TEACHER REFERENCE PAGES—ESTERIFICATION LAB

USING THE MICRO MINI-LAB

Assembly of the Reflux and Distilling Apparatus

1. **HANDLE EQUIPMENT CAREFULLY.** Do not force any fittings or caps. Wash completely with distilled water followed by an acetone rinse at the end of the lab.

2. To construct the apparatus in Figure 1, you will need: a hot plate with magnetic stirrer, sand bath about half filled with sand, drying tube, 3mL conical vial, cooling condenser, magnetic spin vane, metal thermometer which reads to at least 200°C and its clamp, two 3' lengths of Tygon tubing, cooling tube (with cotton and CaCl₂) and a clamp for the apparatus.

3. Place sand bath on hot plate and heat to between 160°C and 180°C. Be careful not to overheat the sand bath. Adjust as necessary to maintain the temperature throughout the lab.

4. While the sand bath is heating construct the apparatus as in Figure 1. When making the connections between the different parts slide the cap over the ground glass followed by a large rubber O-ring. Secure the parts by screwing the cap down. Make sure O-rings are in place. Caps should be snug, but **DO NOT FORCE CAPS.** Before you connect the 3mL vial add the chemicals and the magnetic spin vane. Secure the apparatus to the ring stand with one clamp only so that the glassware can be rotated without breaking.

5. Hook up input cooling water at the lower inlet with the outlet at the upper inlet. **Slowly** turn on water and check for leaks.

6. When the reaction is complete, carefully remove the conical vial and transfer the solution as directed in the lab write-up. Disassemble the apparatus, let it cool and clean with distilled water and acetone.

7. Construct the distilling apparatus as in Figure 2. Use the 3mL conical vial, Hickman still, cooling condenser, hot plate and sand bath, thermometer, clamp, Pasteur pipet and magnetic spin vane with spinning band. Make sure the glass wool is wrapped around the bottom of the Hickman still. Connect cooling condenser as in Figure 1, but do not run water through it; the boiling point of the ester is high enough to make that unnecessary. Do not use the drying tube and leave the top open. Use the magnetic vane with spinning band and spin it as slowly as possible. Make sure that the extraction tube on the Hickman still is capped with a vinyl plug so that no oil will escape. At the end of the lab extract oil from the Hickman still with the Pasteur pipet and save in a sample vial (be sure to weigh the vial first.)