

## Step 1

### 5.0 g scale

1. Put a metal pin or stirrer into the oil bath and heat it up to by setting the thermometer up to 130 °C.
2. Turn on the stirring knob to stir up the oil while it heats up.
3. Put a small magnetic stirring bar into a 100 mL round bottomed flask.
4. Weigh 1.15 g NaOAc into the 100 mL round bottomed flask.
5. Pipette about 24 mL acetic anhydride solvent into the 100 mL round bottomed flask with the NaOAc.
6. Slowly immerse the 100 mL round bottomed flask with the contents into the heating oil bath and clamp the 100 mL round bottomed flask. *(the solutions stir and heats up to dissolve the solids)*
7. Cap the 100 mL round bottomed flask with a plastic cork or cap.
8. Weigh 5.0 g of glucose.
9. Insert a plastic funnel into the neck of the 100 mL round bottomed flask and scoop the glucose into the reaction mixture in small portions for about 15 mins.
10. Wash the neck of the funnel with about 1 mL of acetic anhydride solvent to push all any remained glucose in the funnel into the 100 mL round bottomed flask.
11. **Skip step 10 if there are no glucose solids left in the funnel.**
12. Securely clamp the reaction flask.
13. Increase the temperature of the reaction mixture to 160 °C by using the thermometer to check.
14. Attach a reflux apparatus on the reaction flask by clamping in position with the reaction flask. Connect one end of a rubber tube to the top port of the condenser and direct it into the drain. Connect another tube to the bottom port and attach the other end to the water supply.
15. Turn on the faucet to start the water flow. (this allows the water vapor to condense back into the reaction flask).

16. Once refluxing starts (when you see the first drop of condensed vapor into the reaction flask), start your clock and keep the refluxing till 5 – 6 mins.
17. After the 5 or 6 mins of refluxing, raise the reaction flask up and above the oil bath to cool the slightly yellow reaction mixture in the air. **Do not** remove the reflux apparatus from the reaction flask while it cools in the air.
18. Start to clean up while your reaction mixture cools to room temperature.
19. Fill the sonicator with more than half with water and turn it on.
20. Fill a 250 mL beaker with about half with water and allow it to sit in the sonicator.
21. After your reaction flask is cooled enough to touch, unclamp it and insert the reaction flask into the 250 mL flask in the sonicator.
22. Add some ice to about half of the 100 mL round bottomed flask which contains the reaction mixture and sonicate for about 10 – 15 mins. (quenching the acetic anhydride).
23. After the sonication, cap and keep the reaction flask with its contents in the fridge for overnight.

#### Work up.

24. **Filtration:** Set up your Buchner funnel apparatus to perform a vacuum filtration. *(by clamping your vacuum flask in place, insert into the neck a rubber disc and then gently place the Buchner funnel on top of the rubber disc. Place your filter paper inside the buchner funnel, attach one end of the rubber tubing to the outlet on the vacuum flask, then attach the other end to the arm of the faucet, turn on the faucet to turn on the suction).*
25. Pour all the solids from the reaction flask into the bunchner funnel and Wash the white solids with distilled water for about 2 – 3 times to remove all or almost all acetic acid.
26. Carefully transfer all the white solids into a 100 mL beaker and dissolve the solids with 20 mL DCM. Rinse the filter paper with some amount of DCM to dissolve any remained portions of the white solids into the beaker.
27. Prepare about 60 mL saturated  $\text{NaHCO}_3$  solution.
28. Using the Pasteur pipette (or droppers) wash the DCM layer in the 100 mL beaker with **30 mL** of the supernatant (**only** the upper liquid layer) of the saturated  $\text{NaHCO}_3$  solution.
29. **Extraction:** Clamp a separatory funnel and put a glass funnel on the neck of the separatory funnel.

30. Pour the reaction mixture into the separatory funnel via the glass funnel from the beaker.
31. Rinse the beaker, funnel, neck of the funnel with DCM into the separatory funnel.
32. Cap the separatory funnel with the stopcock, shake the separatory funnel and release the pressure from the separatory funnel by opening the turning the stopcock. Repeat this for about 4 to 5 times till you hear no popping sound whenever you open the valve. (*do this under the fumehood*).
33. Clamp the separatory funnel to let the solution to settle and separate. While you are waiting for the partitions, make a saturated solution of  $\text{NaHCO}_3$  by dispensing about 15 to 20 mL distilled water into the beaker that already contained  $\text{NaHCO}_3$  solids.
34. Add about 9 mL supernatant of the  $\text{NaHCO}_3$  saturated solution with Pasteur pipette solutions in the separatory funnel.
35. Repeat step 32.
36. Clamp the separatory funnel from 35 to allow the solutions to settle and separate.
37. Turn the stopcock of the separatory funnel to deliver the bottom organic layer (which has the desired analyte) of the partition into the Erlenmeyer flask set directly below the separatory funnel.
38. Perform another extraction by rinsing the 100 mL beaker used, the 100 mL round bottomed flask, glass funnel, with DCM.
39. **Drying:** dry the reaction solution in the Erlenmeyer flask with reasonable amount of  $\text{Na}_2\text{SO}_4$ . Swirl the Erlenmeyer flask and allow the content in it to settle.

### Rotary evaporation

40. After step 39, block the neck of a glass funnel with cotton and transfer the content in the Erlenmeyer flask into 150 mL round bottomed flask through the funnel.
41. Wash the Erlenmeyer flask and neck of the round bottomed flask with DCM about 2-3 times and add to the contents in the 150 mL round bottomed flask.
42. Use rotary evaporator to concentrate the solution in the 150 mL bottomed flask by removing the DCM.
43. Cap the 150 mL round bottomed flask with the analyte and keep it for the next steps.
44. Label the 150 mL round bottomed flasks, leave it under the hood to dry, clean up, wash your hands.