the mixture at reflux for 30 minutes.
- Cool the mixture to room temperature and prepare an ice bath using a 150-mL beaker. Then cool the mixture in the ice bath for approximately 5 minutes.

- Collect the recrystallized solid by vacuum filtration using a Hirsch funnel and rinse the crystals with 1 mL of ice-cold xylene.
- Weigh the crude product and set aside a small sample to dry.
- This sample will later be used to measure the melting point of the crude product.

### **Part 2- purification:**

Purifying the Product To perform recrystallization.

- place the product in a 10-mL Erlenmeyer flask.
- Add ethyl acetate and heat the mixture gently in a water bath until the ethyl acetate boils.
- Gradually add more ethyl acetate until all the product dissolves or until no more appears to be dissolving.
- Transfer the solution (filtration) to another 10-mL flask containing a small amount of boiling ethyl acetate.
- Allow the solution to cool to room temperature and then cool the solution in an ice bath for 5 minutes. If it's necessary, scratch the bottom of the flask with a glass rod to induce crystallization.
- Collect the crystallized solid by vacuum filtration, using a Hirsch funnel.
- Spread the product crystals thinly over a clean watch glass and allow them to dry for approximately fifteen minutes.
- Then weigh the product and record the mass. Place the product in a labeled vial.

### **Part 3- Characterization:**

Characterizing the Product Measure the melting points for both the crude product and the recrystallized product. Perform an infrared spectrum on both products. Add 100 mg KBR to approximately 1 mg of the product. Mix the two using a mortar and pestle, being sure to grind the two compounds into a fine powder. Place some of this mixture in a microcup, making sure the surface is smooth. Then place the pellet in the infrared spectrometer and obtain an infrared spectrum of the product. Compare the product spectrum with the spectra of maleic anhydride and anthracene in order to characterize the product.

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### Laboratory Report

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Name: -----

Date: -----

Experiment Subject: -----

**- Reaction:**

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**Calculations:**

Compound	Mol. Formula	Mol. Wight	Moles	Wight, mg	Density	Volume	Limiting reagent

**Purification:**

Recrystallization solvent: -----

Purity check by melting range: -----

TLC: -----

**Physical Data (Product):**

State: -----

Melting Point: -----

Color: -----

Solubility: -----

**Yield:**

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**Characterization:**

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