***The Diels-Alder Reaction: Identification of an Unknown Dienophile***

**Introduction**

The purpose of this experiment is to react 1,3-cyclopentadiene with either maleic acid or fumaric acid to form one of the Diels Alder adducts.

**Reaction**

**Mechanism**

**Physical Data**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Compound** | **Molar W. (g/mol)** | **Melting p. (°C)** | **Boiling p. (°C)** | **Density (g/cm3)** | **Solubility**  **g/L** | **Hazards** |
| **1,3-cyclopentadiene** | *66.1* | *-90* | *39-43* | *0.786* | *-* | *Flammable, toxic* |
| **Maleic anhydride** | *98.06* | *52.8* | *202* | *1.48* | *Reacts with water* | *Toxic, flammable, reactive, corrosive* |
| **Toluene** | *92.14* | *-95* | *111* | *0.87* | *0.47* | *Flammable, toxic* |
| **Hexanes** | *86.18* | *-94* | *69* | *0.6548* | *0.0095* | *Flammable* |
| **Ethyl acetate** | *88.11* | *-83.6* | *77.1* | *0.897* | *0.83* | *Flammable* |
| **Fumaric acid** | *116.07* | *-* | *165* | *1.635* | *0.63* | *Irritant* |
| **HCl** | *36.46* | *-62.25* | *108.58* | *1.18* | *Infinite* | *Corrosive and toxic* |
| **Iodine** | *53* | *113.7* | *184.3* | *4.933* | *-* | *Corrosive and toxic* |

*Sources: Handbook for Organic Chemistry,* ***CRC Handbook of Chemistry and Physics*** *(especially Section C: "Physical Constants of Organic Compounds" ), available at the information desk in the Science Library (in Norlin) and in the Organic Chemistry Stockroom.*

***Safety Precautions***

*Dicyclopentadiene is flammable and toxic. Unpleasant odor. Maleic acid and fumaric acid are irritants. Concentrated hydrochloric acid and iodine are corrosive and toxic.*

***Wastes***

*Organic Waste: Reaction solvents and filtrates when collecting the crystal*

*Solid chemical Waste: Pipets, weighing paper, filter paper.*

*Recovery Jars: the products – maleic acid adduct and fumaric acid adduct.*

**Procedure**

1. Dicyclopentadiene should be crecked to cyclopentadiene in a single apparatus in the main fume hood for all the students.
   1. The TA will supervise this procedure, or have it done before the lab.
2. Meanwhile the cyclopentadiene is being cracked. Wigh 1.5g of the unknown compound and place it into a 50mL round-bottom flask with a stirbar.
3. Add 15mL of 95% ethanol and place the condenser.
4. Set up the apparatus for the reflux reactions.
   1. Refer to figure 15.2 in your Experimental book.
5. Turn on the stirrer to mix up.
6. Get the cyclopentadiene from the fume hood in a vial and capped.
7. Remove your condenser and quickly add the cyclopentadiene to the reaction mixture, place the condenser back on.
8. Turn on the water hoses, and heat the flask on medium magnitude.
   1. Note the time it starts to boil or refluxes.
9. After 10 minutes have been passed of reflux.
   1. Remove the condenser
   2. Place a TLC spotter into the flask.
10. Spot the sample onto a TLC plate.
    1. Have the dienophile TLC standard as well.
    2. Develop the plate in 20:80:1 (hexanes:ethyl acetate: acetic acid)
    3. Use the silica iodine in the hood to stain it.
11. Sketch the plate into your lab notebook.
12. Follow the waste instructions stated before the procedure section.
13. When the reaction has been at reflux for around 30 minutes, its most likely at completion.
14. Remove the condenser
15. Add 15mL of water to the reaction flask.
    1. Heat it
16. When the volume of the liquid has been reduced by half, add 15mL of water and heat it.
17. Repeat this for 30 minutes.
18. Remove the flask from the heat source
19. Collect 1mL of Concentration HCl from the hood through a Pasteur pipet and a vial.
20. Add HCl to the reaction flask
    1. Makes the pH drop to 1
21. Cool the mixture to room temperature
22. Place it into an ice bath for 5 minutes
    1. Crystals should be appearing by now
23. Isolate the product with vacuum filtration
24. Rinse the reaction flask with cold water
25. Let the crystals air-dry for some time
26. Determine the melting point and yield of the product.
27. Perform an NMR, by obtaining an NMR tube from the TA
28. Submit a sample of the compound for NMR analysis
29. You’ll get the results in a few days