DIELS-ALDER REACTION

TECHNIQUES REQUIRED : Reflux apparatus, Filtration (vacuum), Recrystallisation, Melting Point Determination, Yield calculation

EXPERIMENTAL PROCEDURE Work individually

- **Maleic anhydride is corrosive and toxic.**
- **Wear gloves, avoid contact with skin, avoid breathing the dust.**
- **Anthracene causes irritation.**
- **Wear gloves, avoid contact with skin, eyes and clothing.**
- **Xylene : flammable liquid and vapour. Irritant : avoid contact with skin and eyes Avoid inhaling the fumes and ingestion.**
- **Work in the fumehood at all times.**

The glassware components of the reflux apparatus need to be clean and dry.

In the fumehood, set up a hot plate stirrer with a metal heating block (check the fit size for your 25 mL round bottomed flask). Make sure the power cords are well away from the heated surface. **Do not turn the power on yet.**

Weigh out approximately 0.25 g of anthracene into a 25 mL round-bottom flask and add a stirrer bar. Carefully support the round bottom flask in an upright position in the "bowl" of a metal heating block and with the set-up centered over the middle of the stirrer hotplate. Add 5mL of xylene to the round bottom flask and start gentle stirring (enough to get the bar spinning consistently). Weigh out approximately 0.125 g of maleic anhydride and add it to the 25 ml round bottom flask.

Complete the reflux apparatus set up by connecting water hoses to the condenser, the tap and the outflow to the sink. Now insert the condenser into the round bottom flask joint checking to make sure it is seated correctly. Use a plastic Keck clip to attach the condenser to the round bottom flask.and support the condenser using a clamp to the fumehood racking / support stand (don't over tighten the clamp using the screws on both sides of the clamp, but make sure it adequately supports the apparatus in an upright position : it doesn't need to be rigid, just secure so it can't fall over). Get your TA to check the set up before turning on the heat or the gentle flow of cooling water.

Turn on the heat on the hot plate and adjust it to about 200-250. Bring the reaction mixture to a boil (monitor continually and adjust the heat control as necessary) and then gently heat (*i.e.*enough to maintain a steady reflux) for 30mins. Note any changes in the appearance of the reaction as it refluxes.

After the reflux period, remove the reflux apparatus from the heat source and let it cool to room temperature, then use an ice bath to help cool the flask for a further 5 mins. While the apparatus is

cooling, set up the vacuum filtration apparatus and put 5ml of ethyl acetate to cool in a test tube in an ice bath in a beaker.

Once cool, collect the crystalline product by vacuum filtration then wash the collected solid with about 3 mL of cold ethyl acetate to remove any organic impurities from the crystalline solid, then allow the crystals to dry in the Buchner funnel under vacuum for a few minutes. Record the weight your crude product and record the melting point.

Based on the melting point, if required, recrystallise approximately 200 mg of crude solid from a minimum volume of ethyl acetate (heat the solvent using a hot plate). Collect the purified solid and allow it to dry in the Buchner funnel under vacuum for a few minutes. Once dry, weigh the purified product and then re-measure the melting point of the recrystallised product.

Submit a labelled sample of your product to your TA.

Clean any glassware and equipment used (rinse with acetone, then wash with hot soapy water, rinse with distilled water then rinse with acetone) before returning it to your equipment drawer.

CLEAN UP

- All organic solutions (including rinse acetone) should be placed in the organic waste drum in the waste station fumehood.
- Aqueous waste should be poured into the aqueous waste container.
- Remember that you heated the metal heating block and the hot plate stirrer so it might be hot.

REPORT

The written report is due by the start time of your regularly scheduled laboratory section next week (i.e. you get one week to complete the report).

The report for this experiment is a formal report (*i.e.* no template). **More details on writing formal reports can be found in the introduction to this** student laboratory manual that discusses how to write reports and/or from "writing reports". Students often don't get the grades they would like because they make errors that are addressed in that section of the manual. Remember that more it not necessarily better. It is important to be accurate and concise rather than verbose and vague. There is a "hard" **4 page maximum** (Arial font 12, 1.5 line spacing as stated in the CHEM353 student laboratory manual) including the answers to the questions. Proper English should be used and it should be written in your own words. A **guide** to the sections you should include is shown below.

1. Title and Date, laboratory section Bxx, TA name
2. Introduction: Describe the purpose of the experiment, include balanced equations for the reactions.
3. Procedure
4. Results – g yield, % yields, melting points *etc.*

5. Analysis of product spectra
6. Discussion - (*e.g*. yield, purity, success, sources of error *etc.*)
7. Conclusions - summarise your results and discussion
8. References
9. Questions:

(a) Based on principles of aromaticity chemistry from CHEM353 explain the selectivity of the reaction.

(b) In a published article[4], the author reported the melting points for the isolated product from the 30 min. reaction in different solvents : reaction in ethyl acetate (218-220 ^{o}C) and in ethanol (217-219 ^{o}C). How would you interpret these results ?

(c) Draw the mechanism for the reaction of anthracene with maleic anhydride.