The Synthesis of Salicylic Acid from Wintergreen Oil – Instructor Guide

Learning Objectives:

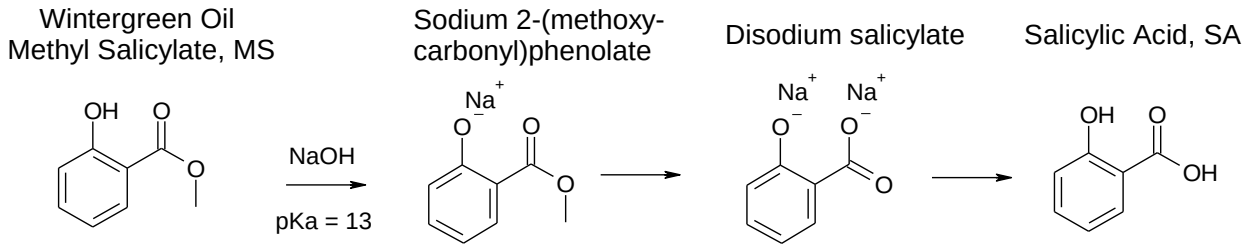
- Perform typical organic synthesis chemistry laboratory techniques
  - Equipment setup, Refluxing, Thermocouple controller use, Crystallization, Filtration, Drying,
- Perform Hydrolysis Reaction and Neutralization
- Perform identification and quantitative analyses using: UV, IR, MP
- Calculate Moles, Grams, Stoichiometry, Yields and Purity

Background:

Our new company is preparing to manufacture aspirin by synthesis from evergreen oil. There is a procedure developed intended for manufacturing on a large scale (100 kg) that requires testing for reproducibility as regards to yield and purity of the API. The two step synthesis encompasses aqueous base hydrolysis of wintergreen oil (methyl salicylate, MS) to salicylic acid (SA) followed by acetylation to acetylsalicylic acid (aspirin, ASA).

Scheme 1: Synthesis of Aspirin

STEP 1: Hydrolysis of methyl salicylate



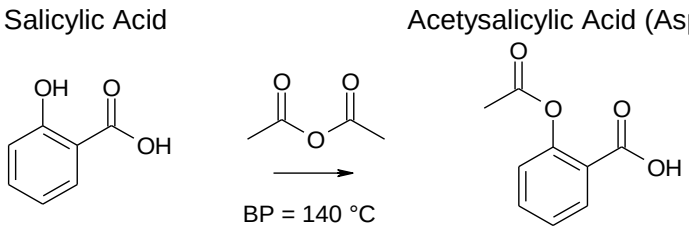
MW = 152.15
MF = $C_8H_8O_3$
d = 1.18 g/ml
pKa = 9.8

MW = 174.13
MF = $C_8H_7O_3Na$

MW = 159.10
MF = $C_7H_4O_3Na_2$

MW = 138.12
MF = $C_7H_6O_3$
MP = 159 °C
pKa = 2.9

STEP 2: Acetylation of Salicylic Acid

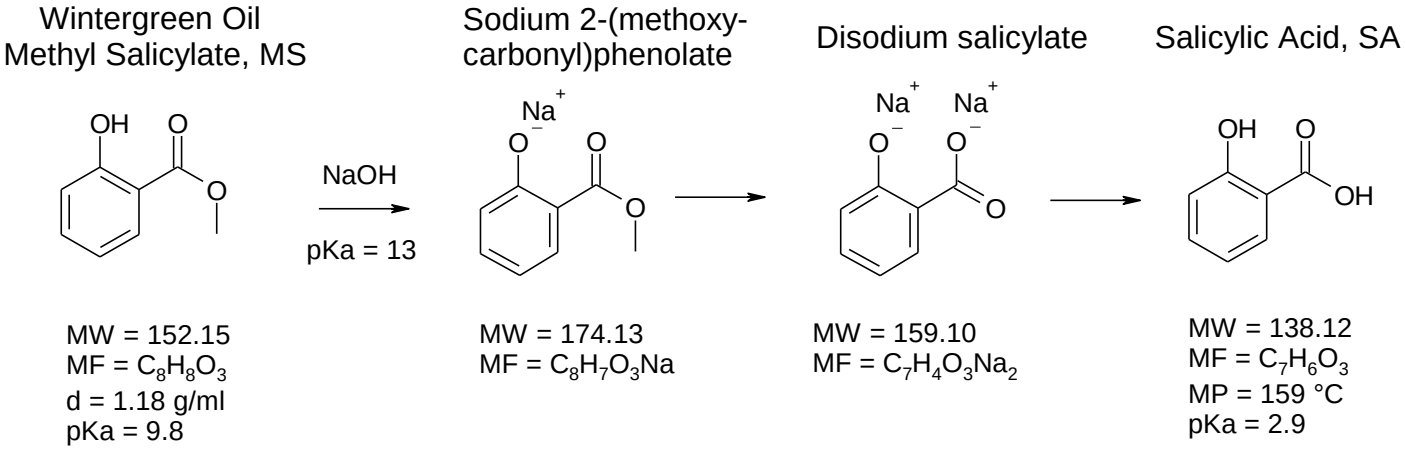


MW = 138.12
MF = $C_7H_6O_3$
MP = 159 °C

MW = 180.16
MF = $C_9H_8O_4$
MP = 135 °C
pKa = 3.5

Experimental Procedures

STEP 1: Hydrolysis of methyl salicylate

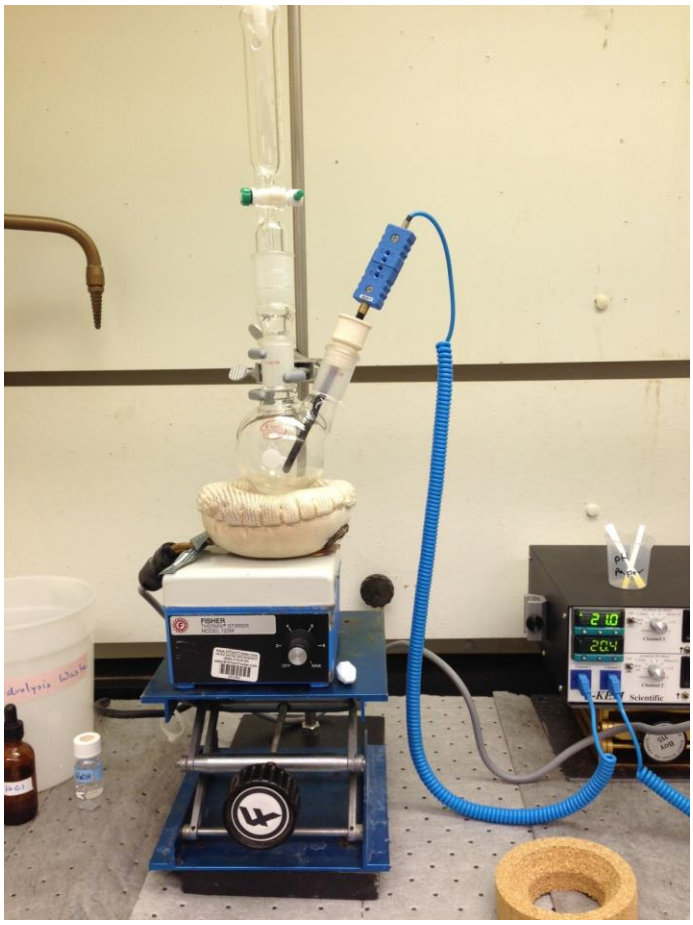


Important: Check off each step as you complete that step

- ☐ **"Break the reaction down"** by removing the pressure equalizing addition funnel, the plastic funnel, the thermocouple probe and the 250 mL 2-necked round bottom flask.
- ☐ Place a clean 250 mL 2-necked round bottom flask on a cork ring on a top load balance, zero the balance, add ~4.0 g of wintergreen oil with a pipette and record the weight to the nearest 0.01 g. Record the mass here: _____.
- ☐ Gently add a stir bar by sliding down the side of the flask. **Do not drop** the stir bar directly into the flask.
- ☐ Securely clamp the reaction flask to a ring stand
- ☐ Place the heating mantle on top of the magnetic stirrer and raise the jack until it is slug to the flask.
- ☐ Place the pressure equalizing addition funnel and the short stem funnel to the top hole of the 250 mL flask.
- ☐ Place the thermocouple probe assembly into the side hole of the 250 mL round bottom flask.
- ☐ Add 25 mL of water to a clean 100-mL beaker labeled "3 N HCl" using the bottle top dispenser.
- ☐ **Check that the bottom valve of the addition funnel is closed.**
- ☐ Pour 25 mL of water into the addition funnel using the labeled 100-mL beaker labeled "3 N HCL"

- Practice adding the water dropwise by **carefully and slowly opening the bottom valve** of the addition funnel. Add the entire 25 mL dropwise
  - Is this a homogeneous or heterogeneous mixture? _____
- Turn on the stirrer to mix.

Your reaction setup should look like PICTURE 1

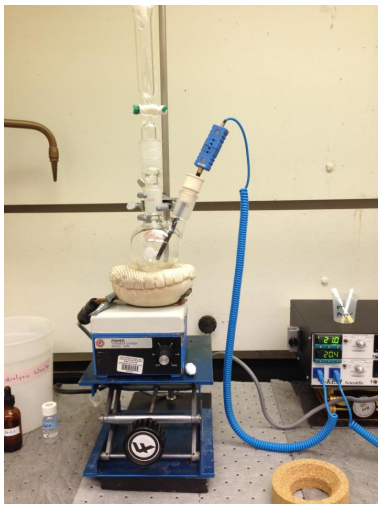


Picture 1

- **Check that the bottom valve of the addition funnel is closed.**
- Pour 15 mL of 6*M* aqueous NaOH into the addition funnel using the vial labeled **6*M* NaOH**
  - **WARNING: sodium hydroxide (NaOH) is very caustic to the skin. If you get any on your skin, let the instructors know and immediately wash it off at the sink with cold water for 10 minutes.**
  - **Replace your gloves immediately if you get any of the hydroxide solution on them. Do not touch anything with contaminated gloves.**

- ☐ Add the first 5 mL of the 6*M* NaOH dropwise over 3-4 minutes by **carefully and slowly opening the bottom valve** of the addition funnel. <u>You must do this carefully to avoid creating a gum ball in the reaction flask</u>.
  - Does anything physically change during this addition?
  - What do you think is happening? (Hint: the pKa of methyl salicylate is 9.8, look at the reaction scheme above.)
  - Does the temperature of the reaction change? If so, how and why?
- ☐ Add the remaining hydroxide to the flask over 1 minute by opening the bottom valve of the addition funnel.
- ☐ After complete addition of the hydroxide, rinse the addition funnel with approximately 5 - 10 ml of di ionized water using a wash bottle. Add the rinse water to the reaction.

  Remember to be careful.



Picture 2

Picture 3

- ☐ The power to the J-Kem Controller has been turned on and pre-set to 102 °C, do not flip the switch on the left side.
- ☐ Turn the power dial from "OFF" to "300 mL—2L" for your position number. Note the start time here: _____

  While the reaction is heating, perform the following calculations in Table 1, but one team member must continue to monitor the reaction temperature. <u>Lab awareness must be constantly maintained</u>. Do not get distracted.
  - ☐ Record the weight of methyl salicylate used on line (**1**) of Table 1.
  - ☐ Calculate the amount of moles of methyl salicylate and record on line (**2**) in the Table 1. moles = g / Molar Mass
  - ☐ Calculate the theoretical yield of salicylic acid and record on line (**3**) in Table 1.

g of salicylic acid = moles of methyl salicylate × Molar Mass of salicylic acid

- When the reaction temperature reaches 96 °C. Note the time: _____
- Continue to heat the reaction for 20 minutes. Notice the droplets of water in lower part of the addition funnel. The water in the reaction is boiling and condensing, which is called refluxing.

  Instructor Note: The instructor may choose to have each lab pair run their reaction for different durations, for example 5, 10, 15 and 20 min to determine the impact time has on the yield. Also, this option allows slower groups to complete their reactions at the same time.
- After heating the reaction for 20 minutes, turn the power dial on the controller to "OFF." Do not touch the switch on the left side of the controller. Note the time: _____

Warning: the reaction vessel and heating mantle are HOT

- Lower the lab jack and remove the heating mantle (**HOT**). Place a crystallizing dish beneath the reaction flask. Raise the lab jack so that the bottom of the crystallizing dish is just below the bottom of the reaction flask.
- Fill the crystallizing dish only half way with room temperature water and continue stirring.
- Close the bottom stopcock of the addition funnel.
- Place the plastic funnel in the top opening of the addition funnel.
- To a clean 100-mL beaker add 36 mL of 3*M* aq HCl and transfer the acid to the addition funnel.
- When the reaction temperature is ~60°C, begin adding ~20 mL of the acid drop wise to the reaction solution over 2-4 minutes.

Warning: Acid addition to base is exothermic (produces heat). You may observe a temperature rise. Stop adding the acid if the temperature rises above 60°C

- Continue adding the remaining acid solution drop wise over 5 minutes while maintaining a temperature of less than 60°C.
  - What happened during the initial addition of acid? _____
  - What happened during this final addition of acid? _____
- When the addition of acid is complete, check the pH of the reaction mixture and ensure that the pH is < 2 using the pH paper supplied.
  - Remove the addition funnel from the reaction flask and dip the glass rod into the reaction mixture.
  - Touch the wet end of the glass rod to the pH paper.
  - If the pH is < 2, place the glass rod in the Hydrolysis Waste container and proceed to the next step
  - If the pH is >2, drop wise add 3*M* HCl from the 3*M* HCl dropping bottle and retest the pH. Continue until the pH is < 2 and proceed to the next step.

- Replace the addition funnel into the reaction flask.
- Add lots of ice to the water bath and cool the reaction mixture to 10°C.
- "Breakdown" the reaction by performing the following steps:
  - Using the lab jack, lower the stirrer and ice bath from the reaction flask.
  - Remove the additional funnel and carefully place it in the Hydrolysis Waste container.
  - Remove the thermocouple and lay it aside.
  - Turn off the J-Chem Controller for your position.
  - Carefully remove the reaction flask from the apparatus.
  - One member of the team should hold the reaction flask upright so that it does not tip and spill your product.
- Weigh a 60-mL sintered glass filter funnel and write the weight on the funnel with a Sharpie.

  Record the Mass of the funnel (g):_____
- Place a 60-mL sintered glass filter funnel onto a cone rubber adapter set on a 500 - 1000 mL filter flask that is securely clamped on a ring stand. Attach the side arm of the flask to a vacuum source and turn on the vacuum.
- Gently swirl the reaction flask, and then pour the contents of the reaction flask into the filter funnel.
- Using the chilled DI water squeeze bottle, rinse the reaction product off the sides of the reaction flask , swirl gently, and transfer any remaining solids to the filter. Repeat twice.
- Using a magnetic retriever, remove the stir bar from the reaction mixture, Wash the magnetic stir bar and filtrate with water two times each with approximately 20 mL of cold DI water.
  - What is being washed from the product? _____
- When almost no solution is dripping from the filter funnel, turn off the vacuum source.
- Weigh a 60-mL sintered glass filter (funnel + filtrate) and write the weight on the funnel with a Sharpie.

  Record the Mass of the funnel + filtrate (g):_____
- Calculate the Wet Cake Weight = Funnel + Filtrate(g) – Funnel (g) _____g
- Label a small crystallizing dish with your names. Weigh the labeled crystallizing dish and record its mass to the nearest 0.01 g. Tare mass: _____ g
- Using a spatula, carefully transfer approximately 0.25g to the weighed crystallizing dish.
- Weigh the crystallizing dish to the nearest 0.01 g. Wet mass:_____
- Place a Kimwipe and rubber band on top of the crystallizing dish to prevent contamination.
- Place the covered crystallizing dish into the vacuum oven and dry at 90°C under vacuum for 30 minutes.

- Turn off the vacuum and carefully remove the crystallizing dish from the oven.

Warning: the crystallizing dish will be very hot!

- Allow the crystallizing dish to cool to room temperature. Remove the Kimwipe and rubber band, and weigh the crystallizing dish to the nearest 0.01 g. Gross mass: _____ g
- Calculate the Dried Product Mass:
  - Weight Loss = Gross Mass (g)/ Wet mass (g) _____g
  - Dried Product Mass= Wet Cake Mass(g) X Weight Loss_____g
- Record the Dried Product Mass in Table 1 on line (**4**).
- After the solids have dried for 30 minutes, one team member is to proceed to the IR station. Bring your sample of methyl salicylate with you for identity assays. When finished with the IR, perform a Melting Point Determination.
- The other team member is to proceed to the Spectrophotometer station for the Fe(III)-Salicylic Acid Assay.

Analytical Procedures

IR (identity)

The infrared spectrum of each organic compound is unique to the types and number of bonds in the molecule. Obtain an IR spectrum for methyl salicylate and salicylic acid. Compare to known identified spectra.

Method:

Sample preparation:

The instructor reviews the following steps before the students begin their work.

Running a Background IR Spectrum

- Place one salt plate in the sample holder and lay the holder flat on the bench top.
- Place a second salt plate over the first.
- Place the cover over the plates and insert the holder into the IR spectrophotometer.
- Record the background spectrum.
  - Select **"Instrument"** from the toolbar
  - Click **"Scan Background"** from the dropdown menu
  - Enter the sample ID: "Background"
  - Enter the Description: "Air Background"
  - Click **"OK"** to begin scan

- Click "**Overwrite**"
- Select **"File"**
- Click **"Close"** from the drop down menu

Preparing a Sample of Methyl Salicylate

- Place one salt plate flat on the bench top.
- Add 1 drop of the Methyl Salicylate starting material on a salt plate.
- Carefully place the second salt plate on top.
- Carefully place the salt plate sandwich into the sample holder.
- Carefully place the cover over the plates and insert the holder into the IR spectrophotometer.
- Record the sample spectrum.
  - Select **"Instrument"** from the toolbar
  - Click **"Scan Sample"** from the drop down menu
  - Enter the sample ID: MS
  - Enter Description: "Team 1, 2, 3 or 4"
  - Click **"OK"** to begin scan
  - Click **"Overwrite"**
  - Select **"View"**
  - Click **"Label Peaks"** from the drop down menu
  - Print a copy of the spectrum by clicking on the Instrument icon on the toolbar
  - Select **"File"**
  - Click **"Close"** from the drop down menu

- After obtaining the spectrum, disassemble the holder
- Place a few drops of isopropanol on each plate and wipe clean with a new Kimwipe.

Preparing a Sample of Salicylic Acid in Acetone

- Using the spatula, transfer a very small amount of your sample to the agate mortar. Note each spatula is marked with line to provide guidance on the amount of sample required.
- Grind the sample to a very fine powder using the agate pestle.
- Add 4 drops of Acetone using micro pipette to dissolve the sample.

- Place one salt plate flat on the bench top.
- Place one drop of the Acetone / sample solution on the face of the salt plate.
- Allow to evaporate and the sample to crystallize.
- Carefully place the salt plate sandwich into the sample holder.
- Carefully place the cover over the plates and insert the holder into the IR spectrophotometer.
- Record the sample spectrum.
    - Select **"Instrument"** from the toolbar
    - Click **"Scan Sample"** from the drop down menu
    - Enter the sample ID: SA
    - Enter Description: "Team 1, 2, 3 or 4"
    - Click **"OK"** to begin scan
    - Click **"Overwrite"**
    - Select **"View"**
    - Click **"Label Peaks"** from the drop down menu
    - Print a copy of the spectrum by clicking on the Instrument icon on the toolbar
    - Select **"File"**
    - Click **"Close"** from the drop down menu

- After obtaining the spectrum, disassemble the holder
- Place a few drops of isopropanol on each plate and wipe clean with a new Kimwipe.
- Replace salt plates into their container and dispose of used Kimwipes and micro pipette

Assistant Instructions

- Instruct students on operating the spectrometer for each spectrum
    - Select instrument from the menu
    - Select scan background or scan sample
    - Enter the sample ID: background, Methyl salicylate or Salicylic Acid ID
    - Wavelength range is set for 4000 – 600 cm^{-1}
    - Click OK to begin scan
    - Select View and Label Peaks from the menu
    - Print a copy of each spectrum for each student

- Instruct students on basic operation of double beam IR spectrometer using the schematic over each instrument. FTIR spectrometer is too complex to describe.

- For background spectrum, ask students for the gases in air that could absorb IR light. Point out carbon dioxide as a green house gas and hence a strong global warming gas. Explain that computer saves the background spectrum and subtracts it from each of the sample spectra.

- Point out the loss of background peaks in the Acetone spectrum. Note Acetone is scanned as a sample spectrum and not as background.

- Remind students that IR spectra reveal the type of bonds present in a molecule. Point out some bond type absorptions.

Reviewing the Sample IR Spectrum

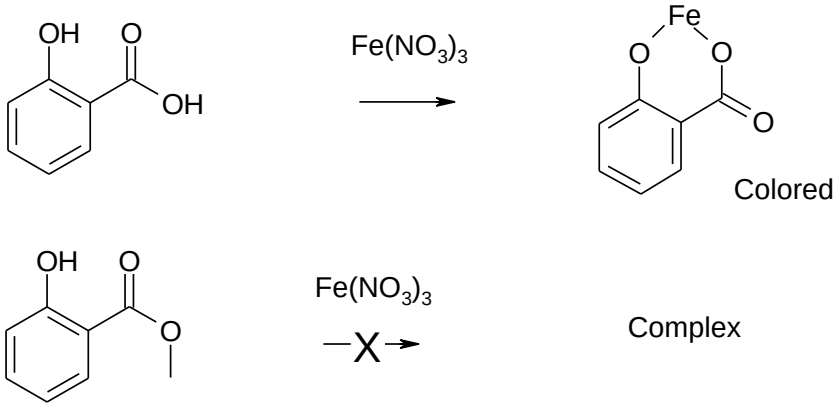
Look at the IR absorbance bands which are present in the range of 2500 cm-1 to 3500 cm-1. Identify differences between the methyl salicylate and the unknown.

Methyl Salicylate: What is the absorbance of the ester carbonyl:_____

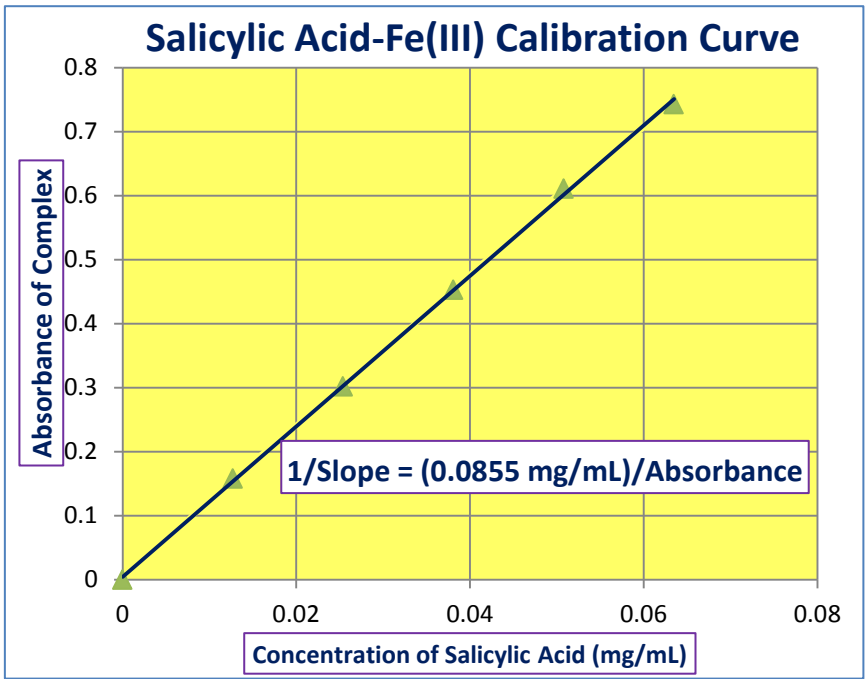
Salicylic Acid: What is the absorbance of the carboxylic acid:_____

Spectrophotometric Fe^{+3} Assays

Salicylic Acid is an *ortho*-substituted carboxylic acid phenolic aromatic ring system. These ring systems can uniquely bind to iron(III) salts which are purplish in color and absorb visible light in a range with a maximum at 520 nm. Methyl salicylate does not bind with Fe(III), remains colorless, and does not absorb light at 520 nm.



This allows a quantitative measurement to be made of solutions of salicylic acid in water. Known samples of the iron complex were prepared at various concentrations and the amount of light absorption was measured at each concentration. A graph was prepared of absorbance (y-axis) vs. concentration (x-axis). If a sample of salicylic acid is weighed and the absorbance measured, then the difference between the expected absorbance and the observed absorbance can be used to determine the amount, or purity, in the sample.



Spectrophotometric purity and quantitative analysis

Method:

Sample preparation:

- ☐ Weigh 25-35 mg (to 3 significant figures [e.g., 33.2 mg]) of the isolated salicylic acid into a 200 mL volumetric flask. Record the mass of salicylic acid _____.
- ☐ Fill the volumetric flask to the 200 mL mark with DI water and shake to dissolve the salicylic acid.
- ☐ Using a bottle top dispenser, measure 5.0 mL of the 0.02*M* $Fe(NO_3)_3$ solution into a 10 mL volumetric flask.
- ☐ Dilute the solution to the 10 mL line with salicylic acid solution. The clear solution will turn purple when the yellow Fe(III) solution is added.
- ☐ Calculate the theoretical concentration = mass of salicylic acid (mg) / (2 × 200 mL) (Why is the factor "2" in the denominator?)
- ☐ Record the theoretical concentration _____.
- ☐ Run the spectrometer blank using the Fe(III) solution in both cells.

- Measure the absorbance (peak height) at the absorbance maximum (~520 nm) and calculate the actual concentration of salicylic acid and the product purity.

Actual concentration = 0.0855 (mg/mL) × Absorbance _____

Purity (%) (Fe^{+3} assay) = actual concentration /theoretical concentration ×100%

_____

Record the Purity (%) (Fe^{+3} assay) (**5**) in Table 1.

MP (identity and purity)

Each crystalline organic compound has a unique melting point related to the stability of its crystal lattice energy. Measure the melting point of each dried crystalline solid that was obtained and record on line (**9**) in Table 1. Compare to the Theoretical melting point for pure salicylic acid listed in line (**10**) in Table 1

Method:

- Dip the open end of a melting point capillary into the pile of crystals.
- Invert and tap the tube on the desk to collect the crystals at the bottom.

Instructor Note: Use long glass tubes if the samples don't go to the bottom of the capillaries. Hold the capillary at the top of the tube and drop causing the capillary to bounce up and down. Slide the glass tube up, remove the capillary and inspect the sample. Repeat as needed.

- Place the capillary into the melting point apparatus.
- Turn the unit on and watch the sample through the sight glass.
- Record the temperature when the sample begins to melt and when fully melted
- Record melting point range on line (**9**) in Table 1. Compare to the Theoretical melting point for pure salicylic acid listed in line (**10**) in Table 1

Calculations:

Upon completion of assays, complete Table 1.

- Actual Yield (**6**) = Dried product mass (**4**) × Purity % (Fe^{+3} assay) (**5**) / 100%
- Amount of salicylic acid (**7**) = Actual Yield (**6**) / Molar Mass (**0**)
- Hydrolysis % Yield (**8**) = Actual Yield (**6**) / Theoretical Yield (**3**) × 100%

Table 1

	Starting Material/ Product:	Methyl Salicylate	Salicylic Acid
	Molar Mass (g/mole)	152.15	138.12
1	Mass of methyl salicylate (g)		
2	Amount of methyl salicylate (moles)		
3	Theoretical yield of salicylic acid (g)		
4	Dried product mass (g)		
5	Purity % (Fe^{+3} assay)		
6	Actual Yield of salicylic acid (g)		
7	Amount of salicylic acid (moles)		
8	Hydrolysis % Yield		
9	Melting Point observed (°C)		
10	Theoretical Melting Point (°C)		159°C

Conclusions and Evaluation

1. Summarize the results in the tables.
2. Pool your results on the hydrolysis and acetylation steps and prepare a short presentation on your evaluation of the hydrolysis of methyl salicylate to salicylic acid.
3. How might you evaluate the success of the hydrolysis of methyl salicylate to salicylic acid?
4. What was the reproducibility of the reaction yields?
5. What other assays might be of value in determining the quality of the products?
6. What recommendations does the synthesis team have to make?

S2S Team Report

Step 1. Hydrolysis

Methyl salicylate identification (yes or no)

Tests	Team 1	Team 2	Team 3	Team 4
IR conformity (Y/N)				

Step 1 Salicylic Acid Analyses

Tests	Team 1	Team 2	Team 3	Team 4
MP (159 lit)				
IR conformity (Y/N)				
Spectrophotometric Weight % Assay				
% Yield				

Salicylic Acid Synthesis – Set up

Supplies and Equipment

Lab station Set-up -Step 1 Hydrolysis of methyl salicylate

- Magnetic stirrer controller (4)
- J-Kem reaction controller (2 Apollo models) and thermocouples assembly (4)
- Heating mantles (4 x 250 mL size)
- Rubber septum (4 x 24/40)
- Ring Stands (8) with assorted clamps
- 250 mL reaction flask 4 x 250 mL 2 or 3 neck)
- 50 - 125 ml pressure equalizing addition funnels (4)
- Stir bar (8 x 1.5 in football shaped)
- 250 ml Cork rings (4)
- Small Plastic Funnel with a short stem (4)
- Glass rods- 12” (4)
- 0-50 ml bottle top dispensers (4)
- Dispenser bottles (1- 2 L size (4, for 6N NaOH, 3N HCl, 2 -DI water)
- Assorted beakers;
  - 2 x 50 Labeled pH paper
  - 4 x 100 Labeled 3M HCl
  - 2 x 250 Labeled Stir Bars
  - 4 x 800 Labeled Ice
  - 4 x 1000 ml Labeled Water
- Small Lab Jacks (4)
- Large Lab Jacks (2)
- pH paper (0-2.5 range)
- Crystallizing dish for cooling bath 150x75 (4)

- Crystallizing dish for drying 70x50 (4)
- Calculator (4)
- Stopwatches (4)
- Sharpie Markers (4)
- Tissues (4)
- Rubber bands (4)
- Clip board with pen (4)
- Balances 200 - 1000 g d=.01 (2)

Filtering

- Sintered glass funnel (4 x 60 mL medium or coarse)
- Filter flask (4 x 500 - 1000 mL)
- Neoprene cone adapter for funnel/filter flask (8)
- Vacuum system (4 x aspirator vacuum system)
- Vacuum oven (1)
- Vacuum pump for oven (1)
- Vacuum tubing for filter funnels and vacuum oven

FTIR

- NaCl salt plates (2)
- Salt plate holders (2)
- Hyman Spatula (2)
- Agate Mortar and pestle (2)
- Acetone in a Dropping Bottle
- Micro pipettes
- Isopropanol in a wash bottle for cleaning
- Kimwipes
- Infrared spectrometer – 2 units

UV / Vis

- Volumetric Flask Class A (6 x 200ml)
- Volumetric Flask Class A (4 x 10ml)
- 0-10 ml Bottle top dispenser
- Disposable pipettes (3ml)
- Hyman Spatulas (2)
- Plastic Beakers (4 x 250ml)
- Plastic Beaker (1 x 500ml)
- Glass Beakers (1 x 500ml)
- Weigh paper
- Analytical Balance capable of weighing to +/- .0001 g (2)

Melting Point

- Melting Point Apparatus (2)
- Melting Point Capillary tubes

- Ring Stand and clamp
- Glass tube

Synthesis Notes - Step 1 Hydrolysis of methyl salicylate

- Turn on vacuum oven, under vacuum, before the class starts to heat up to 90.
- Fill Ice buckets for cooling the reaction flask, chill 4 Di Water bottles and the vacuum trap
- Set the J-Kem Controller to 105 °C, and turn the unit on using the switch on the left side.
- Connect the electric cord from the heating mantle and temperature probe to the correct position number on the controller (1, 2, 3, or 4).
- Arrange the students into pairs.
- Give a brief introduction outlining the two step synthesis of Aspirin

- Student should check each step as they complete the step
- Student 1 starts the hydrolysis experiment assisted by student 2 who reads the instructions and checks completion.
- While the reaction is heating up the Instructor should explain the reaction mechanism and the phases changes that will occur. Also, Student 2 should proceed to the IR station to run the IR of methyl salicylate.
- When the reaction temperature reaches 96 °C. The procedure instructs the students to Continue to heat the reaction for 20 minutes. The instructor may use this opportunity to adjust the reaction times so all of the students finish at the same time. Explain that we are investigating the impact of time on the yield and purity.
- Students should complete the reaction, filtration and have samples in the oven to dry in 2 hours. The samples should remain in the oven during a 30 minute lunch.
- When the dried salicylic acid is taken from the oven, Student 1 weighs the product, prepares and begins to run the Spectrophotometric-Fe(III) assay. Student 2 runs an IR and MP of isolated SA.
- Any student with free time should begin to compile data on the salicylic acid, seek his partner and exchange information.
- Teams prepare final report and team discussion with instructors/team leaders, comparing yield, IR, MP, and Spectrophotometer data. They should assess the quality of the tech transfer package (purity, yields, ID). Make recommendations-does further work need to be performed, gap analysis, should other assays be performed, or is the process ready to test scale-up on 1 kilogram scale?

Reagents and Preparation

Methyl salicylate
Ferric nitrate
Di ionized water
Acetone for Cleaning
Isopropyl alcohol
Ice (2 bags)
10 M NaOH
Concentrated HCl

Reagent Preparation – Step 1 Hydrolysis of Methy Salicylate

6 M NaOH
Add 600ml of 10 M NaOH to a clean dry 1000m ml graduated cylinder. Carefully, dilute to 1000ml wil di ionized water.

3 N HCl
Add 750 ml of di ionized water to a clean dry 1000m ml graduated cylinder. Carefully, add 250 ml of concentrated HCl.

0.02*M* $Fe(NO_3)_3$
Add 1.616g of $Fe(NO_3)_3$ -$9H_2O$ to a 200ml volumetric flask and dilute to volume with DI water.

Note: 200ml gets is adequate for 4 days

Clean –up

Reaction Vessel:

Rinse the addition funnel, plastic funnel, ice beaker, crystallizing dish, NaOH beaker and HCl beaker with di-ionized water

Rinse the reaction vessel, stirring bar, spatulas, temperature probe, filtering flask and weighing dishes with “acetone for cleaning”.

Allow to dry and reassemble the reaction vessel

Miscellaneous:

Empty ice from the ice coolers and vacuum traps

Empty vacuum traps and rinse with water