

Synthesis and Analysis of Acetyl Salicylic Acid

Amy Chan

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Introduction

Acetyl salicylic acid, commonly known as aspirin, is one of the most common synthetic medicines. First discovered in 1897 by Felix Hoffman, it has since been used in over 50 over-the-counter medicines. Aspirin is primarily used to treat pain as well as to avert cardiovascular disease. The purpose of this lab is to synthesize and characterize high purity aspirin.

The aspirin synthesized in this experiment will be crystallized from solution. Crystallization will be induced by scratching the glass beaker to create nuclei around which aspirin may crystallize. The solution will be formed by combining salicylic acid with acetic anhydride. Aspirin and acetic acid are formed when the hydrogen in the phenolic alcohol group of salicylic acid is replaced by an acetyl (H_3CCOO) group. Phosphoric acid is added to catalyze the reaction. The synthesis reaction is summarized below:

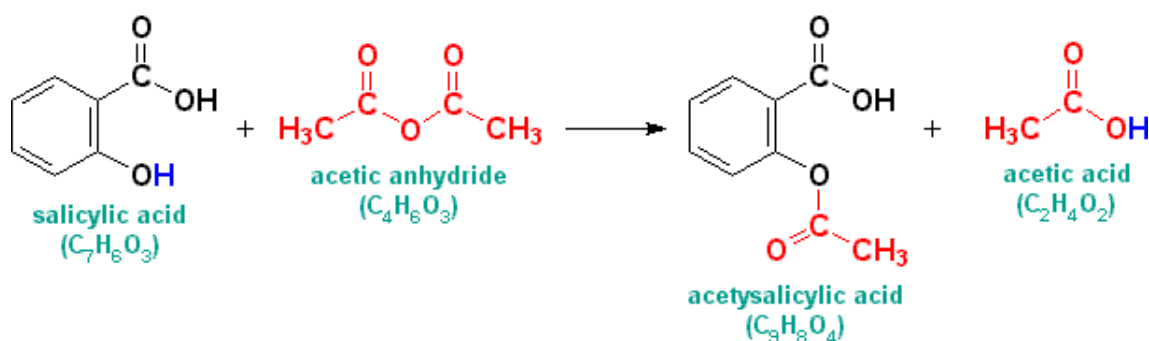


Figure 1 Synthesis Reaction for Aspirin¹

After the crystals are formed, the percent yield will be calculated before the analysis is done.

In order to determine the purity of the aspirin, it must be characterized through various techniques based on an understanding of the energy of the system on the microscopic and atomic scale. The aspirin will be characterized by three methods: melting point analysis, Fourier transform infrared spectroscopy (FTIR), and Fourier transform nuclear magnetic resonance spectroscopy (FTNMR).

¹ Source: Webpage - <http://www.chemheritage.org/EducationalServices/pharm/tg/asp/asp31.htm#major>

Experimental

Synthesis of Aspirin

In the first part of the experiment, aspirin was synthesized. A filter crucible and mortar and pestle were obtained from the instructor. Next, 3.00 g of salicylic acid were weighed into a 100 mL beaker, which was then covered with a watch glass. This beaker, 2 graduated cylinders, a distilled water wash bottle, medicine dropper, and another beaker of distilled water were brought to the hood. The reaction took place after 9 mL of acetic anhydride and 15 drops of phosphoric acid were added to the reaction beaker under the hood. The hood was used as a safety precaution because of the dangers of using acetic anhydride. The solution was gently heated over a Bunsen burner until it was almost boiling. After removing the heat, the solution was left to cool for three minutes before the slow addition of 45 drops of distilled water from the extra beaker of distilled water using the medicine dropper. The mixture was cooled for several minutes before 30 mL of distilled water were added to the solution. The beaker was swirled to mix the solution well before it was left to cool again. During the time of cooling, an ice water bath was prepared in a 400 mL beaker. The mixture was left to cool until the beaker was comfortable to touch before it was carried back to the lab bench.

At the lab bench, the aspirin crystallized out of the solution. Crystallization was induced by scratching the beaker with a metal scoopula. The glass crystals became the nuclei around which aspirin could crystallize. The beaker was placed in an ice water bath immediately after the scratching for a total time of fifteen minutes. After the first five minutes, the beaker was gently swirled for several seconds. During this time, 60 mL of distilled water were chilled in a separate ice bath. When the crystallization was complete, the aspirin crystals needed to be filtered and washed.

The crystals were filtered and washed to purify the yield. First, the crystals were poured into the filter crucible. Next, the liquid was pressed out of the crystals with a glass stopper on top of a piece of filter paper. The crystals were then washed three times with the chilled distilled water. The filter crucible was removed from the aspirator and about 20 mL of the chilled water was added. The mixture was stirred for several seconds before the filter crucible was placed over the aspirator. Stirring continued while the liquid solution was being filtered again. These washing steps had to be performed quickly to minimize the loss of aspirin due to its solubility in water. After the three washings, it was observed that one could not smell acetic acid in the crystals. Thus, the crystals were ready to be dried. This was accomplished by drying the undisturbed crystals with the aspirator on for ten minutes. Then,

the crystals were lightly stirred in the filter crucible for an addition five minutes with the aspirator on. Lastly, the crystals were spread evenly on a labeled watch glass before they were placed in an oven for thirty minutes.

The final step of this stage of the experiment was to determine the percent yield of the aspirin. While the crystals were in the oven, a clean, dry vial was appropriately weighed on a top load balance, and the mass was recorded. When the crystals were finished drying in the oven, they were removed and ground to a fine powder in the mortar and pestle before being poured into the labeled vial. The vial with the aspirin was weighed and the mass was recorded. This vial was then stored in the desiccator until the characterization began.

Melting Point Characterization

The melting point is used to ascertain the temperature at which the kinetic and potential energy of the molecules are equal. However, since melting does not occur at a single temperature, a melting point range is found. This melting range represents the range where the solid aspirin melts into a liquid. In the solid phase, the aspirin is packed to obtain the lowest energy configuration in the lattice. At the melting range, a sufficient amount of energy is added to overcome the hydrogen bonds, dipolar-, electrostatic-, and van der Waals forces that exist in the lattice. These forces also are present in the liquid state, but they are weaker. Purity will be determined based on the values of the melting range: less pure substances will have a lower melting point and a larger melting range.

The melting point range was determined using a Thomas-Hoover apparatus. First, the samples were packed into the melting point tube. This was accomplished by pressing the open end of the tube into the sample until the sample was about 2 mm into the tube. The sample was then packed by dropping the tube in a condenser tube, with its closed end down, which minimized the chance of the melting point tube breaking. This packing technique was important because melting point analysis requires the smallest size sample that can still be visible during melting. Large samples or unevenly packed samples will heat unevenly to give erroneous results. Three tubes were prepared.

The Thomas-Hoover apparatus was turned on after the voltage was turned to zero. The capillary tube was carefully inserted into the capillary tube openings. The stirrer knob was turned so that the mark was about halfway between the slow and the fast settings. Knowing that the melting range was near 130°C, the voltage knob was turned to 3.4, which gave a temperature rate of about 5°C/minute, until 120°C was reached on the thermometer. At this point, the voltage setting was lowered to 3.0. The sample was viewed through a magnifying glass, and when the

sample first started to melt, the temperature was recorded. The final temperature was recorded when the last sample melted. The melted sample was then discarded. This process was repeated three times, and the average was taken to determine the melting point range.

FTIR Characterization

Fourier transform infrared spectroscopy is based on the response of atoms to exposure to infrared rays (IR), which are a part of the electromagnetic spectrum. Energy propagates by electric and magnetic fields, which are orthogonal to each other. The bonds between two atoms act as springs, and these bonds vibrate. The number of different vibrational modes is instrumental in FTIR and is equal to $3N - 6$. Some different modes include the bend, stretch, and scissor modes. These vibrational modes are unique to every molecule. Thus, the FTIR spectrum is sometimes referred to as the “fingerprint” of the molecule.

In the first part, FTIR spectra were collected for three different powders: aspirin, benzoic acid, and sodium salicylate. The MIDAC spectrometer was used. It was first necessary to prepare the sample that was placed in the apparatus. For each powder, approximately 10 mg was massed before it was ground to a fine powder using the mortar and pestle. One drop of Nujol was added and the resulting suspension was thoroughly mixed using the pestle until it was homogenous. The viscous fluid was gathered using the “policeman”, a rubbery scoop, before being placed in the center of the sodium chloride (NaCl) plates. When handling the NaCl plates, special care was taken to not touch the surface with the fingers. This was done because NaCl plates are very hygroscopic, and water absorbed into the plates will appear in the spectrum as noise. The two plates were placed together to create a “sandwich”, which is shown in figure 2, and rotated until the suspension was a homogeneous thin film between the plates.

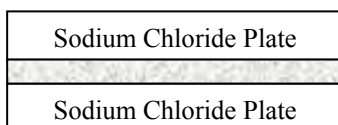


Figure 2 "Sandwich" where gray area is the powder suspended in Nujol

The apparatus was operated in the same manner to obtain all spectra using the following steps. First, it was necessary to take a background spectrum, which compensated for the effect of air in the instrument. To obtain a background spectrum, “Background” was selected in the “Collect” menu. The “Collect” button was selected in the

new window after appropriate data was entered. The background spectrum was then displayed on the chart. Next, the sample was placed in the V-shaped sample holder and “Sample” was selected in the “Collect” menu. The student’s name and sample ID were entered in the window that appeared and the “Collect” button was selected. After the spectrum appeared, “View Data Points” was selected in the toolbar, which showed the wavenumber location of all the peaks. The spectrum of all the powder samples, aspirin, benzoic acid, and sodium salicylate, were collected using these steps. After each scan was taken, it was necessary to wash the plates, mortar and pestle, and “policeman” with ethyl alcohol. These items were then dried before proceeding with the next sample. In particular, the NaCl plates were placed in the desiccating bottle to minimize water absorption. After the powders were analyzed, the polystyrene film was analyzed.

In the second part of the analysis, it was necessary to determine the spectrum of polystyrene film. The film was in a drawer below the apparatus, and no sample preparation was required. The spectrum was obtained using the same directions as described in the above paragraph, but the polystyrene film replaced the sample holder. First, a background scan was obtained. Then, the sample holder was removed and the film was inserted into the grooves of the holder. The sample was scanned, the peaks were located, and the spectrum was printed.

FTNMR Characterization

Nuclear magnetic resonance is based on the property of the atomic nucleus creating a magnetic field because of its spin. However, this magnetic field is only observable in the presence of a magnetic field. A nucleus has ‘spin’ when it has an angular momentum, which is then associated with magnetic moments. The magnetic moments of the nucleus cause the nucleus to exhibit magnet-like behavior that can be observed when a constant magnetic field, \mathbf{B} , is applied. NMR depends on the direct frequency dependence of the magnetic field. If an electromagnetic frequency is added to the sample, the nuclei will have enough energy to flip their magnetic spins to the higher states from the lower states. Neighboring nuclei affect each other to produce a spin-spin coupling effect. The term equivalent refers to nuclei that are in the same chemical environment, while non-equivalent refers to nuclei in a different chemical environment. Thus, NMR may be used to determine the groups of the molecule.

First, it was necessary to prepare the NMR samples. Two 5 mm NMR sample tubes and two clean 15x45 mm, or 4 ml, vials and caps from the cart were obtained. The vials were labeled 0.3 and 1.0 molar. Pasteur pipettes were calibrated to 1 ml by using a 10 ml graduated cylinder filled with water. A water pipette was filled with 1 ml

of water, and the level was marked with a narrow label strip. Two dry pipettes, which were used later to measure the CDCl_3 and alcohol, were then marked at the same level. For the 0.3 M vial, 0.0543 g was weighed on the analytical of the synthesized aspirin and added. In addition, 1 ml of CDCl_3 was added to the 0.3 molar vial. In the 1.0 M vial, 0.1800 g of aspirin and 1 ml of absolute alcohol were added to the vial. The NMR tubes were filled to a column height of 0.039 m, and they were labeled on the plastic cap using a pen.

The 0.3 M sample tube was used for a proton NMR scan while the 1.0 M sample tube was used for a carbon NMR scan. At the EFT-90 NMR spectrometer, the PNMR program was opened. “nu H1” was entered into the command line above the lower toolbar. The parameters used, as well as their meanings, are shown in Table 1.

Table 1: NMR Parameters

Symbol	Parameter	Meaning	Proton	Carbon
SI	Size	Number of Points/Scan (Resolution)	16384	32768
NS	Number of Scans	More scans give less noise	8	64
DS	Dummy Scans		0	0
RG	Receiver Gain	If gain is too high, it's bad for instrument because of oversaturation. If gain is too low, it's hard to see signal.	100	100
PW	Pulse Width (us)	Duration or width in time	15	12
RD	Relaxation Delay (s)	Time for particle to go back to equilibrium	1	3
SW	Sweep Width (Hz)	Range of scan	1500	7500
SF	Spectrometer Frequency (MHz)	Larmor frequency	90.02	22.5
DF	Decoupler Frequency (MHz)	-	1	90.02
DP	Decoupler Power (db)	-	0	25
Total Acquisition Time (s)		Time to collect signal	5.4	-
Field Offset		Adjusted by TA	-	-

When the correct parameters were entered from the appropriate column, data acquisition was started by entering “zg”. After the scan was completed, the data was transferred to a disk for analysis in the lab.

In the lab, the NUTS program was opened. First, the spectrum was phased before the reference peak was set. A menu appeared on the screen and “0” ppm (H NMR) or “17.9” ppm (C-NMR) was entered for the reference value. Peaks were selected and labeled. It was also necessary to zoom into the aromatic regions of each sample. Consequently, a total of four spectra were obtained in this phase of the lab. One set of each NMR printout was given to the TA.

Results and Discussion

Yield Calculation

During the synthesis, many things were observed. First, as the water drops were added to the reaction solution, a relatively violent reaction occurred. Small “pops” were heard, and the solution bubbled. During the crystallization process, swirling greatly increased the rate of crystallization. The synthesis yield was calculated by the computer from the masses recorded in the experiment. These values are entered in Table 2:

Table 2: Synthesis of Aspirin Experimental Data

Substance	Mass (g)
Salicylic Acid	3.00
Empty Vial	22.52
Vial + Aspirin	25.04

The computer calculated that the actual yield was 2.52 g of aspirin. This is also equal to a percent yield of 64%.

Melting Point

The observed melting point range did include the expected melting point. The expected melting point, also calculated from an average, was 134°C². The four sets of melting point data are summarized in Table 3.

Table 3: Summary of Melting Point Results

Trial #	Temperature (°C)		Notes/Observations
	Initial Melting	Final Melting	
1	125	132	1st run ever
2	131	135	-
3	132	135	-
4	131	129.5	Used different apparatus
Average	131.3	133.17	Samples 2, 3, 4 Used

The results of the first sample were neglected because that was the first melting point trial performed. The experimenter doubted the validity of those values because, at the time, she was not sure what to observe with regards to the aspirin's first melting. The results for the fourth trial were very strange in that the temperature dropped after melting. Also, it was observed that it took a long time for the sample to melt. It is important to note that sample 4 was done after the IR analysis was complete. Because the apparatus used for the initial tests was being used by someone else, a different Thomas-Hoover machine was used for the final test. Hence, the different results could be due to the apparatus itself. If only trials 2 and 3 are used, then the average range would be 131.5 - 135°C, which

² Source: NIST web database:
<http://webbook.nist.gov/cgi/cbook.cgi?Name=acetylsalicylic+acid&Units=SI&cTP=on>

would be very precise results. The narrowness and precision of the range indicates high purity. In addition, the maximum temperature of melting, $T = 135^{\circ}\text{C}$, corresponds with the maximum temperature of the melting point range, $T = 135^{\circ}\text{C}^3$. Consequently, the melting range indicates that the synthesized aspirin is pure.

FTIR Characterization

FTIR were performed for four different substances: aspirin, benzoic acid, salicylic acid, and polystyrene. All of the spectra, with the various bands labeled, are in Appendix A of this report. Table 4 provides a summary of the different band assignments⁴.

Table 4a: IR Band Assignments of Salicylic Acid

Bonds		Stretching Wavelength (1/cm)	
Name	Notes	Theoretical	Experimental
C=O	stretch (Maybe from CO ₂ in air too)	1650-1800	1659.71
C-O	stretch	1020-1275	1156-1248
C-H	stretch, CH, aromatic	3030	3235.34
C-H	stretch, Nujol	2853	2855.0
	• CH ₂ Symmetric	2926	2924.8
	• CH ₂ Antisymmetric	2963	2953.4
	• CH ₃ Antisymmetric		
C-H	antisymmetric scissors, CH ₃ , Nujol	~1460	1445.5-1483.7
C-H	symmetric scissors, CH ₃ , Nujol	1380	1378.29
C-C		1150-1250	1090.1-1031
C=C	bend, Aromatic, Conjugated	1500-1700	1578-1613
O-H	bend (water?)	800-1000	893.879
O-H	stretch (from RCOOH)	1760	1750.67
R-COOH		2500-3000	2594.3-2725.3
o-benzene	Benzene Bonds	730-770	760.4

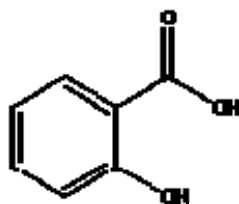


Figure 3a Salicylic Acid⁵

³ Source: Chemfinder: <http://chemfinder.cambridgesoft.com/result.asp>

⁴ Sources: <http://www.wpi.edu/Academics/Depts/Chemistry/Courses/CH2670/infrared.html>,
<http://www.chem.ucla.edu/~webspectra/irtable.html>, Lab Handouts for NUJOL spectra

⁵ All chemical structures taken from ChemFinder Database: <http://chemfinder.cambridgesoft.com/result.asp>

Table 4b: IR Band Assignments of Benzoic Acid

Bonds		Stretching Wavelength (1/cm)	
Name	Notes	Theoretical	Experimental
C=O	stretch (Maybe from CO ₂ in air too)	1650-1800	1687.52
C-O	stretch	1020-1275	1090-1249
C-H	stretch, CH, aromatic	3030	3071.1
C-H	stretch, Nujol	2853	2854.6
	• CH ₂ Symmetric	2926	2924.4
	• CH ₂ Antisymmetric	2963	2952.8
	• CH ₃ Antisymmetric		
C-H	antisymmetric scissors, CH ₃ , Nujol	~1460	1425.8-1496.3
C-H	symmetric scissors, CH ₃ , Nujol	1380	1377.2
C-C		1150-1250	1128.7-1186.3
C=C	bend, Aromatic, Conjugated	1500-1700	1583-1602
O-H	bend (water?)	800-1000	935
O-H	stretch (from RCOOH)	1760	1788
R-COOH		2500-3000	2561.7-2726.6
Benzene	Mono-Substituted	730-770	668.7-708.66

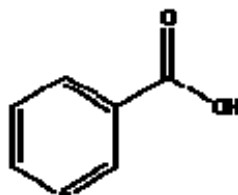


Figure 3b Benzoic Acid

Table 4c: IR Band Assignments of Polystyrene⁶

Bonds		Stretching Wavelength (1/cm)	
Name	Notes	Theoretical	Experimental
C=O	stretch (Maybe from CO ₂ in air)	1650-1800	1667.21-1801.91
C-H	stretch, CH, aromatic	3030	3025.6
C-H	stretch, CH	3000-3100	3001.5-3101.9
C-H	bend, CH ₂	~1400	1451.01
C-H	bend, CH ₃ (end of chain?)	1300-1400	1328.8-1371.7
C-C		1150-1250	1154.5
C=C	bend, Aromatic, Conjugated	1500-1700	1541.8-1601.3
O-H	bend (water?)	800-1000	842.0-964.8
Benzene	Mono-Substituted	730-770	762.2
Nitrogen	Various Nitrogen stretch bonds (from air)	>3000	>3000

⁶ NOTE: Since the peaks were labeled incorrectly on the spectrum obtained for PS in class, another student's spectrum was used for analysis.

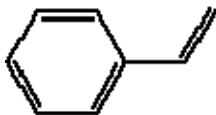


Figure 3d Styrene Monomer

Table 4d: IR Band Assignments of Acetylsalicylic Acid (Aspirin)

Bonds		Stretching Wavelength (1/cm)	
Name	Notes	Theoretical	Experimental
C=O	stretch (Maybe from CO2 in air too)	1650-1800	1690.95
C-O	stretch	1020-1275	1094.8-1255.79
C-H	stretch, Nujol • CH ₂ Symmetric • CH ₂ Antisymmetric	2853	2855.2
		2926	2922.3
C-H	antisymmetric scissors, CH ₃ , Nujol	~1460	1419.1-1483.0
C-H	symmetric scissors, CH ₃ , Nujol	1380	1374.9
C-C		1150-1250	1135.06
C=C	bend, Aromatic, Conjugated	1500-1700	1575.02-1605.57
O-H	bend (water?)	800-1000	917.07
O-H	stretch (from RCOOH)	1760	1753.6
R-COOH		2500-3000	2545.3-2699.2
o-benzene	Benzene Bonds	730-770	755

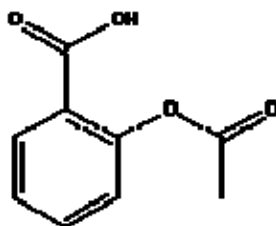


Figure 3d Acetylsalicylic Acid (Aspirin)

Using the bandwidth assignments for the four different substances, one is able to analyze the purity of the aspirin sample. Contamination from the unreacted acetic acid was hard to detect because the characteristic peaks, such as COOH and C=O were present in all the different powder samples. Polystyrene also showed many peaks at a wavenumber greater than 3200. This could be a result of the apparatus detecting the presence of the nitrogen in air. In addition, water and carbon dioxide were the two primary impurities detected by the apparatus.

The source of water impurity was either air or water from the hygroscopic salt plates. One of the impurities existed at about $800\text{ cm}^{-1} < \lambda < 1000\text{ cm}^{-1}$. By looking at the polystyrene spectrum, it was determined that this

impurity was water. The range of $800\text{ cm}^{-1} < \lambda < 1000\text{ cm}^{-1}$ is that of O-H in the bend vibrational mode, which is one of the modes of water. This O-H bend was not one of the modes of O-H in the carboxylic acid or alcohol (salicylic acid) because they appear at very different wavelengths. The O-H stretch for a carboxylic acid is found in the range of $\lambda = 2500\text{-}3000\text{ cm}^{-1}$, while the O-H stretch for alcohols and phenols (for salicylic acid) is $\lambda = 3200\text{-}3550\text{ cm}^{-1}$. Since polystyrene does not have any oxygen atoms, the O-H bend seen in the spectrum must be a result of the water molecules in air. Because air was the background for all the tests, this peak should be visible in all the spectra, which it was. Consequently, water, which was either in the air or in the salt plates, was one of the impurities detected by the FTIR.

Another impurity was carbon dioxide. Once again, this was inferred from comparisons of the polystyrene spectrum with the other three. Polystyrene, which contains no oxygen atoms, has a peak at $\lambda = 1659\text{ cm}^{-1}$, which is the peak for the C=O stretch. This peak appears in all of the graphs, which suggests that the CO_2 in the air was one of the contaminants. Polystyrene, which has no C=O bonds in the structure, shows the lowest transmission of the C=O peak for the 3 spectra. The transmission at the C=O peak was less than 10, while the transmission at the other C=O peak for the other 3 spectra was greater than 10. Consequently, CO_2 is one of the impurities from air detected by the infrared scan.

FTNMR Characterization

The various NMR spectra are attached in Appendix A. Comparing the Aspirin proton NMR with the given spectrum⁷ shows that the two are very similar. The chemical shifts, δ , occur at similar points, and the relative intensities are also very close. Table 5 summarizes the NMR information for aspirin while Figure 4 shows aspirin with its labeled protons.

⁷ Source: Class Notes: NMR 25 and 26

Table 5: H-NMR Spectrum Details

Peak (δ ppm)		Proton	Multiplicity	Coupling Constant
Spectrum from NMR 25	Experimental Spectrum			
0.001	0.013	TMS	Singlet	-
2.354	2.363	CH ₃ (3H)	Singlet	-
7.1 7.11 7.187 7.201 7.27 7.284	7.109 7.12 7.196 7.21 7.283 7.29	C	Triplet of Doublets	$J_{CB} = 0.0885$ $J_{CD} = 0.01$
7.352 7.369 7.436 7.453	7.359 7.374 7.444 7.46	D	Doublet of Doublets	$J_{DC} = 0.016$ $J_{DE} = 0.015$
7.552 7.572 7.639 7.661 7.723 7.744	7.557 7.581 7.646 7.665 7.728 7.75	B	Triplet of Doublets	$J_{BC} = 0.022$ $J_{BA} = 0.083$
8.09 8.109 8.177 8.197	8.098 8.119 8.184 8.205	A	Doublet of Doublets	$J_{AB} = 0.021$ $J_{AC} = 0.086$
~10.8	10.505	H (from OH)	Singlet	

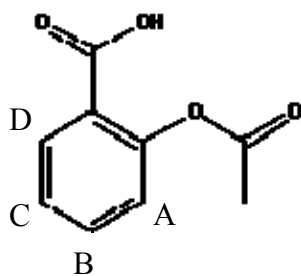


Figure 4 Aspirin Protons

One can see that the “A” proton was the furthest downfield. This is a result of the deshielding from the oxygen in the acetyl group. Proton “B” also experienced some of the effects of the acetyl oxygen. In addition, the “C” proton had the lowest δ because it was the most shielded. Table 5 also shows that many of the peaks were split. While this was expected for the “B” and “C” protons, which were split by two different protons on two different carbons, it was not expected for the “A” or “D” protons. This extra splitting may be a result of impurities. Impurities could also be

seen in the spectrum where the peak may have been split into a triplet. This occurred at $\delta = 7.29$ and $\delta = 7.120$. Consequently, although the NMR spectrum was close to the one given in class, impurities were visible in the spectrum.

The C-NMR was also analyzed. Table 6 summarizes the C-NMR results⁸.

Table 6: CMR Spectrum Details

Peak (δ ppm)		Carbon
Spectrum from NMR 32	Experimental Spectrum	
~17	17.494	Reference
20.629	20.684	
57.544	57.627	CH ₃
124.47	124.54	C
124.895	~125	E
126.621	126.694	B
132.621	132.698	D
134.452	134.506	A
151.971	152.021	F
167.291	167.368	C=O
170.899	171	COOH

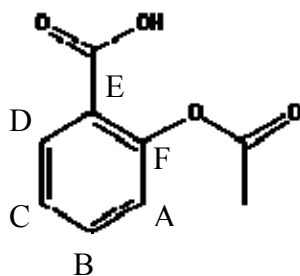


Figure 5 Aspirin Carbons

As for the H-1NMR, the δ of the experimental spectrum and the δ of the spectrum given in class⁹ were very similar. The first two peaks were the reference peaks. The peak at $\delta = 124.895$ was very small in the experimental spectrum. In fact, it had about the same intensity as some of the noise peaks. This peak had such a small intensity because it had a long relaxation time. Consequently, more scans were probably needed to detect it. In addition, the “F” peak was furthest up-field because of deshielding from the oxygen atom of the acetyl group. Impurities may have also contributed to its δ being higher than the δ of the other aromatic carbons. Because of the same deshielding from oxygen atoms, the carbonyl groups have the highest relative δ .

⁸ Source: <http://www.chem.ucalgary.ca/courses/351/Carey/Ch13/ch13-cnmmr-1.html>
http://www.chem.uni-potsdam.de/tools/e_13c.html

⁹ Source: Class Notes: NMR page 33

This lab accomplished many of the desired objectives. First, the aspirin was synthesized using acetic anhydride, salicylic acid, and an acid catalyst. By the melting point, FTIR, and FTNMR analysis, it was determined that the aspirin was relatively pure. Some impurities, such as that of water and carbon dioxide, were detected. However, the amounts were very small. In conclusion, relatively pure aspirin was successfully synthesized.