

## Chemistry 3719L – Lab Session 10

### Diels-Alder Cycloaddition of 1,3-Butadiene and Maleic Anhydride

#### Pre-lab reading from Zubrick:

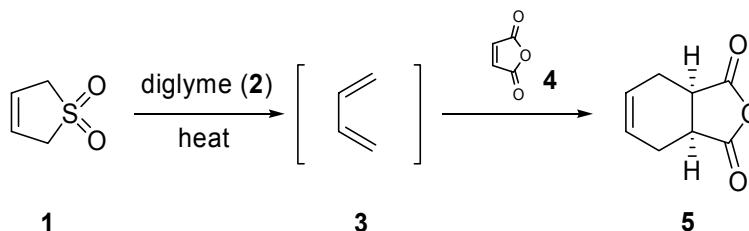
Chapter 23: Pages 201-204 (reflux)  
 Chapter 13: Whole Chapter – review recrystallization  
 Chapter 12: Pages 87-92 – review melting points

#### Aims

Sometimes a chemical we need to use as a reagent is too impractical to be used in the lab and we have to use precursors that allow for the release of the chemical we want under the reaction conditions. In this experiment you will generate 1,3-butadiene from butadiene sulfone in order to carry out a Diels-Alder reaction to make a carbocycle.

#### Reaction

Butadiene sulfone (**1**) is heated in diglyme (bis(2-methoxyethyl)ether, **2**) to produce 1,3-butadiene (**3**), which then reacts with maleic anhydride (**4**) *via* a Diels-Alder reaction to give the *cis*-cyclohex-4-ene-1,2-dicarboxylic anhydride product (**5**).



#### Procedure

Weigh out butadiene sulfone (3.6 g) and add it to a 25 mL round-bottomed flask. Then add the diglyme (7 mL) and a boiling stone. Weigh out the maleic anhydride (3.0 g) and add that to the mixture. Connect the reflux condenser (water in at the bottom, out at the top) and heat the mixture *gently* until bubbles appear in the mixture (SO<sub>2</sub> being evolved). Carefully remove the condenser, measure the temperature of the mixture with your thermometer (it should be ~140 °C), then replace the condenser. Continue heating for 5 minutes or until there are no longer any bubbles evolved. Cool the flask to room temperature then add cold water (35 mL) to induce crystallization. If the solid does not precipitate immediately “scratch” the bottom of the flask with a glass rod. Suction filter the solid and air dry it thoroughly. Record the weight and melting point of the solid.

**Keep good notes to help you write the report. Ask the TA if you don't follow the idea of “per cent yield” since this will be required as part of the report.**

**Synthesis Report = 20 pts**