

The Diels-Alder reaction between anthracene (diene) and maleic anhydride (dienophile or alkene) is demonstrated.

Reading Assignment: CER SYNTH-717

This experiment demonstrates the Diels-Alder reaction between a diene and an alkene by reflux in xylene. The product is recovered by vacuum filtration from the reaction mix. The product is characterized MP and chemical tests.

1. Preheat the hot plate and aluminum block at a heat setting of ~130-145 °C while you assemble your glassware.
2. Put together a microscale reflux set-up (**SYNTH 717, Fig. 3a**). Transfer the reactants to a 5-ml conical vial. The reactants should be weighed since yield is based on their amount. Record masses to the nearest mg. The xylene solvent can just be measured with a grad cylinder. Add your large magnetic stir bar (to regulate boiling and avoid bumping), and attach the water condenser. Water goes in the bottom slowly. Do not allow the condenser hoses to touch the top of the hotplate.
Reflux for 30 min. Start timing after condensation has been observed, indicating boiling.
3. The product will precipitate on cooling. Allow to cool to RT, then place in an ice bath to minimize solubility. Isolate the product by vacuum filtration using your small, 1.0 cm Hirsch funnel. Weigh to determine actual yield. Calculate theoretical yield & percent yield. Show calculations.
4. Run MP of product. Record MP range and apparatus used. Calculate % error for MP. Show calculations.
5. Tabulate your results in your lab book. Comment on the outcome.

The FTIR will not be run due to time constraints, however it would show a doublet in the carbonyl region indicating an anhydride functionality and numerous sharp peaks indicating the presence of aromatic rings.

Recrystallization could be done using hot xylene if the color or MP are bad.