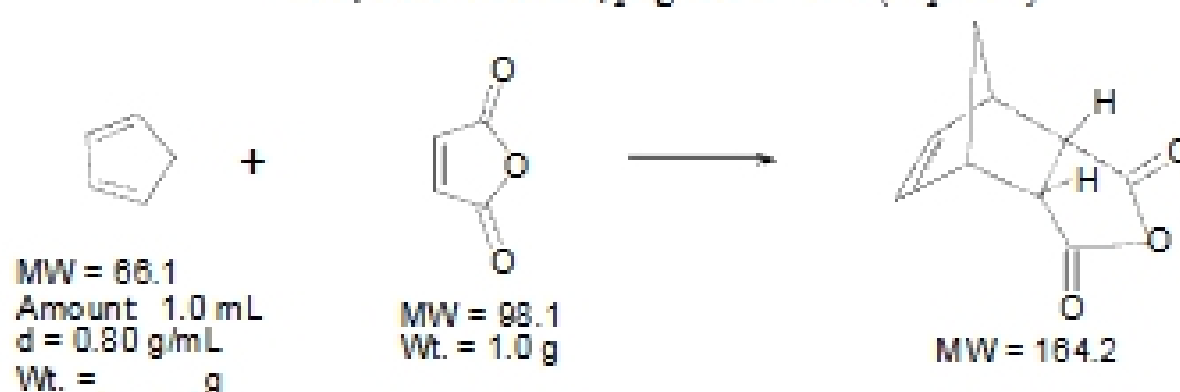


Diels-Alder Reaction

The Reaction of Cyclopentadiene with Maleic Anhydride to form *endo*-Norbornene-5,6-dicarboxylic anhydride

Period 8 – June 27, 2011

Bell, Clark & Taber, pages 271 – 272 (Expt 32B)



Conduct this experiment in your hood. Wear gloves while handling the chemicals in this experiment. **Cyclopentadiene** (and its dimer, dicyclopentadiene) is an irritant, is flammable, has an unpleasant odor and is harmful if inhaled — avoid breathing its vapors. **Maleic anhydride** is corrosive and toxic--wear gloves while handling it. **Ethyl acetate** and **hexanes** are flammable liquids and irritants.

1. Add 1.0 g of maleic anhydride to a 50 mL Erlenmeyer flask.
2. Add 4 mL of ethyl acetate to the maleic anhydride and dissolve it by warming the Erlenmeyer flask on a steam bath.
3. Allow the resulting maleic anhydride solution to cool slightly and then add 4 mL of hexanes.
4. Cool the resulting solution in an ice bath for a minute or two and then immediately have a TA add the cyclopentadiene to your cold solution (as described in Step 5). **NOTE:** If you cool for a prolonged period of time, the maleic anhydride may precipitate out of solution; if this happens, you should redissolve the precipitated solids by warming on the steambath and then briefly cooling in an ice bath before adding the cyclopentadiene (Step 5); a solution (no solids present!) is required for the next step.
5. To the cool maleic anhydride solution, add 1.0 mL of cold cyclopentadiene (this will be dispensed by the T.A.s at the community hoods; bring your Erlenmeyer containing the maleic anhydride to the hood) and immediately after the cyclopentadiene addition gently swirl the resulting solution for 10 – 15 seconds. A slight exothermic (heat releasing) reaction should occur.
6. After the swirling is done, allow the flask to stand in your hood at room temperature for 5 – 10 minutes to ensure that the reaction is complete. If white solids do not begin to form after 10 minutes, try scratching the inside of the Erlenmeyer flask below the level of the solution to induce crystallization. To ensure that crystallization of your product is complete, immerse the Erlenmeyer flask in an ice bath and allow the reaction mixture to cool for several minutes.
7. After crystallization of the product is complete, stir the crystalline mixture with a spatula to make sure there are no large chunks and filter off the solids using a Hirsch funnel with suction filtration.
8. Add 4 -5 mL of cold hexanes to the Erlenmeyer flask to help wash out the remaining solid product and pour this wash on the collected solids on the funnel with the vacuum remaining on.
9. Then allow the collected solids to air dry by continuing to suck air through the funnel for 5 – 6 minutes.

10. Weigh the dry collected solids, calculate your % yield.
11. Determine the melting point range of the product and compare it to the literature value.
12. Have a TA help you take the IR spectrum of your product and compare it to the reference IR spectrum of *endo*-norbornene-5,6-dicarboxylic anhydride that was distributed to you as a handout. Are they identical? If not, can you explain any differences between the 2 spectra?
13. Assign key infrared absorption bands in the spectra of maleic anhydride and the Diels-Alder product (*endo*-norbornene-5,6-dicarboxylic anhydride) which were provided to you as handouts.

Some Information about Cyclopentadiene

Cyclopentadiene is not a stable compound since, even at room temperature, it readily undergoes a Diels-Alder reaction with itself to form the stable dicyclopentadiene dimer. Dicyclopentadiene is commercially available and, on heating in a fractional distillation apparatus, readily undergoes a reverse Diels-Alder reaction to form 2 molecules of cyclopentadiene. For this lab, cyclopentadiene has been previously prepared by heating (cracking) the dicyclopentadiene dimer and collecting the lower boiling cyclopentadiene using a fractionating column. The cyclopentadiene is subsequently stored in an ultracold freezer (-80 °C) until it is needed. The cold temperature slows the cyclopentadiene Diels-Alder reaction so that it is not a significant problem during the week of this experiment.

