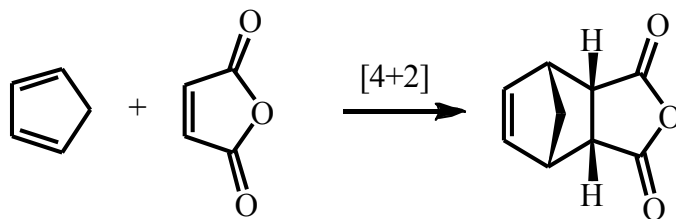


**The Diels-Alder Reaction. Preparation of *cis*-norbornene-5,6-*endo*-dicarboxylic anhydride**

**Reading** Experiment 47 in Pavia, and Section 17.7 in Klein, *Organic Chemistry* 2<sup>nd</sup> edition.



**Prelab**

Along with the usual **Name**, **Title**, **Purpose** and **Outline**, show the **Chemical Equation** and a complete **Reagent Table**, with the theoretical yield, in your notebook. (Hint: You can calculate the MW of the product from the molecular formula, which you can deduce from the structure).

**Prelab Exercise**

Find any information (mp, MW, correct name, etc.) about the product in the literature (Merck Index, CRC Handbook of Chemistry and Physics, Aldrich, on-line, etc.). Find as many names as you can for the product (IUPAC, common, or other – there are at least four different names). This information should be turned in on a separate piece of paper, but put the pertinent information in your Reagent Table, as well. Finally, find any data that you can about the *exo* isomer of the Diels-Alder product and include it.

**Procedure**

All glassware should be dry! Follow the procedure in the text, except scale up the reaction using the following amounts for reagents. Use a 25 mL Erlenmeyer flask 1.0 g of maleic anhydride, 4.0 mL of ethyl acetate, 4.0 mL of ligroin, and 1.0 mL of cyclopentadiene (which will be freshly distilled for you, just prior to the laboratory).

The final product should be crystallized as suggested in the text, but in the Erlenmeyer flask, not a Craig tube. Note that the product is water sensitive, since it is an anhydride. Any water that is present in the system can hydrolyze the anhydride to give a dicarboxylic acid, which has a much lower solubility than the anhydride. You may need to filter your solution before the final crystallization.

**To Complete the Experiment – Partial Report**

Your product should be dry by the end of the laboratory period. Determine the m.p. (lit. m.p. 164-165 °C), obtain an IR spectrum (KBr pellet) and calculate the percent yield for the final product. Interpret the <sup>1</sup>H NMR spectrum provided by the instructor. Answer all of the Questions at the end of the experiment in Pavia (p 409). (At the instructor's request you may also perform GC/MS analysis of your product.)

*Optional* Propose a method to analyze your product to determine the stereochemistry (*endo* or *exo*) of the product and include this discussion in your conclusion.