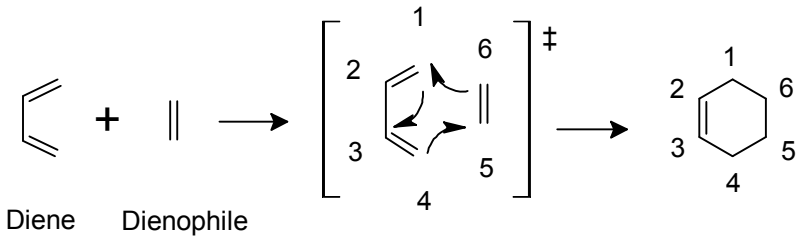
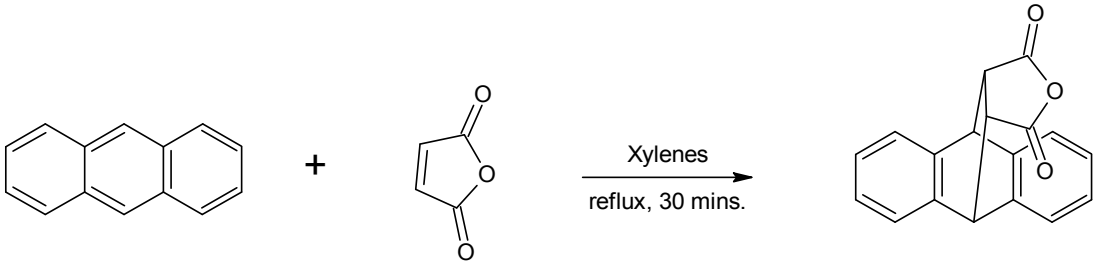
The Diels-Alder Reaction of Anthracene with Maleic Anhydride

The *Diels-Alder* reaction is a member of a class of reactions called cycloadditions. The reaction involves three π bonds, two from the diene and one from the dienophile in a concerted reaction to form a six-membered ring. Since the reaction involves four π electrons in the diene and two π electrons from the dienophile, it is sometimes referred to as a 4 + 2 cycloaddition.

Normal Diels-Alder reactions are favored by electron donating groups on the diene and electron withdrawing groups on the dienophile. The diene must be capable of achieving an *s-cis* conformation to generate the *cis* double bond in the cyclohexene product. Acyclic dienes may rotate around a single bond, but dienes locked in the *s-trans* conformation do not react.



The purpose of this experiment is to form 9,10-dihydroanthracene-9,10-α,β-succinic anhydride by way of a Diels Alder reaction between anthracene and maleic anhydride, as shown in the reaction below. Anthracene acts as the diene and maleic anhydride functions as the dienophile. Xylene (dimethylbenzene) is used as a high boiling temperature solvent so that the reaction will proceed quickly .



Anthracene *Maleic anhydride* *9,10-dihydroanthracene--9,10-α,β-succinic anhydride*

OBJECTIVES

In this experiment, you will

- Synthesize 9,10-dihydroanthracene-9,10-α,β-succinic anhydride.
- Isolate the product.

- Measure the melting temperature of your product ONLY if the Lab Instructor confirms that the thermometers in the lab can handle the relatively high melting point (>250 deg C)

MATERIALS

Part I Synthesis

50 mL round bottom flask
stir bar
reflux condenser
heating mantle and power controller
sand
drying tube
ring stand with two utility clamps
ring stand with filtering flask
Büchner funnel
filter paper
10 mL graduated cylinder
disposable Pasteur pipets and bulb
100 mL beaker
two 250 mL beakers
watch glass
anthracene
maleic anhydride
xylene
Drierite®
distilled water
ice
magnetic stir plate
balance
weighing paper
grease
spatula
cotton plug
compressed air
Temperature Probe or thermometer
boiling stone (optional)

PROCEDURE

Part I Synthesis

1. Wear goggles. If possible, protect your arms and hands by wearing a long-sleeve lab coat and gloves. Conduct this reaction in a fume hood.

2. Weigh out 0.80 g of anthracene and 0.40 g of maleic anhydride and transfer the reagents into a 50 mL round bottom flask containing a stir bar. Record both masses to the nearest 0.01 g.

3. Add 10 mL of xylene to the round bottom flask. **CAUTION:** *Xylene is flammable. Keep away from open flames and hot plates*.

4. Set up a reflux with the condenser and a drying tube, making sure to clamp the flask and condenser securely. Remember to grease the joints to prevent the glass from sticking. **Note:** Maleic anhydride is water sensitive and the drying tube will help prevent water in the air from entering the flask.

5. Heat the reaction mixture using a heating mantle to reflux (~180°C) for approximately 30 minutes. Monitor the temperature using a Temperature Probe or thermometer.

6. While waiting, prepare two ice water baths using two 250 mL beakers.

7. Obtain approximately 5 mL of xylenes in the 100 mL beaker. Place the beaker in the ice water bath to cool.

8. After 30 minutes, let the reaction flask warm to room temperature. Wait at least 15 minutes for the flask to warm to room temperature. Then, place the flask in the second ice water bath for 10 minutes. You should observe crystallization at this point.

9. Collect the solid.
   a. Weigh the filter paper and record the mass to the nearest 0.01 g.
   b. Set up a vacuum filtration with the Büchner funnel.
   c. Filter the solid and wash the solid with ~5 mL of cold xylene.
   d. Place the filter paper containing the solid on a watch glass and gently direct a stream of air (low flow) to thoroughly dry the solid.

10. Weigh the filter paper and dried product. Record the mass to the nearest 0.01 g. The sample should be completely dried before taking a melting temperature.

Record the weight of the product and calculate the percent yield for the reaction.

If your instructor confirms that the thermometer in the melting point apparatus can handle m.pts. above 250 deg C, measure the m.pt.

If time permits do a TLC of the product along with the starting materials. Instructor will elaborate.

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DATA TABLE

Part I Synthesis of 9,10-dihydroanthracene-9,10-α,β-succinic anhydride

Mass of anthracene (g)	
Mass of maleic anhydride (g)	
Mass of filter paper (g)	
Mass of filter paper and product (g)	
Mass of product (g)	

Part II Melting Temperature

	9,10-dihydroanthracene-9,10-α,β-succinic anhydride
Measured melting temperature range (°C)	

DATA ANALYSIS

1. What is the theoretical yield of 9,10-dihydroanthracene-9,10-α,β-succinic anhydride in your synthesis? What is the actual yield?

OTHER INFORMATION/ HAZARD ALERTS

1. Xylenes are flammable. Keep away from open flames and hot plates.
2. Assemble the drying tube by placing a small piece of glass wool or cotton near the tip, fill with Drierite®, and cover with a second piece of glass wool or cotton.
3. The product will start to precipitate as the reaction flask cools to room temperature.
4. Dry the product completely before taking a melting temperature.
5. Dispose of waste appropriately.

Anthracene: May cause respiratory tract irritation. Skin and eye irritant. Moderately toxic by ingestion. HMIS Classification: Health hazard–0, Flammability–1, Physical hazard–0.

Maleic anhydride: Toxic by inhalation and ingestion. Severe eye and skin irritant. HMIS Classification: Health hazard–3, Flammability–0, Physical hazard–0.

Xylenes: Moderate fire hazard (flash point 25°C). Moderately toxic by inhalation or ingestion. Causes skin and eye irritation. HMIS Classification: Health hazard–2, Flammability–3, Physical hazard–0.

Drierite®: Toxic by ingestion. May cause respiratory tract irritation. Skin and eye irritant. HMIS Classification: Health hazard–2, Flammability–0, Physical hazard–0.

The hazard information reference is Sigma-Aldrich Co.,
1-800-325-3010, www.sigmaaldrich.com/safety-center/msds-search.html.

COMPOUND INFORMATION

Compound	Chemical formula	Melting temperature range (°C)	Molar mass (g/mol)
anthracene	$C_{14}H_{10}$	210–215	178.23
maleic anhydride	$C_4H_2O_3$	51–56	98.06

Compound	Chemical formula	Boiling temperature range (°C)	Molar mass (g/mol)	Density (g/mL) at 25°C
xylenes	C_8H_{10}	137–140	106.16	0.86