

NOTES ON EXPERIMENT 2

The Diels-Alder Reaction: Preparation of hexaphenylbenzene

Chapter 53, Experiment 2, page 606-7. This experiment will be done in pairs.

Be sure to put a sand bath in the hood when you arrive in lab, and set it to heat up on the maximum heat setting. At the start you should also set a Mel-Temp machine to heat up, on close to maximum heat.

We will be using commercially made tetraphenylcyclopentadienone.

A flame will not be used- the reaction works well simply with a sand bath on full heat.

The mixture will not melt at the end of reaction as described in the book, but we have found that this is unnecessary.

Let the reaction run until much of the purple color (the ketone) has gone, and only then should you try to suck out excess alkyne. Do not spend too long doing this- excessive time (10 minutes or more) at this high temperature will result in charring and decomposition of the product. If you see a brownish color beginning to develop, remove the mixture from the heat **immediately**.

After washing the crystals with toluene at the end of the **diphenyl** ether recrystallization, you should also wash them with a further 0.5ml ice-cold **diethyl** ether. This makes the product very easy to dry.

Problems

1. Chapter 53, questions 2, 3. See p606 for the energy-minimized conformation.
2. In *meta*-dichlorobenzene, how many environments for hydrogen are there?
3. Identify the substance X from its NMR (^1H , ^{13}C) spectra, shown below.