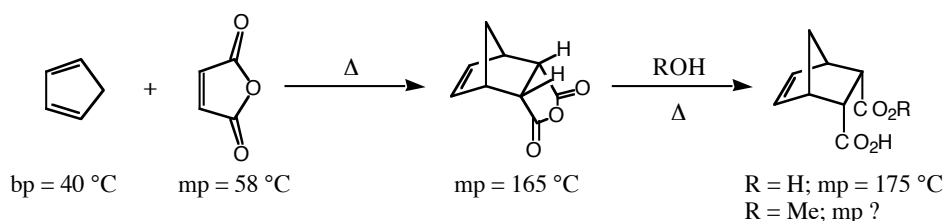


# CHEM-333: Lab experiment 11:

## The Diels-Alder Reaction:

### Read Chapter 32.

In this experiment, *endo*-norbornene-5,6-dicarboxylic anhydride will be synthesized from cyclopentadiene and maleic anhydride. The product will then be hydrolyzed to the diacid. Both products will be tested for unsaturation (carbon-carbon double bonds) by the Baeyer test (potassium permanganate).



The Diels-Alder reaction will be carried out on half the reaction scale given in the text book. The dicyclopentadiene will be cracked for you by your T.A. Heed the safety notes in the book.

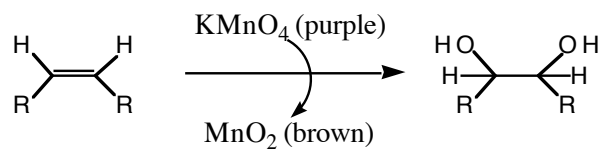
**Preparation of the Diels-Alder Adduct:** Place 2 g of maleic anhydride in a 50-mL Erlenmeyer flask and dissolve it in 8 mL of ethyl acetate by warming on a hot plate. Add 8 mL of hexane or petroleum ether, and then cool the solution in an ice bath. Obtain 2 mL freshly cracked cyclopentadiene and add it to the maleic anhydride solution. Swirl to mix. Wait until the product crystallizes from solution, then heat it on the hot plate to redissolve, and allow it to recrystallize. Collect the product by suction filtration, and record the melting point, weight, and percentage yield (calculate the molecular weights *before* you come to lab).

**Preparation of the Diacid:** Dissolve 1 g of the product anhydride in 15 mL distilled water in a 50 mL Erlenmeyer flask. Heat the mixture to boiling on a hot plate. After all the oil that has initially formed has dissolved, remove the heat.

**Preparation of the Monoester:** Dissolve 1 g of the product anhydride in 15 mL of methanol in a 50 mL Erlenmeyer flask. Carefully heat the mixture to boiling on a hot plate for 15-20 minutes, and allow to cool to room temperature.

If necessary induce crystallization by scratching or seeding. Collect the product by vacuum filtration, and record the melting point, weight, and percentage yield. Dissolve a small amount of the diacid and monoacid in distilled water, and determine the pH of the solution. Compare to the pH of distilled water.

**The Baeyer Test for Unsaturation:** A dilute solution of potassium permanganate oxidizes double bonds to diols. The disappearance of the permanganate color (purple) is a positive test for unsaturation. Note, however, that other compounds with oxidizable functional groups can also give a positive test (*e.g.* aldehydes).



**Procedure:** Dissolve a small amount of the compound to be tested (about 1/4 of a microspatula) in 2 mL ethanol. Add 0.1 M potassium permanganate dropwise (**caution:** stains). Note any changes. Count the number of drops until the purple color persists. Run a blank test: count how many drops of permanganate must be added to 2 mL of ethanol until the color persists. Test both the anhydride and the diacid for unsaturation, and record the results in your notebook.