Purpose

In this lab, you will perform several tests to attempt to confirm the identity and assess the purity of the substance you synthesized in last week's lab.

Equipment

- Melting point apparatus
- Melting point capillary tubes
- Nicolet IR-100 infrared spectrometer
- Teflon tape
- Plastic IR card
- Cellophane tape
- Test tubes (as needed)
- Drying oven

Chemicals required

- Deionized water
- 3M NaOH
- 3M HCl
- Saturated $NaHCO_3$ solution
- Acetone
- Methyl salicylate
- Salicylic acid

Procedure

***Due to the limited number of instruments available, you may need to rearrange the order you do these tests!**

Physical appearance

Record the appearance, phase, and odor of your compound into your laboratory notebook. For comparison, record the same information about the starting material (methyl salicylate) used to prepare your compound and a known sample of salicylic acid.

Solubility tests

Place about five milliliters of deionized water, dilute (3M) NaOH, dilute (3M) HCl, and saturated $NaHCO_3$ solution into separate clean, dry test tubes. Add a small scoop of your compound to each test tube. Shake, and record your observations for each test tube into your laboratory notebook. Also note whether or not any gas is evolved. For comparison, record the same information about known samples of salicylic acid and methyl salicylate. (Use 20 drops of methyl salicylate in each tube instead of a small scoop. Make sure to use clean, dry test tubes for each test.)

Melting point analysis

Your instructor will briefly demonstrate the use of the melting point apparatus.

Place a few grains of your compound into a melting point capillary. If your compound has large crystals, you may need to crush it with a mortar and pestle before you are able to put it into the capillary.

Record the temperature as soon as the compound **begins** to melt. Turn the melting point apparatus off and **allow it to cool well below the temperature at which your compound melted** (but NOT all the way back to room temperature). Repeat this procedure for a total of three melting point measurements. Calculate the average melting temperature, and compare this temperature to the melting point for salicylic acid found in the CRC Handbook of Chemistry and Physics. A copy of the CRC Handbook is available in the laboratory. **Make sure to record the CRC value before you leave the lab**.

Dispose of the the used melting point capillary tubes i**n the broken glass container**.

Infrared (IR) spectroscopy

Your instructor will briefly demonstrate the use of the Nicolet IR-100 infrared spectrometer.

In a small test tube, dissolve a small scoop of your compound in approximately half of a milliliter of acetone.

Prepare your IR card sample holder by cutting an inch-long section of teflon tape. Using some cellophane tape, tape the teflon tape just under the bottom of the opening in the IR card.

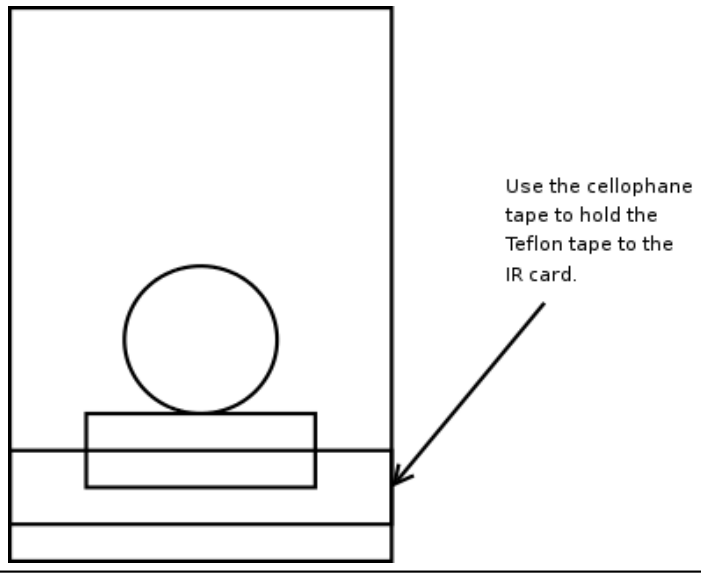


Illustration 1 - Attaching teflon tape to the IR card.

Using another piece of cellophane tape, stretch the teflon tape over the opening in the IR card. Secure the cellophane tape, and the IR card is ready for use.

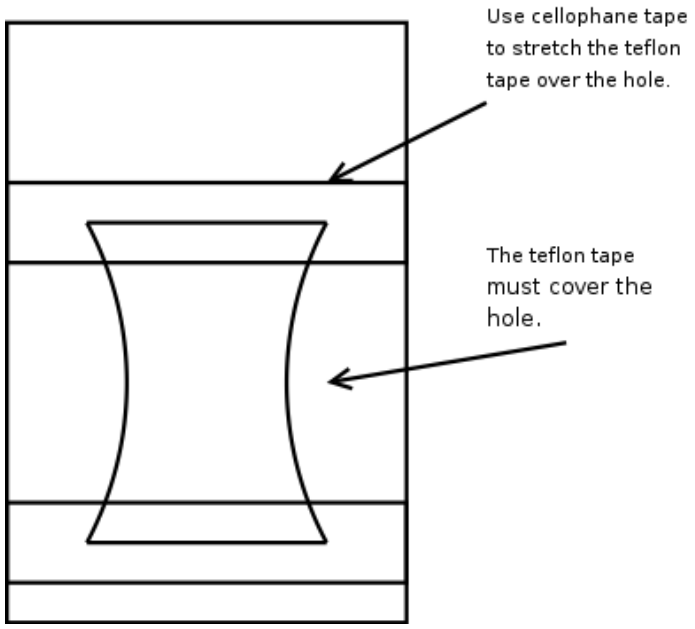


Illustration 2 - Using another piece of tape to stretch the teflon.

Put the IR card into the sample slot of the spectrometer with the teflon side facing left.

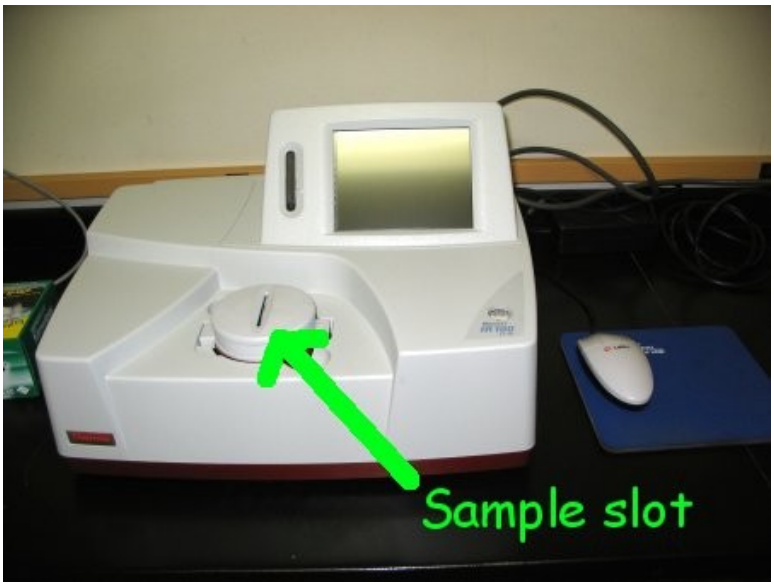


Illustration 3 - Sample slot of the Nicolet IR-100 spectrometer

On the spectrometer, click "Collect". A submenu will pop up. Click "Background". (Most of the menus on the Nicolet IR-100 Spectrometer work this way.)

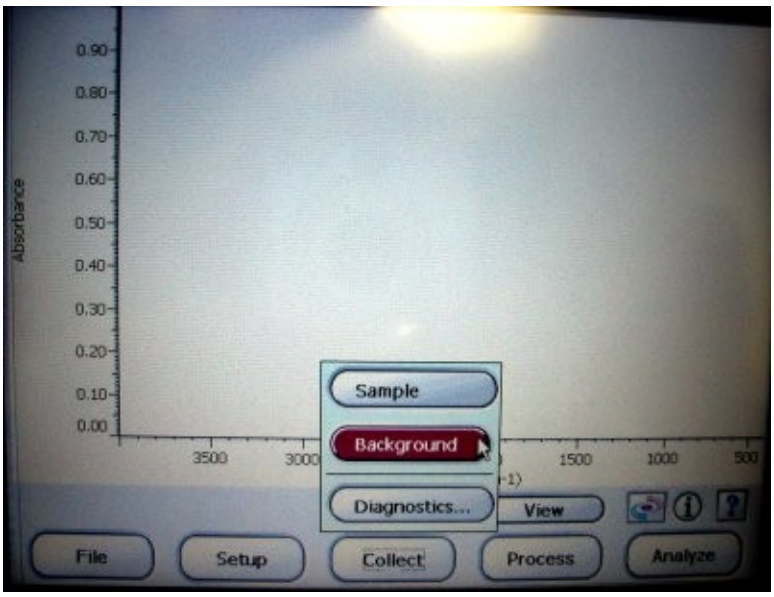


Illustration 4 - Menus of the Nicolet IR-100 spectrometer

When the spectrometer asks you to name this spectrum, just click enter on the on-screen keyboard to continue. (The background spectrum will not be displayed or printed, so you do not need to name it.) After the spectrometer collects a background scan, remove the IR card. Using a dropper, put about four drops of the solution of your sample in acetone onto the teflon. The teflon should change colors from white to clear when coated with the sample solution. Put the IR card on a paper towel in the drying oven (60 ^{o}C) for one minute to dry the acetone. When dry, the teflon will again be white.

Put the IR card into the sample slot of the spectrometer again with the teflon side facing left. On the spectrometer, click the "Collect" button, then click "Sample". **If the instrument give an error at this point, then you have too much sample on your IR card, and you will need to start again after diluting your sample solution with more acetone.**

Name the spectrum using the on-screen keyboard. Use a name like "Synthesized salicylic acid on teflon". When the scan is complete, it will be displayed on the screen. Click "Process", then click "Correct Baseline" to clean up the spectrum. If you would like to label peaks, you may use the "Annotate" function. To print the spectrum, click "File", then "Print".

If you need to rescan a spectrum, you may delete the old spectrum using the delete command found in the "View" menu. Otherwise, the instrument will display the next spectrum on top of your old one.

When you are finished with the instrument, delete all your spectra using the delete command found in the "View" menu.

Remove the IR card from the spectrometer, then remove the teflon tape and cellophane tape from the IR card. Dispose of the cellophane tape and teflon tape in the trash can, then leave the IR card by the spectrometer for the next group to use.

Discussion

Physical appearance

Describe the physical appearance and odor of your compound Is it similar to salicylic acid? How do the physical appearance and odor of your compound compare with that of methyl salicylate?

Solubility tests

Do the solubility tests show that your compound is an acid? In particular, consider the results of the base sodium hydroxide and the salt sodium hydrogen carbonate (a bicarbonate, which should react with acids to produce carbon dioxide gas). How are the results for your compound similar to known salicylic acid and different from methyl salicylate?

Melting point analysis

Does the melting point of your compound agree with the established melting point of salicylic acid? In general ,the more impure a compound is, the lower its melting point (this is an effect called freezing point depression which we will study in CHM 111). Does the melting point you measured indicate anything about the purity of your compound?

Infrared spectroscopy

Infrared spectroscopy works in a manner similar to the ultraviolet/visible spectroscopy (UV/VIS) that you have performed in previous laboratories. An important difference, however, is what kind of information is returned. Infrared light interacts with the **chemical bonds** in many compounds, causing them to vibrate. The wavelengths of infrared light that are absorbed by a sample can be used to show the kinds of bonds present in the compound.

Salicylic acid is a carboxylic acid. Carboxylic acids contain the following arrangement of atoms.

Illustration 5 - Carboxylic acid group

In the illustration, "R" is another carbon-containing part of the molecule. For acetic acid, R is CH_3. For salicylic acid, R consists of a benzene ring with a hydroxyl (OH) group attached..

The carboxylic acid group gives rise to three peaks in an infrared spectrum. One is the "carbonyl" (C=O) bond, another is the C-O single bond, and the third is the O-H bond.

Bond	**Wavenumber (cm^{-1})**
C=O	1660-1820 (probably near 1670 for salicylic acid)
C-O	1000-1300 (probably near 1300 for salicylic acid)
O-H	2400-3400 (very broad - may overlap other peaks)

The most obvious peaks in your spectrum, assuming your compound is a carboxylic acid, will probably be the O-H and C=O peaks (with the O-H being the most obvious feature). Other peaks you might be able to identify in your spectrum are listed below.

Bond	**Wavenumber (cm^{-1})**
C-H	around 3000 (There may be several peaks here, depending on how the carbon atoms are bonded.)
aromatic carbon-carbon bond	multiple peaks at 1600 and 1475 (Salicylic acid contains a benzene ring.)

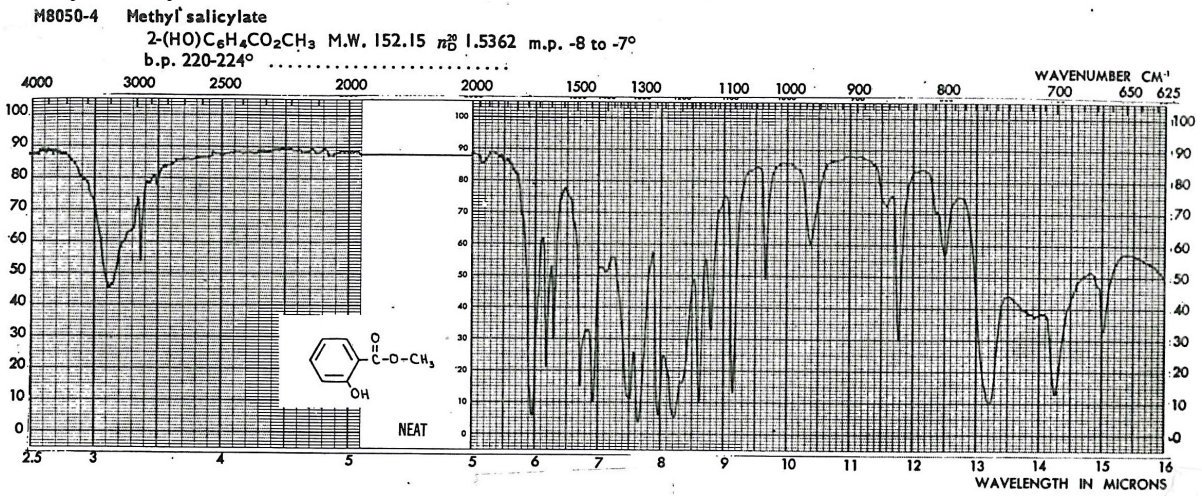
Try to identify peaks in your spectrum that are consistent with salicylic acid - in particular the carboxylic acid peaks. Compare the spectrum you produced with a known spectrum for salicylic acid found in the lab's library of infrared spectra. Do you see similar features? Also compare your spectrum with the library spectrum of methyl salicylate and observe any differences.

The infrared spectrum **may vary** slightly depending on the technique used to prepare the sample. Since the spectrum in the lab's library of spectra were acquired using a different sample preparation technique that you used, your spectrum may differ slightly in the location and (in particular) height of peaks.

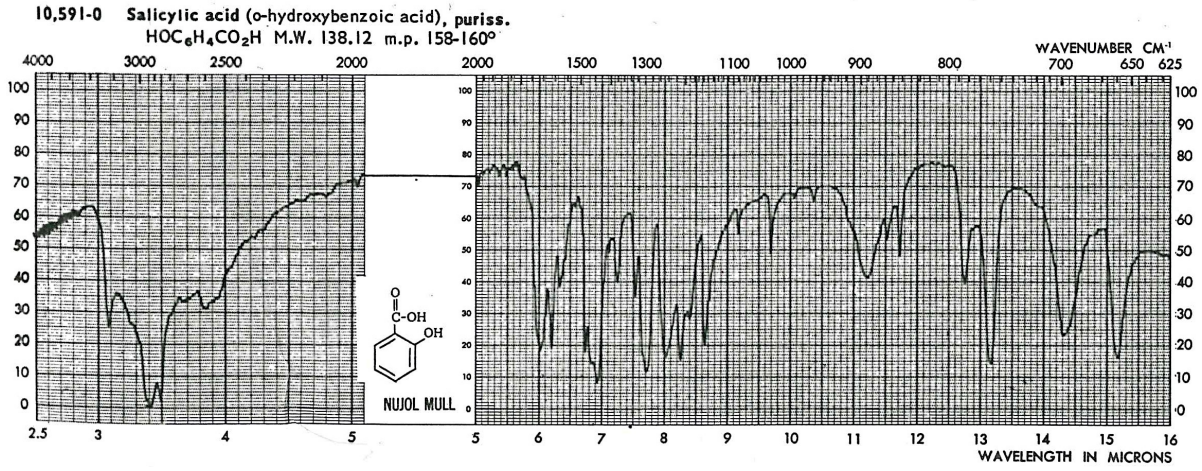
Is the information you get from the spectrum consistent with the other information you have obtained so far?

Sample IR Spectra for Methyl Salicylate and Salicylic Acid

Methyl Salicylate



Salicylic Acid



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Group members

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Data tables

Physical characteristics

Physical appearance ...	
Synthesized compound	
Methyl salicylate	
Known sample of salicylic acid	
Phase ...	
Synthesized compound	
Methyl salicylate	
Known sample of salicylic acid	
Odor ...	
Synthesized compound	
Methyl salicylate	
Known sample of salicylic acid	

Solubility

Compound	*Soluble in deionized water?*	*Soluble in 3M NaOH?*	*Soluble in 3M HCl?*	*Soluble in saturated $NaHCO_3$?*
Synthesized compound				
Methyl salicylate				
Known sample of salicylic acid				

Melting point

Melting point of synthesized compound – first trial (°C)	
Melting point of synthesized compound – second trial (°C)	
Melting point of synthesized compound – third trial (°C)	
Literature value of melting point of salicylic acid – from *CRC Handbook of Chemistry and Physics* (°C)	

Note: Staple your IR spectrum from the instrument to these report pages.

Questions

1) Are the appearance and odor of your synthesized compound more similar to the methyl salicylate starting material or to the sample of salicylic acid provided?

2) Which compound – methyl salicylate or salicylic acid – had solubility characteristics more similar to your synthesized compound? Also, describe any differences between the solubility synthesized compound and the compound it's most similar to (if any).

3) If acidic compounds tend to be soluble in bases and basic compounds tend to be soluble in acids, is your synthesized compound an acid or a base? Why?

4) What is the average melting point of your synthesized compound? Is it consistent with the literature value for salicylic acid?

5) As the purity of a compound decreases, the melting point tends to decrease. What do you think about the purity of your synthesized compound? Explain your reasoning.

6) Compare your IR spectrum to the provided spectra for methyl salicylate and salicylic acid. Which of the provided spectra is more similar to yours?

7) Label the following peaks on your IR spectrum: C=O, C-O, O-H.