21. Hydrolysis of Maleic Anhydride

Anhydrides are derivatives of carboxylic acids. All derivatives of carboxylic acids can be converted to the corresponding acid through reaction with water. Some less reactive derivatives require an catalyst for the reaction to occur in a reasonable time, but anhydrides do not.

When a cyclic anhydride, such as maleic anhydride, is the dienophile in a Diels-Alder reaction, the product is also an anhydride. Hydrolysis of the anhydride often follows as the next reaction in a synthetic sequence. Instead of hydrolyzing the anhydride that you made in the previous experiment, we will hydrolyze maleic anhydride itself in this experiment.

PRE-EXPERIMENT ASSIGNMENT

Study this chapter of the manual, the lecture notes on the Chemistry Department web site, and the sections on hydrolysis of carboxylic acid derivatives in your lecture textbook. Do the first parts of your notebook writeup.

A student who has prepared for the Hydrolysis of Maleic Anhydride experiment should be able to:

1. Define and/or identify: reflux and anhydride, and be prepared to give examples of the latter.
2. Give a balanced equation for the hydrolysis of maleic anhydride (and of any other anhydride).
3. Calculate the theoretical yield and the percent yield for this and similar experiments given the necessary data, and perform any of the intermediate calculations required by this process.
4. Draw the structure given the name, or give the name from the structure, of the compounds used in the day’s experiment, and give the role of each (reactant, solvent, catalyst, etc.).
5. Identify and explain safety considerations for this experiment.
6. Perform the day’s experiments safely and successfully.
7. Identify and explain the effects of failure to dry the reaction product on the percent yield and the melting point range of the product.

**The Reaction**

Turn on your sand bath and set it to about 30. Obtain about 0.2 g of maleic anhydride and add it to a test tube. Add a boiling stick and then 0.5 mL of water. Heat the test tube for 5 minutes. The mixture should boil and the solids dissolve. **The anhydride melts before it dissolves. Make sure that it has dissolved, not just melted.** Remove the boiling stick and allow the solution to cool to room temperature.

**The Workup**

After the reaction mixture has cooled to room temperature, add 1 mL of tert-butyl methyl ether (MTBE) to the test tube. Mix thoroughly and then allow the layers to separate. Using a Pasteur pipette, transfer the organic layer to a clean test tube, leaving the aqueous layer behind in the original test tube.

Add a second 1 mL portion of MTBE to the aqueous solution in the original test tube. (If two layers do not form, you had the organic and aqueous layers mixed up in the previous step.) Mix thoroughly and then allow the layers to separate. Using a Pasteur pipette, transfer the organic layer to same test tube that you put the first organic layer in. Save the aqueous layer until the experiment is complete, just in case.

Add a small amount of anhydrous sodium sulfate, a drying agent, to the bottom of the test tube that contains the organic layer. Swirl it and observe the solid. If it clumps together, it has picked up water. If so, add a little more fresh drying agent, swirl, and observe the freshly-added drying agent. When the fresh solid does not clump, it is no longer picking up water, so the solution should be dry at that point. It should also be clear rather than cloudy. Be careful not to add so much drying agent that it absorbs or soaks up all of the solution.

Weigh a watch glass and set it in the hood. Decant the solution (that is, pour the liquid off, leaving the solid behind) from the test tube onto the watch glass so that the solvent evaporates. Weigh the watch glass after the solvent has evaporated and calculate the mass of the maleic acid. Determine its melting point.
CLEANUP

Used boiling sticks can be thrown in the trash. Pasteur pipettes are placed in the broken glass box after use. Excess solvents are poured into the non-halogenated liquid waste bottle in the hood, and your product is placed in the non-halogenated solid waste jar if your instructor does not collect it.

POST-EXPERIMENT ASSIGNMENT

Write the lab report and have it ready to turn in by the beginning of the next lab. Your writeup of this experiment (as for every synthesis experiment) should include a calculation of the theoretical and percentage yields. Your Conclusions section should include a comparison of the melting point of your product with that reported in the literature for maleic acid and possible reasons for any difference. If your percent yield was not 100%, you should explain where the missing material might have gone (or where extra material might have come from). Prepare for the hydrolysis of maleic anhydride portion of the next quiz.