**The Diels-Alder Reaction: A reaction of a conjugated diene**

**Purpose**
To perform a reaction of a diene with a dienophile, to perform a microscale recrystallization of the product, and to gain experience using the IR spectrometer.

**Background**
Conjugated dienes can react with alkenes via a 1,4-cycloaddition reaction, a Diels-Alder reaction. Interestingly, when the reaction creates a fused ring system, such as the products pictured in eq. 1., often, one of the two possible products is preferred. The two possible products are an *exo*, large parts for the fused rings point away from each other, and an *endo*, large substituents of the fused ring system point toward each other, product.

![Diels-Alder Reaction Diagram](image)

When the dienophile contains a π bond adjacent to the π bond that is reacting with the diene a process referred to as secondary orbital overlap explains the selectivity of the reaction. When the reactants are arranged in an *endo* configuration, the adjacent π bond creates an opportunity for additional frontier orbital interaction. This additional interaction stabilizes the transition state. On the other hand, when the reactants are arranged in an *exo* configuration, no such interaction is possible (fig 1.).

![HOMO-LUMO Overlap Diagram](image)

**Figure 1.** (Left) The highest occupied molecular orbital (HOMO) overlapping with the lowest unoccupied molecular orbital (LUMO)

For comparison, when there isn’t a π bond on the dienophile adjacent to the π bond that is reacting with the diene there is no enhanced frontier orbital interaction.
**Procedure**

**Reaction of cyclopentadiene with maleic anhydride**

1. Add 0.100 g of maleic anhydride and 0.40 mL of ethyl acetate to a Craig tube.
2. Gently shake (or use a vortex stirrer) and warm the tube (approximately 40 °C) to dissolve the maleic anhydride, a small amount of ethyl acetate can be added.
3. Add 0.40 mL of ligroin and shake.
4. Add 0.10 mL of cyclopentadiene and shake the tube to dissolve the cyclopentadiene.
5. If a solid forms immediately, add a drop of ethyl acetate and gently warm the suspension. When the exothermic reaction begins, you should be able to feel the heat escaping from the reaction.
6. Allow the reaction to cool slowly to room temperature. Once crystal formation at room temperature has stopped, cool the Craig tube in an ice water bath.
7. Collect the crystals by centrifuging the Craig tube.
8. Dry the product on a watch glass.

**Analytical Data**

8. Collect IR data for maleic anhydride and for your product, cis-5-norbornene-endo-2,3-dicarboxylic anhydride.
9. Determine the melting point of your product.

**Report**

Draw the reaction of cyclopentadiene with maleic anhydride. Determine the limiting reagent for the reaction (show all data and calculations). List the mass of the product, determine the percent yield (show all data and calculations), and list the melting point of your product. Include copies of your IR data to support the identification of the molecule as cis-5-norbornene-endo-2,3-dicarboxylic anhydride.

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