Procedure

You will work with a partner in this laboratory.

Part I: Preparation of Crude ASA

1. Prepare a boiling water bath. Put ~300 mL of tap water in a 600 mL beaker on a hot plate. Add one or two boiling stones and turn on the hot plate to high. Do **NOT** add boiling stones to a liquid that is already hot. This may result in sudden, violent boiling. A ring clamp can stabilize the beaker. Once the water is boiling, reduce the heat on the hot plate.

2. "Tare" a 125 mL Erlenmeyer flask on an open pan balance and add ~2.1 g of salicylic acid to it. Record the mass of salicylic acid to two decimal places.

3. In the fumehood add 4 mL (density = 1.08 g mL^{-1}) of acetic anhydride to the Erlenmeyer flask using the dispenser. Using a disposable pipette add ~5 drops of concentrated sulphuric acid (H_2SO_4) to the flask. Use great care. Both acetic anhydride and sulphuric acid are highly corrosive. If you get any on your skin, immediately flush with large amounts of cold, running water. If any is spilled on the bench, alert the teaching assistant.

4. Gently swirl the flask to mix the contents. The solid may or may not completely dissolve.

5. Place the Erlenmeyer flask in the water bath and hold it in place with a clamp. Make sure no water from the beaker gets in the flask. Heat the flask for 15 - 20 minutes. At this point all of the solid should have dissolved.

6. Prepare an ice bath by combining ice and tap water in a 1 L beaker. Cool ~60 mL of deionized water in a beaker.

7. Remove the flask from the hot water bath. The flask will be hot, so use the clamp as a handle. In 1 - 2 mL portions slowly add 10 mL (use a graduated cylinder) of the chilled water. Swirl the mixture gently after each addition.

8. Cool the flask in the ice-water bath to induce crystallization of the ASA.

9. Add 25 mL of cold, distilled water. With a clean stirring rod carefully break up any large lumps of crystals that form.

10. Collect the crystals of ASA using suction filtration and a Buchner funnel. See the beginning of the lab manual for instructions on how to perform a suction filtration.

Part II: Recrystallization of the ASA

1. Obtain ~50 mL of 3:1 water / ethanol solvent in a small beaker. This is the solvent used in the recrystallization.

2. Reserve a small portion of the crude ASA to test its melting point. Transfer the remaining crude ASA to a 50 mL Erlenmeyer flask.

3. On the hot plate warm the water / ethanol solvent until it is just boiling.

4. Add a small amount of the solvent to a 150 mL beaker. Cover the beaker with a stemless funnel. In the funnel place a piece of fluted filter paper which has been moistened with a little bit of the water / ethanol solvent. Place the beaker and funnel on the hot plate.

5. Place the flask with the crude ASA on the hot plate and add the hot solvent until the ASA just dissolves. Add an extra ~2 mL of solvent.

6. Now, working quickly empty the 150 mL beaker, replace the funnel on top of the beaker. Immediately pour the hot ASA solution onto the hot filter paper and once some liquid is in the beaker, place the beaker on the hot plate. Do not place the empty beaker on the hot plate - it will likely break. If crystals form on the filter paper, use a little hot solvent to re-dissolve them.

7. Once the filtration is complete remove the beaker and allow it to cool (undisturbed). Crystals should form quite easily, but if not, scratch the side of the beaker with a stirring rod to induce crystallization.

8. After the beaker has cooled to room temperature, cool it a little further by placing it in an ice bath.

9. The re-crystallized ASA should be quite pure. Collect the purified ASA using a Buchner funnel and suction filtration.

10. Weigh a clean, dry watch glass on an open pan balance and record the mass. Transfer the purified ASA to the watch glass and re-weigh it. Record this mass and the mass of ASA recovered.

11. Using a scoopula, grind up some of the purified ASA on the watch glass and put a few of the ground crystals in a melting point tube.

12. Using the Mel-Temp (see the instructions below), determine the melting points of the crude and the purified ASA.

Using the "Mel-Temp" Melting Point Apparatus

A Mel-Temp apparatus allows the rapid and accurate determination of melting points to roughly 525^{0}C.

1. Use a thoroughly dry sample that has been finely ground. This ensures uniform distribution of any impurities. The ASA sample can be ground on the watch glass using the stirring rod or the end of a scoopula.

2. Put sufficient solid in the capillary tube to fill the capillary tube ~ 3 - 4 mm deep. This can be tricky, but the easiest method is probably to dip the open end of the capillary into some of the solid and then gently tap the closed end to pack the solid into the tube.

3. Place the thermometer into the well of the Mel-Temp. Be very careful - these are mercury thermometers. Report any breakage or mercury spill to your instructor immediately. If the Mel-Temp apparatus has been set up for you, do not handle the thermometer.

4. Plug in the unit and turn it on.

5. Put the capillary tube(s) into the holder. The Mel-Temp can accommodate up to 3 capillaries at one time.

6. Initially you may heat the samples rapidly (at a setting of 5 or 6). Once the temperature reaches ~120^{0}C reduce the setting to about 0.5.

7. Observe the samples with your eye about 6" (15 cm) from the lens.

8. Record the temperature at the first sign of the formation of liquid.

9. Record the temperature once the last bit of solid turns to liquid. The temperature recorded in step 8 and this temperature represent the melting point range for your sample.

10. Once the melting point has been measured turn off the Mel-Temp and allow it to cool down. To do a second determination you will have to allow the apparatus to cool below the expected melting range.

1. If, for example, a student mistakenly added only 2 mL of acetic anhydride, would the theoretical yield be higher, lower or unaffected? Why?

2. If the purified ASA is not completely dry before the final weighing, how will this affect the theoretical yield and the calculated percent yield? What could be done to ensure the ASA was as dry as possible?

3. When esters are exposed to moisture they can be hydrolyzed forming a carboxylic acid and an alcohol (see reaction (5.1)). If ASA is hydrolyzed what will the products be? What is a quick test to determine if ASA in the home has been hydrolyzed?

4. A student is given two pure samples, 'A' and 'B'. Describe a simple procedure (based on the methods used in this experiment) the student could use to determine if 'A' and 'B' are the same compound.

Results, Discussion and Questions

Mass of salicylic acid used: 2.12 g

Mass of acetic anhydride: 4~~m~~.00 ml

Ques 1 Calculate the theoretical yield of ASA. The theoretical yield is the maximum amount of product that could be expected from the given amount of reactants.

Mass of clean, dry watch glass 31.4002 g

Mass of watch glass + ASA 32.1865

Mass of purified ASA 0.7863

Ques 2 Calculate the percent yield of ASA (the percentage of the theoretical yield that was actually recovered).

Melting Range of crude ASA 74.5 – 79.5 °C

Melting Range of purified ASA 80.5 – 89.0 °C