

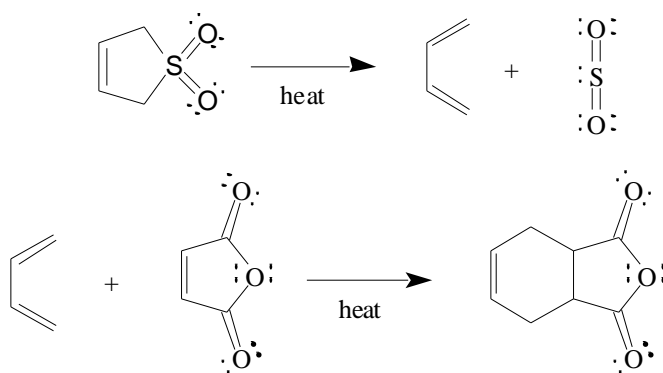
# Diels-Alder Reaction

## Introduction

In this reaction, a diene will be reacted with a dienophile to generate a new cyclohexene ring in a Diels-Alder reaction.

The diene is generated in the reaction vessel by heating butadiene sulfone until it decomposes to form 1,3-butadiene and sulfur dioxide. Evidence that this reaction is happening can be obtained by holding a wet piece of pH paper above the reflux condenser – the sulfur dioxide gas reacts with water on the paper to form an acid, which turns the pH paper red.

The dienophile is maleic anhydride. It must be kept dry to prevent it from reacting with water to form maleic acid, which will still undergo the Diels-Alder reaction, but which forms a product that does not easily crystallize under the work-up conditions we will use.



We will use p-xylene (the common name for 1,4-dimethylbenzene) as the solvent for this reaction because it has a much higher boiling point than most organic solvents, allowing the reaction to reflux at a high enough temperature for both of these reactions to occur. It is very important that the reaction not be overheated, however, because the product can decompose.

Reagents	volume	density	mass	MW	mmoles	eq
maleic anhydride	x	x	124 mg			1.00
butadiene sulfone	x	x				1.74
p-xylene						0.84
cis-4-cyclohexene-1,2-dicarboxylic anhydride	x	x				1.00

## Procedure

### Before you come to lab

- Read the entire lab to make sure you understand what you will be doing.
- Review the following techniques:

Refluxing a Reaction  
Filtering a Solid from Solution  
Measuring a Melting Point

- Do all of the calculations needed to fill out the reagent table.
- Write an introduction in your lab manual, making sure to include the reaction and the table.
- Answer the pre-lab questions on-line.

**During lab**

Bring me your calculations so that I can check your reagent amounts.

Set up the reaction:

- Set up a reflux condenser. Prepare a calcium chloride drying tube, and connect it to the reflux condenser using a yellow clip.
- Add butadiene sulfone, maleic anhydride, p-xylene, and a spin vane to a 5 ml conical vial. (Try not to use a dark colored spin vane, as the color may bleed into the solution and make it difficult to determine if the color is changing because of decomposition.)
- To help keep the xylene from escaping, seal the joint between the conical vial and the reflux condenser with a small amount of grease (just enough to make the ground glass not look frosted).

Run the reaction:

- Heat the reaction slowly to reflux.
- After your reaction has been refluxing for 2-3 minutes, wet a piece of pH paper with distilled water, and stick it into the end of your drying tube to test the vapors coming out.
- Continue refluxing for 30 minutes. Watch the reaction very carefully – if you observe the solution turning yellow, reduce the heat and add 2-3 drops of xylene. (Not more! If you add too much it won't crystallize out later. Adding xylene will not reverse the damage, only keep it from getting worse.) Then carefully heat it up to reflux again.

Isolate the product:

- Allow the reaction to cool just enough that you can handle it. Then remove the reaction from the hot plate, take out the spin vane, and add 0.25 ml of toluene. Swirl or stir it to mix in the toluene.
- Let it cool to near room temperature, then cool it in an ice bath. The product should crystallize from the solution; the network of crystals may appear totally solid (until you stir it up).
- Wipe any residue of the grease from the mouth of your conical vial with a paper towel. Then filter the crystals under vacuum, scraping the crystals out of the vial as best you can with a spatula (if you try to wash them down with a solvent, they will dissolve; water would contaminate the product).

Characterize the product:

- The crystals still contain some impurities from the solution, but they are clean enough to characterize. Observe the appearance of the product; it should be a white, needle-like crystals.
- Obtain the mass of the product and calculate the % yield. The average student yield for this reaction is 45%.
- Measure the melting point and compare it to the mp of the authentic product (101° C). Note the range of your melting point, and use it to determine the purity of your product.
- Your conclusion should discuss the evidence you saw of the reaction occurring, the appearance of the product, its mass and % yield, and the melting point (compare to authentic value and comment on purity).

**After lab:**

- Finish writing up your procedure and observations.
- Print out the questions on the next page and fill them out. Turn in this sheet with the carbon copies of your lab (in 2 separate piles).

## Questions for Diels-Alder Reaction

Name: \_\_\_\_\_

- 1) Draw the mechanism for the Diels-Alder reaction, labeling the diene and dienophile.
- 2) What would happen if cyclohexane (bp 80° C) were used as the solvent in this reaction?
- 3) Why do the vapors coming out of the reaction turn wet pH paper red?
- 4) If your reactions turns black, what happened?
- 5) Why is it important to use a drying tube to keep this reaction free from water?
- 6) Is the product we formed a racemic mixture of enantiomers, or is it a meso compound? Draw a picture of the product(s) showing the orientation of all stereocenters.
- 7) Why do you think excess butadiene sulfone was used instead of a 1.00 equivalents?
- 8) Why do you think the butadiene needed in this reaction generated by decomposing butadiene sulfone rather than just adding it? (Look up butadiene and think about its physical properties.)