

## Lab 04: Diels-Alder Reaction (Pavia 3<sup>rd</sup> Edition)

### Reading

- Pavia, et al., Expt. 48, pp. 406–409.
- Pavia: Essay pp 402-406.
- Pavia, et al., Technique 4.7, pp. 556–557.
- Pavia, et al., Appendix 3, pp. A14–A32 (review as necessary).
- Wade, 6th ed., Section 15-11, pp. 680–689

### Prelab Assignment:

Your prelab should include: Date, Introduction, Haz-Mat, Chemical Equation, Reactant Table, and Procedures.

### Prelab Notes

In the Chemical Equation section, just write the equation for the Diels-Alder reaction. The reaction for the cracking of dicyclopentadiene is not necessary. Use cyclopentadiene in the Reaction Table calculations. The density of cyclopentadiene is 0.8021 g/mL.

*Please note the following:*

### Preparation of the Adduct:

**1, Do not shake the Craig tube to dissolve the maleic anhydride in ethyl acetate.** Instead of shaking, mix the solution by inserting the end of a microspatula into the tube and twirling the spatula using your fingers. **Make sure the maleic anhydride is almost completely dissolved in ethyl acetate before adding the hexanes (ligroin).** *Ligroin is another name for a mixture of hexanes. We may be using hexanes instead of ligroin, depending on what is available.*

- Most of the solid should be dissolved before adding the cyclopentadiene. The tube can be heated in water bath made by putting about 10 mL of water in a 25 mL Erlenmeyer flask and heating to 50-60 °C. This water bath can be used during the cooling and recrystallization.
2. We will use a syringe and needle to measure the cyclopentadiene. After dispensing, rinse the syringe with a small amount of acetone in the hood. **Cyclopentadiene has a strong odor. Please try to keep it in the hood as much as possible.**
3. If no solid forms, add more cyclopentadiene and heat in the 50-60 °C water bath. This will often help the reaction to proceed.

### Crystallization

**4. Important safety note: Make sure that the centrifuge is balanced by placing tubes on opposite sides of the rotor.** If no other tubes are available, balance using a centrifuge tube that contains water.

- *The Craig tube may need to be centrifuged several times to get all the liquid out.*
- *Remember to lift the Craig tube out of the centrifuge tube with the wire attached to the plug, so that the crystals don't spill out.*

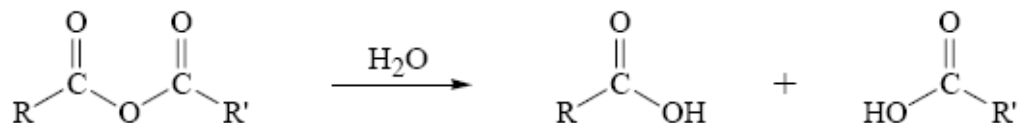
### Analysis of Product

5. Leave the product crystals loosely covered overnight (until the next lab period) before weighing, determining the melting point, and taking a KBr IR. A good way to do this is to spread them on watch glass and then cover with an upside-down beaker.

**Postlab Questions**

Answer the following questions in you lab notebook after your conclusions in a section labeled "Post-Lab Questions". These are good to know for quiz material too!

1. a. Draw and label the structure(s) of the product of this reaction. (b) If more than one product is formed, label the products as major and minor. (c) Label the product(s) as kinetic or thermodynamic.
2. Maleic anhydride is an *acid anhydride* (a functional group that we will study later this semester). Acid anhydrides react with water by adding a molecule of water and splitting into two carboxylic acids:



- a. Draw the reaction of maleic anhydride with water. (not the mechanism)
  - b. Why is it important that the equipment used in the experiment be dry?
3. The infrared spectrum (IR spectrum) of the desired Diels-Alder product is given in the text. Sometimes the IR spectrum of some students' products has an additional large, broad absorption peak from 3300–2900  $\text{cm}^{-1}$ .
    - a. What functional group does this peak represent?
    - b. Why might this peak be present? What does it indicate about the product when it is present?