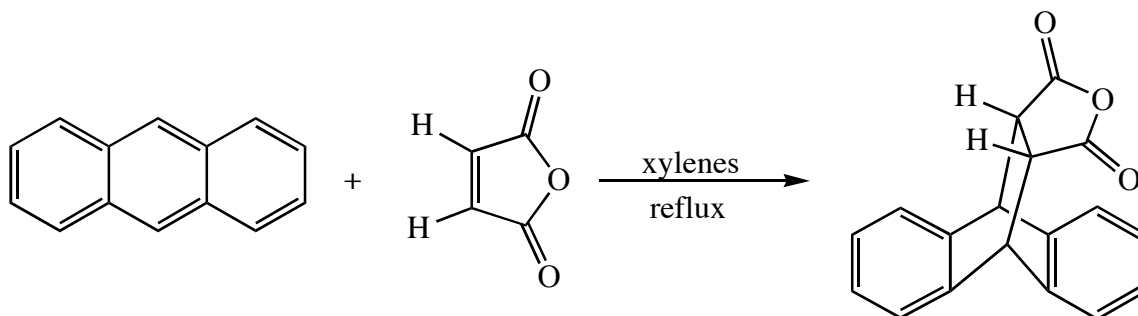


A Diels-Alder Reaction: 9,10-dihydroanthracene-9,10- α,β -succinic acid anhydride

Organic Lab -- AEMoody

Source: AEM Handout derived from D. W. Mayo, R. M. Pike, S. S. Butcher **Microscale Organic Laboratory**, 2nd Edition, Wiley: 1989; p 189-193.

The Diels-Alder reaction is a concerted 4+2 cycloaddition process. In general, the reagents include a diene and a dienophile (often an alkene), and the reaction mechanism is facilitated if the diene is electron-rich and the dienophile is electron-poor. The specific reaction that we will carry out is shown here.



Pre-Lab notes:

- High purity grade anthracene and maleic anhydride is recommended. (Although students probably will not need to do so, the anthracene can be recrystallized from 95% EtOH. Maleic anhydride is purified by sublimation.)
- A mixture of xylenes with a bp range of 137-140°C is sufficient and it should be dried over molecular sieves prior to use.
- The CAS registry number for this product (9,10-dihydroanthracene-9,10- α,β -succinic acid anhydride) is 5443-16-3. Use that number to access physical property information on this substance.

Experimental:

Start by heating an aluminum block on your hotplate using the highest setting, since it will need to be hot enough to vigorously reflux the xylenes (bp range of 137-140°C). Collect the following equipment: a 4 or 5 mL conical vial, an air condenser, and a drying tube. Charge the drying tube with a plug of cotton, calcium chloride, and a second plug of cotton. Add the following to the conical vial: 0.16 g of anthracene, 0.08 g of maleic anhydride, 2 mL of xylenes and a boiling chip. Place the condenser on top of the conical vial and add the drying tube on top of the condenser.

Place the conical vial in the correct hole of the aluminum block, and heat the reaction mixture to vigorous reflux. The initial yellowish color of the mixture should gradually disappear while it is refluxing. After it has refluxed for at least 30 minutes, let the mixture cool slowly to RT and then cool it further in an ice bath to complete the crystallization process.

Collect the crystals using suction filtration and wash them with ice-cold xylenes. Since the xylenes are hard to remove, we will use a trick to absorb the xylenes from the crystals. Put the crude crystals on your porous plate on a wax weighing paper. Also put a pile of small pieces of Parafilm (of approximately equal volume to your product crystals) on the porous plate nearby the crude product but not mixed with it. Cover both piles (the product and the Para film) with a single watch glass or beaker until the next lab period.

In the next lab period, measure the mass and the mp of your product. (It is usually not necessary to recrystallize this crude product, but if your melting point deviates dramatically from the literature value, you can recrystallize it from ethyl acetate. If you do recrystallize, then the recrystallized sample must dry until the next lab time before you measure its mass and its final mp.) Calculate the theoretical and percent yield for the reaction. This sample will be turned in for a grade.

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