Synthesis of Propanol Acetate – Reminiscent of Pear…

Required Chemicals/Amounts and Glassware

16 ml of propanol

22ml of glacial acetic acid

3.6ml of concentrated sulfuric acid

100ml of cold 4M NaCl

60ml of 1M $NaHCO_3$ – CO_2

5g sodium sulfate

100ml round-bottom flask

Separatory funnel

50ml Erlenmeyer flask

Distillation apparatus

2-3 vials

Step 1

Pour 16 ml of propanol and 22ml of glacial acetic acid into a 100ml round-bottom flask. Carefully, with swirling, add 3.6ml of concentrated sulfuric acid to the contents of the flask. Add two or three boiling stones.

Assemble a reflux apparatus and bring the mixture to a boiling using a heating mantle as a source of heat (see figure 1, next page). Allow to reflux for one hour. It will turn a slight pink color during this time.

While the reflux is occurring, prepare 100ml of cold 4M NaCl and 60ml of 1M $NaHCO_3$ – CO_2 in order to use for the final step.

Step 2

After the reflux is complete, remove the heating mantle and let the mixture cool to room temperature. Pour the cooled mixture into a separatory funnel, and carefully add 50 ml of the cold 4M NaCl (bubbles may form when the reaction begins, wait until after it is complete before placing stopper on top). Stopper it, shake the funnel with the mixture inside a few times, and vent to allow gas to be released (you will hear the sound of the gas). Repeat the shake and release method until all the gas has escaped. Once all the gas has been vented and the ester has clearly separated, place funnel in ring stand, remove stopper, and discard the aqueous layer allowing the ester to remain in the funnel.

Pour 25 ml of the 1M $NaHCO_3$ – CO_2 into the separatory funnel, stopper it, shake the funnel with the mixture inside a few times, and vent to allow gas to be released. Once all gas has been released, funnel out the aqueous layer. When a drop of the lower aqueous extract turns red litmus paper blue, you have finished the neutralization of all the acid.

Place the remaining ester in a 50ml Erlenmeyer flask. Place about 5g (almost a scoop full) of sodium sulfate. The water will clump with the sodium sulfate, leaving no reason to swirl the mixture in the flask. Wait about five minutes and pour liquid mixture into tube before continuing onto the next procedure of lab.

Step 3

Pour mixture into distillation apparatus (make sure thermometer is not touching glass). Heat the distillation flask with a heating mantle and collect the liquid that has a boiling point of around 100^{o}C (see figure 2). Make sure to previously weigh vials before collecting liquid. Once all liquid has been collected, removed flask from heating mantle to ensure you are not heating up an empty glass. Reweigh the vial and determine your percent yield.

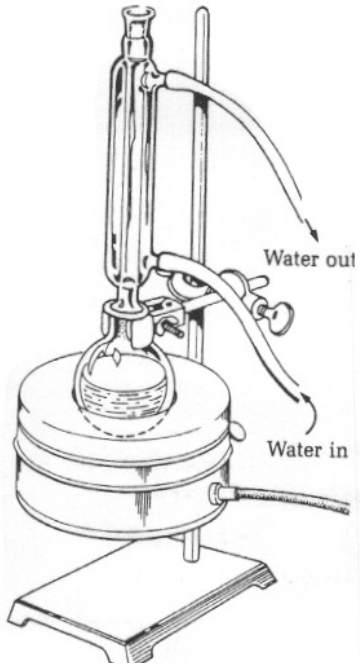

Figure 1. Reflux condenser set-up. Note that it is set up above the bench top.

Figure 2. Distillation apparatus set-up.