

Unknown Compound Report: Squinting Fry a.k.a. Succinic Acid

CORRECT!

I. Physical Properties

	Experimental	Literature <sup>[1]</sup>
Physical State	solid ✓	solid
Melting Point	169-171°C <i>169 low</i>	184-186°C
Boiling Point	n/a	235 °C
Color	White (powder) ✓	White (powder)

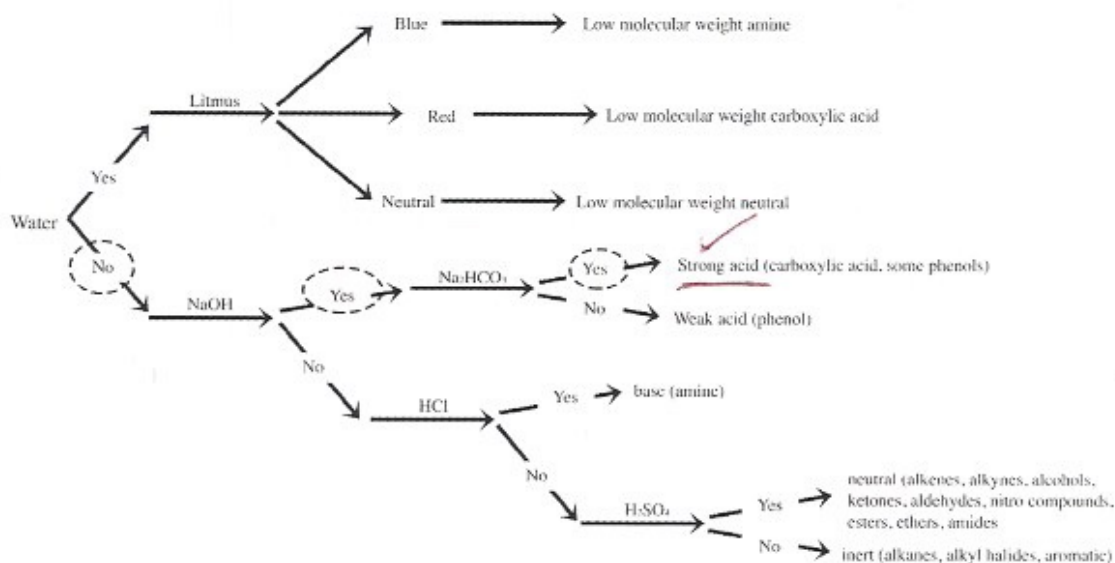
Table 1: Physical Properties of Succinic Acid

*But OK, due to impurity.*

The physical properties of Squinting Fry match those of succinic acid well. Both are solids and the melting points are somewhat close. However, the experimental melting point is slightly low, and that is likely due to the inherent impurity in the compound. This impurity was discovered through NMR, which showed benzylic peaks, even though the compound is not aromatic, which was confirmed by Ryan. The experimental melting start point has an error of 8.15%.

II. Solubility

Figure 1: Solubility Diagram for Squinting Fry



The compound was not soluble in water, but was soluble in both NaOH and NaHCO<sub>3</sub>, which indicated a strong acid that was either a phenol or a carboxylic acid. However, it was not highly

soluble in either, possibly due to the dicarboxylic acid aspect of the compound, which was unknown at the time of the solubility test. It also did not dissolve in HCl or H<sub>2</sub>SO<sub>4</sub>.

### III. Classification Tests

Test	Tests For	Observations	Positive or Negative
Beilstein	Halide	No green flame	negative ✓
Bromine – Methylene Chloride	Unsaturation	Solution did not turn clear	negative ✓
Potassium Permanganate	Unsaturation	Solution remained purple	negative ✓
Ignition	Aromaticity	No soot or yellow color, but compound is highly flammable.	negative ✓
2,4-Dinitrophenylhydrazine (DNP)	Aldehydes/Ketones	No orange precipitate, very different from positive control	negative ✓
pH of an Aqueous Solution	Acid	pH paper turns red, compound dissolves after adding a ridiculous amount of ethanol	positive ✓
sodium bicarbonate	Acid	Solution formed bubbles	positive ✓

Table 1: Classification Test Results

The classification tests indicated a lack of unsaturation, aromaticity, ketones, aldehydes, and halides. They confirmed the presence of an organic acid, which could either be a strong phenol or a carboxylic acid.

Other than these classification tests, it was also observed that the compound did not dissolve in CDCl<sub>3</sub>, D<sub>2</sub>O, or DMSO. It dissolved best in acetone, although some effort was needed to dissolve it, and it would dissolve, albeit unwillingly, in ethanol.

#### IV. IR Spectrum

Figure 2: Experimental IR Spectrum Using KBr Pellet

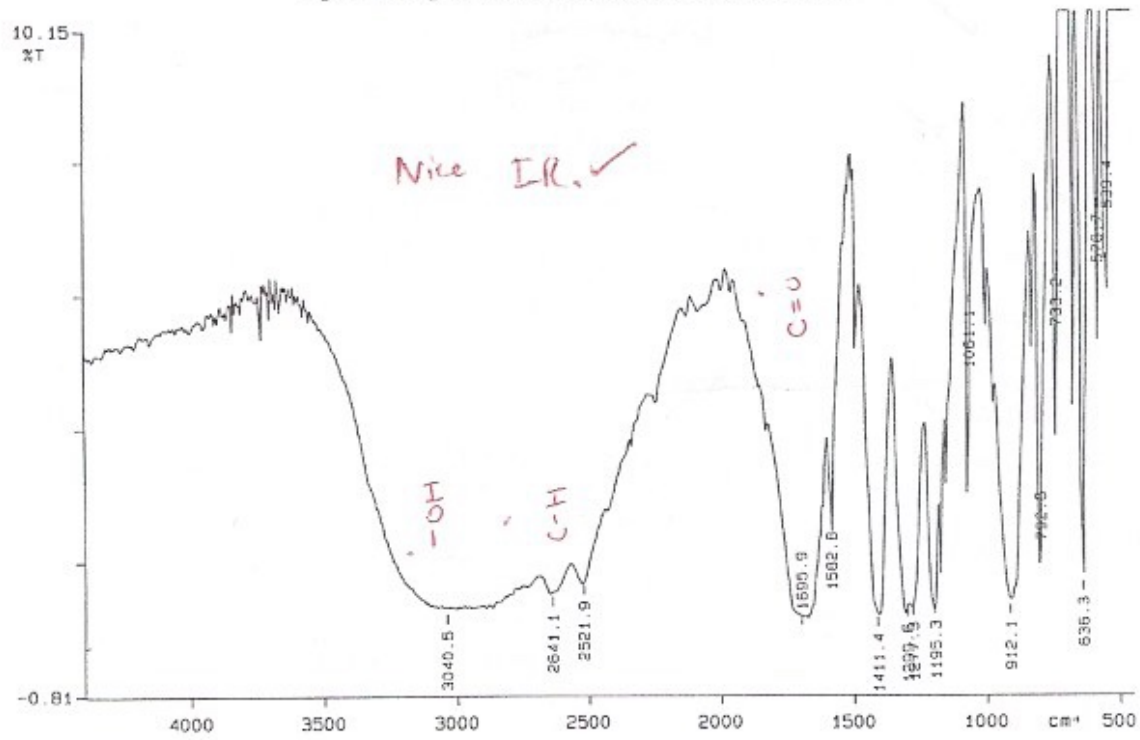
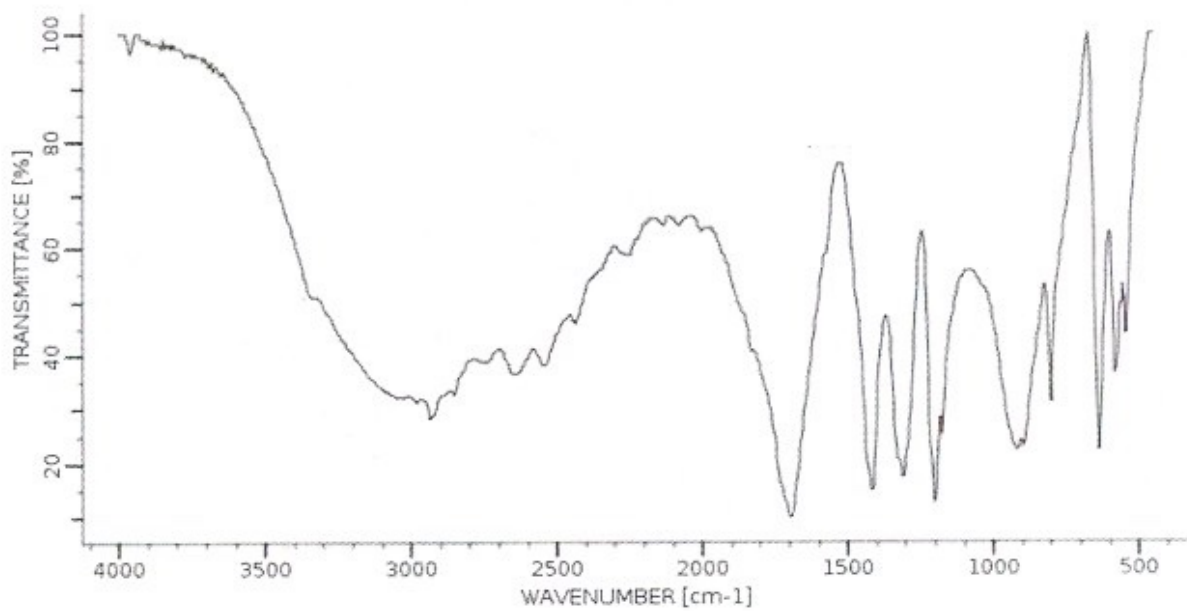


Figure 3: Literature IR Spectrum<sup>21</sup>



Functional Group	Expected Peak (cm <sup>-1</sup> ) [14]	Observed Peak <sup>*</sup>
C=O ✓	1725–1685 (aldehyde) – often 1725 1780–1665 (ketone) – often 1715 1725-1690 (carboxylic acid) – broad ~1735 (ester)	1695.9
O-H (alcohol) ✓	3600–3200 (alcohol) 3300-2500 (acid)	3040.5
C=C	1680-1620 (aliphatic) 1600–1450 (aromatic, often four sharp absorptions that occur in pairs near 1600 cm <sup>-1</sup> and 1450 cm <sup>-1</sup> )	---
C≡C	2250-2100 (sharp) None in symmetrical alkynes	---
C≡N	~2250	---
C-H (saturated) ✓	3000-2850 (alkane, stretch) 1465 (CH <sub>2</sub> , bend) 1450, 1375 (CH <sub>3</sub> , bend)	2641.1, 2521.9
C-H (unsaturated)	3310-3200 (alkyne terminal) 3150-3000 (vinyl stretch, alkene) 1000-700 (out of plane, alkene) >3000 (stretch, aromatic) 900-690 (out of plane, aromatic), often ~700	792.8 – impurity?
N-H	3500-3300 (two peaks for aliphatic and primary) 1640-1560 (primary) 1580-1490 (secondary) 1600-1450 (aromatic)	---
NO <sub>2</sub>	1560, 1350 (strong)	---
C-X	C—F Stretch, 1350–960 (C-F) 850–500 (C-Cl) right of 667 (C-Br, C-I)	---
C-O	1300-1000 (ether) 1730-1700 (acid) 1320-1210 (acid, strong) 1700-1640 (amide) 1830–1800 & 1775–1740 (anhydride)	Covered by C=O 1299.6/1277.3

Table 2: IR Spectrum Analysis

When done in nujol mull, Squinting Fry had an IR that that was uncertain in the alcohol region, which is undesirable for a compound known to be an acid. The compound also did not work well in the nujol mull and gave a suspect IR spectrum. In order to measure an accurate IR spectrum, a KBr pellet was created. However, as is the commonly a frequent problem with a KBr plates, the

transmittance is very low. As a result, the peaks in the fingerprint region look quite jagged. However, the IR is enough to show an extremely broad alcohol stretch, a C=O bond, and little of anything else. The IR spectrum taken experimentally, even if the absorbance is somewhat uneven, also matches the literature IR spectrum fairly well. While some of the thinner peaks on the experimental spectra do not show up on the literature spectra, the more major peaks do. The broad peak at  $\sim 3000\text{cm}^{-1}$ , the two sharper peaks at about  $\sim 2500\text{cm}^{-1}$ , the carbonyl peak at  $\sim 1700\text{cm}^{-1}$ , the three sharp peaks between  $1000\text{cm}^{-1}$  and  $1500\text{cm}^{-1}$ , and the peak around  $900\text{cm}^{-1}$  all match up with the literature spectrum. The various sharp peaks in the experimental IR spectrum are likely specific to the molecule but only show up so poorly due to improper scaling of the spectrum.

### V. $^1\text{H}$ NMR Spectrum

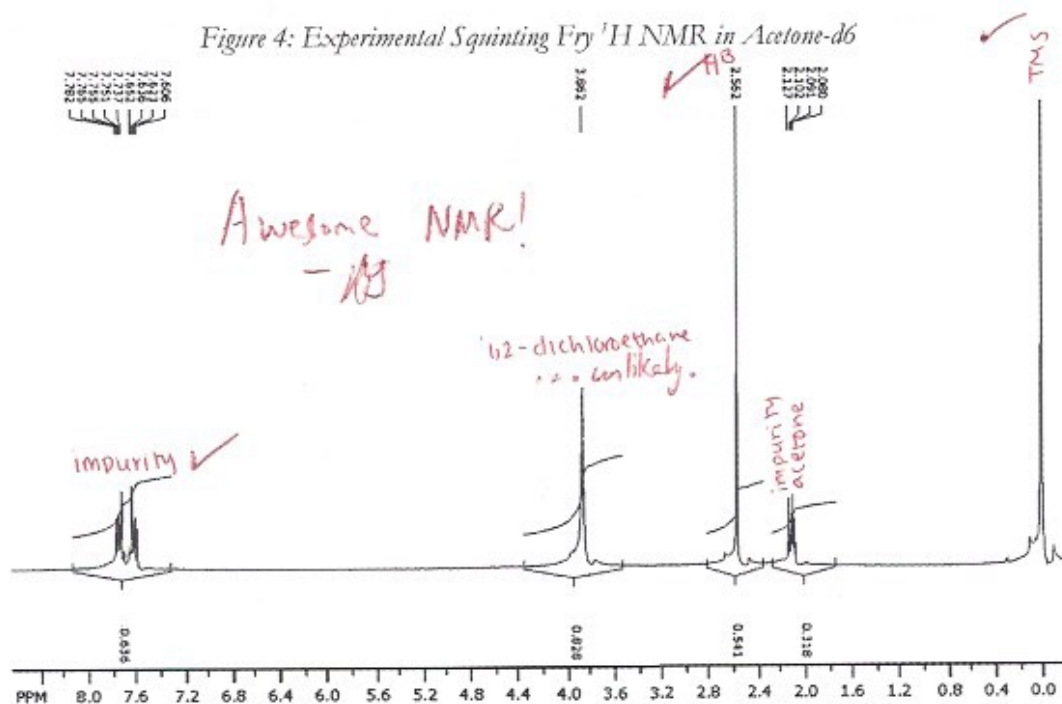


Figure 5: Experimental Squinting Fry  $^1\text{H}$  NMR in Acetone- $d_6$  – 1.2-4.2ppm Expansion

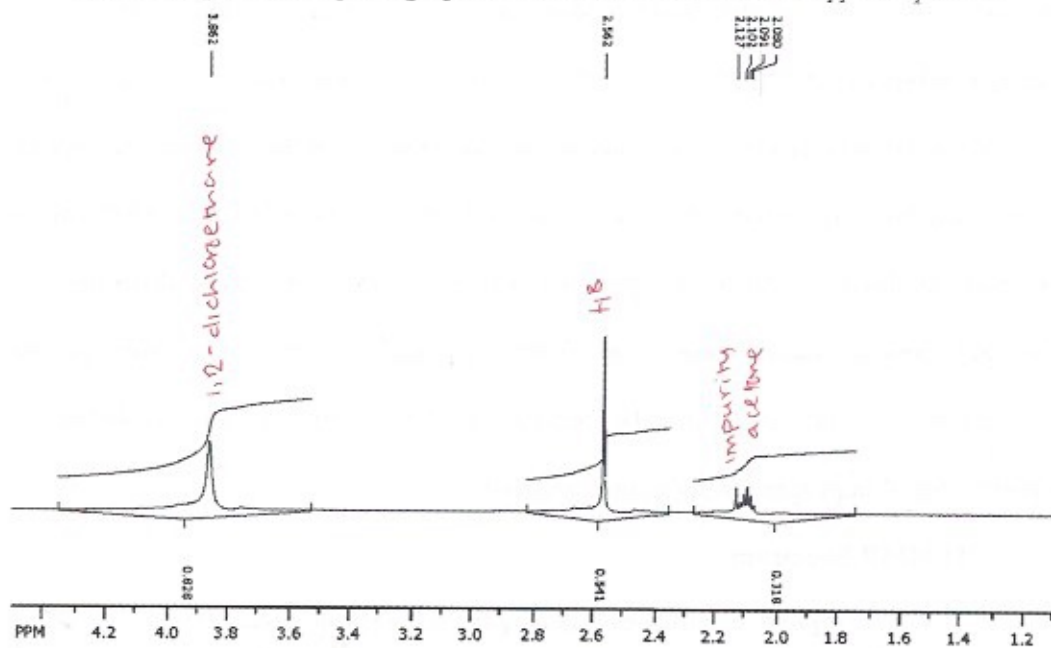


Figure 5: Experimental Squinting Fry  $^1\text{H}$  NMR in Acetone- $d_6$  – 1.88-2.26ppm Expansion

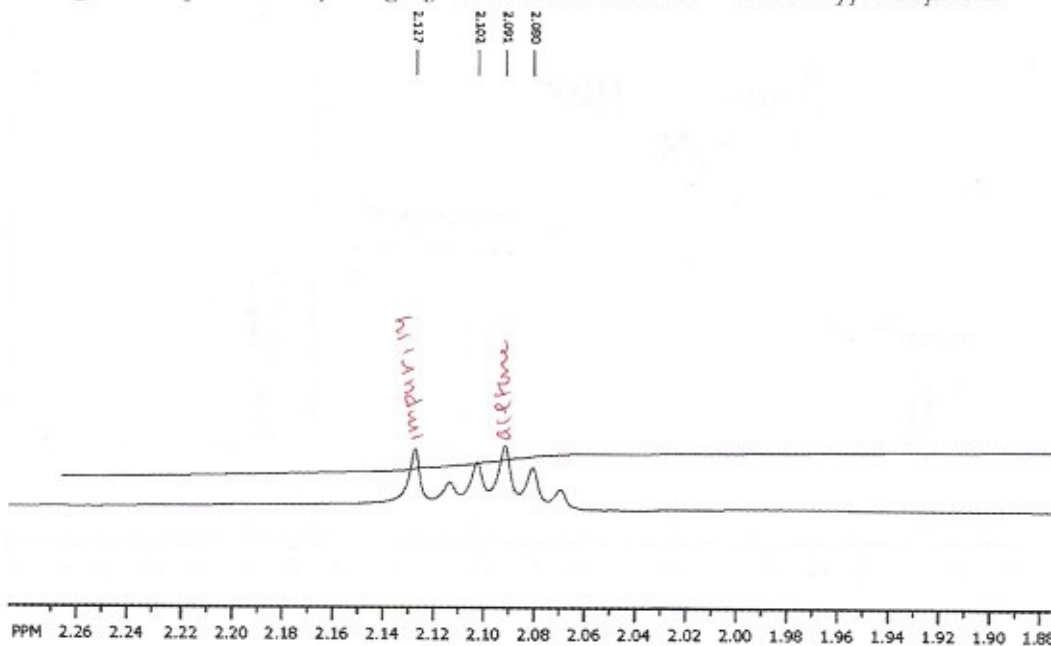


Figure 5: Experimental Squinting Fry  $^1\text{H}$  NMR in Acetone- $d_6$  – 7-8.10ppm Expansion

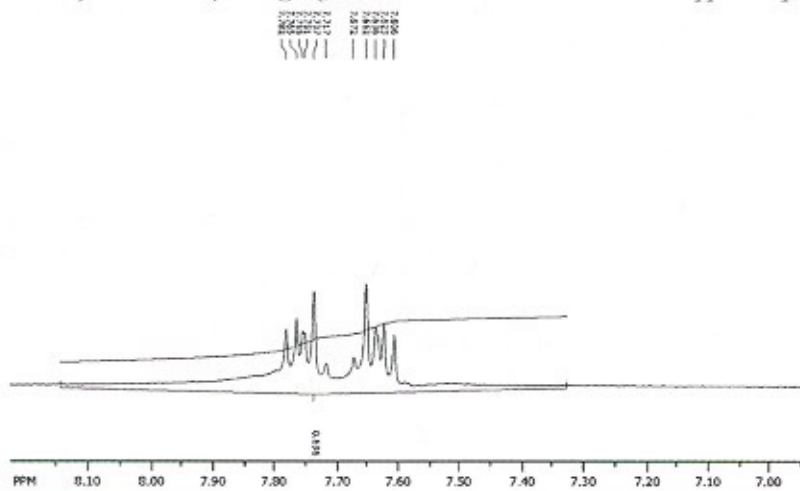


Figure 7: Succinic Acid Literature Spectrum<sup>31</sup>

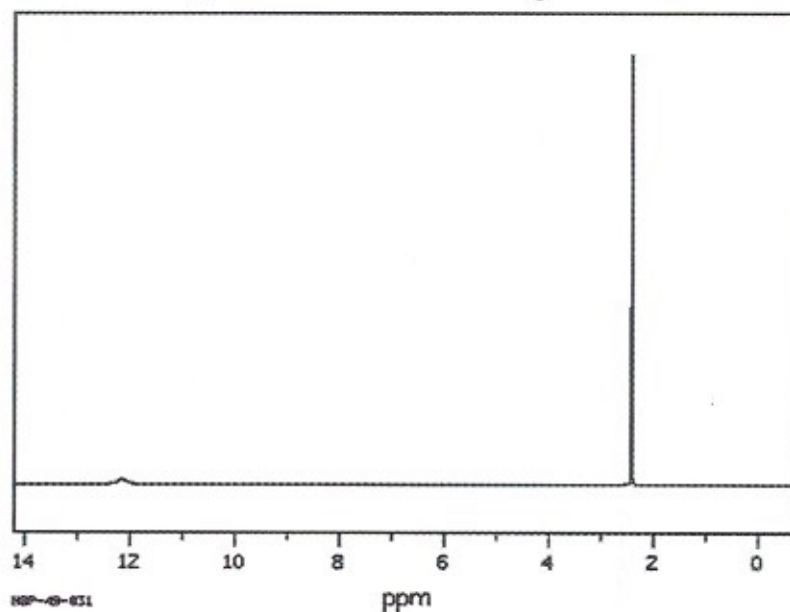
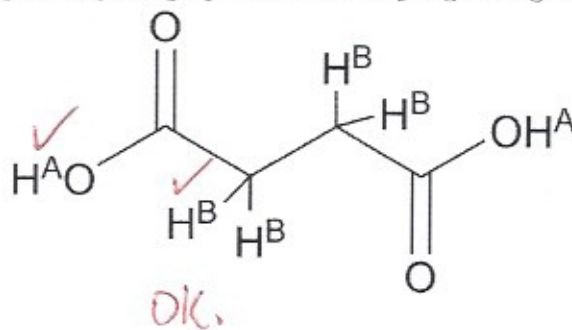


Figure 8: Squinting Fry/Succinic Acid Hydrogen Assignments



	Chemical Shift	Multiplicity	Integration	J-Value (Hz)	Assignment
Compound	2.562	s	0.541	-	H <sup>B</sup>
Solvent	2.091	q	0.318	-	Acetone <sup>[6]</sup>
Impurities	2.127	s		-	-
	7.672	m	-	-	
	7.717	m	-	-	
	3.862	s	0.828	-	1,2-dichloroethane <sup>[4]</sup>

Table 3: NMR Analysis

Toluene is a decent guess.

Figure 9: Toluene Literature NMR<sup>5</sup>

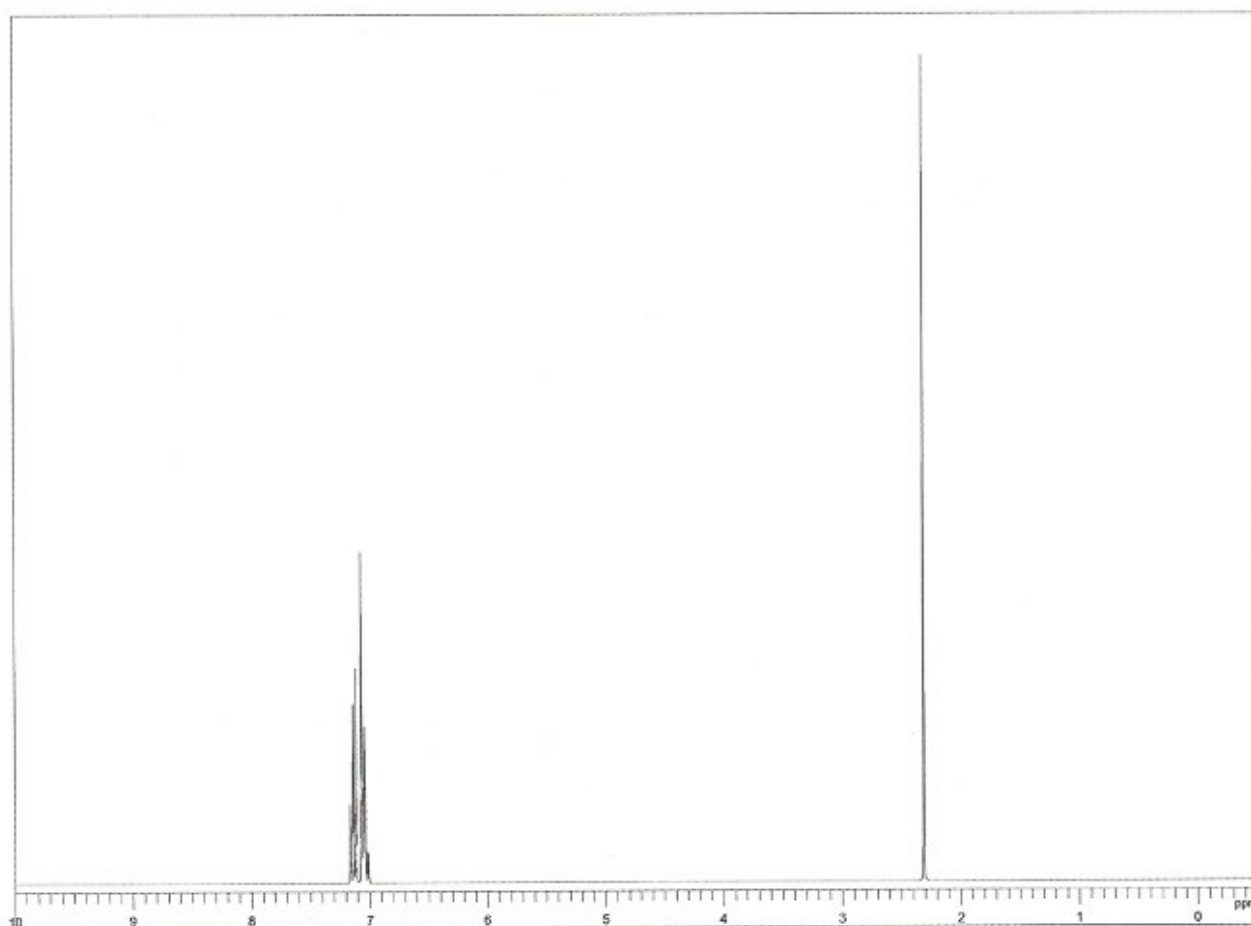




Figure 10: Aniline Literature NMR <sup>71</sup>

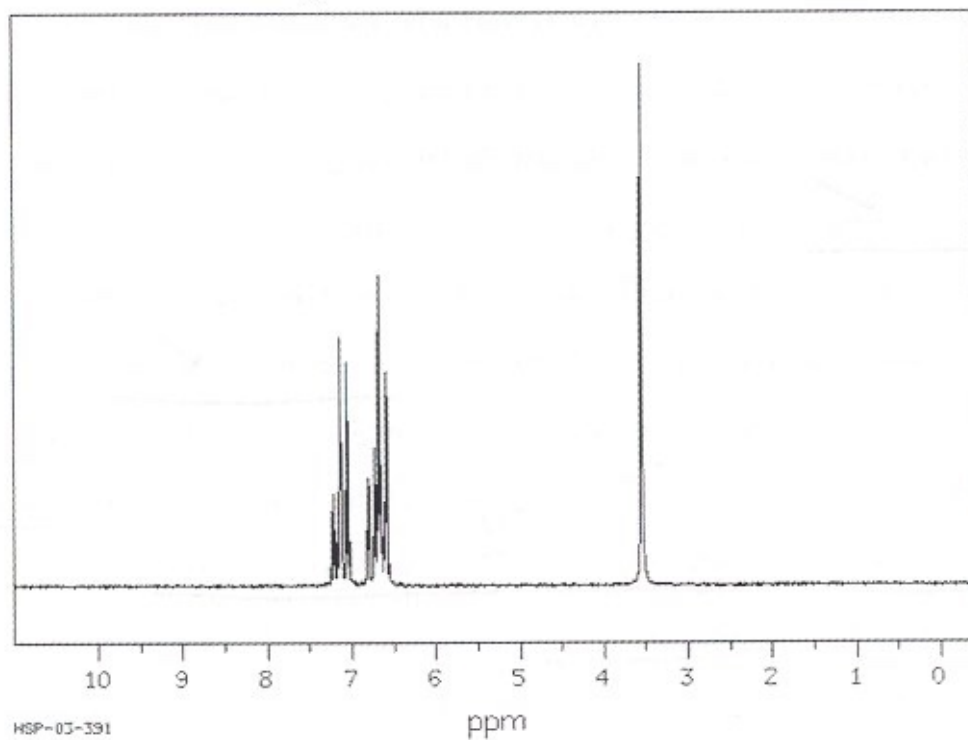
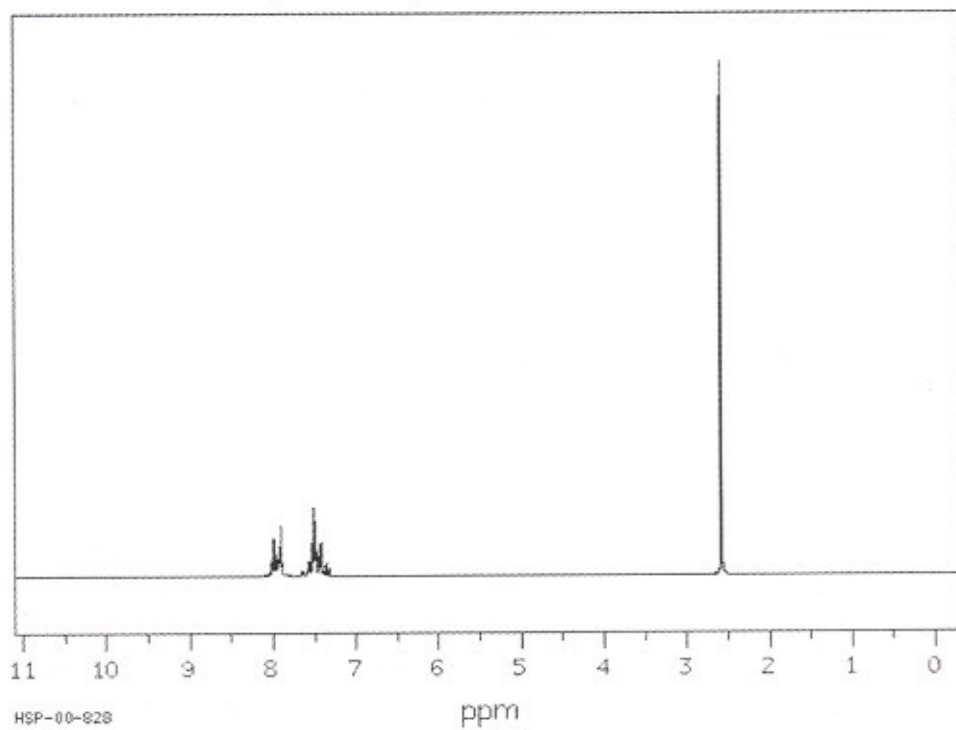


Figure 11: Acetophenone Literature NMR <sup>81</sup>



Succinic acid is a compound for which  $^1\text{H}$  NMR is not particularly useful since it essentially gives a singlet at around 2ppm, and then, possibly, a broad singlet around 12ppm for the carboxylic acid. The singlet appropriately shows up at 2.562ppm. The broad singlet is not visible in the spectrum, likely due to proton exchange. Acetone displays a quintet at 2.091ppm.

Had this been all of what had shown up in the NMR, there would have not been as much confusion about it. However, the  $^1\text{H}$  NMR spectrum shows an inherent impurity in the unknown compound. It has shown up on every NMR taken, including the one in  $\text{CDCl}_3$ , the one in DMSO, and the other two taken in acetone- $d_6$ . This particular NMR spectrum was taken in a new container, with a new pipette tip, a new NMR tube, a different pipette bulb from all the rest, and the compound was transferred with a carefully cleaned spatula. There is some compound with benzylic hydrogens, and likely a few hydrogens with a shift around 2.127ppm mixed with Squinting Fry. This could belong to any number of monosubstituted benzene products and, although at least 30 different spectra were compared to these shifts, none of those compounds had a spectrum perfectly matching what was seen. Aniline, toluene, and acetophenone all had spectra that were somewhat close, especially aniline because the nitrogen hydrogens can exchange and very chemical shifts. These spectra are attached for comparison.

Thorough Search!

The other impurity in the compound appears to be dichloroethane, which is unfortunately also likely mixed into the compound due to the care taken for gathering this spectrum.

#### VI. Derivative

Amount Compound (g)	Average Amount 0.1 M NaOH (mL)	Observations	NE (g/mol)	Error for NE
0.200	32	Phenolphthalein was used. Substance did not dissolve well in ethanol OR water, 50/50 solution dissolved only some of it initially. At equivalence point, all was dissolved.	62.5	5.93%

Table 4: Neutralization Equivalence Analysis

( $\times 2 = 125$ )

For the derivative test, the neutralization equivalent test was used. The neutralization equivalence is used to either determine the molar mass of a compound per each mole of the functional group that reacts. Since the compound was known to be an is a avid, 0.200g was dissolved in about 25mL of deionized water and 25mL of ethanol, and then the solution was titrated with 0.1M NaOH. The equation for the neutralization equivalent for an acid is as follows:

$$\frac{\text{Neutralization equivalent}}{\# \text{ acid groups}} = \frac{\text{mg acid compound}}{(\text{molarity of NaOH}) \times (\text{mL of NaOH})}$$

This equation can also be written as:

$$\frac{\text{Neutralization equivalent}}{\# \text{ acid groups}} = \frac{\text{g acid compound}}{\text{moles of NaOH to neutralize}}$$
$$\therefore \text{Neutralization equivalent} = \frac{(\# \text{ acid groups})(\text{g acid compound})}{\text{moles of HCl to neutralize}}$$

Logically it can be understood that if the compound only has one acid group per mole of compound, then the number of moles of NaOH required to deprotonate it is equal to the number of moles of compound.

Preliminary analysis of the GC-MS in Section VII indicated a molecular weight of 100. Further analysis indicated that the molecular weight was 118g/mol. As such, it can be calculated that 17mL of NaOH would be needed neutralize the acid if only one carboxylic acid functional group was present. Instead, an average of 32mL from three trials was required to neutralize the acid. As a result NE=62.5 g/mol. As the compound is known to be 118g/mol, this indicated that the substance was a dicarboxylic acid. ✓

Since the equivalence point of a weak acid titrated with a strong base is usually slightly higher than 7, phenolphthalein, which has a pH range of 8.2-10.0 for indication<sup>[9]</sup>, was used as a pH indicator. This was a valid indicator for the substance and so the result can be relied on. This

neutralization confirmed the molecular weight of the compound and, more importantly, suggested the substance was a dicarboxylic acid.

### VII. GC-MS

Figure 12: Provided GC-MS Spectrum of Squinting Fry

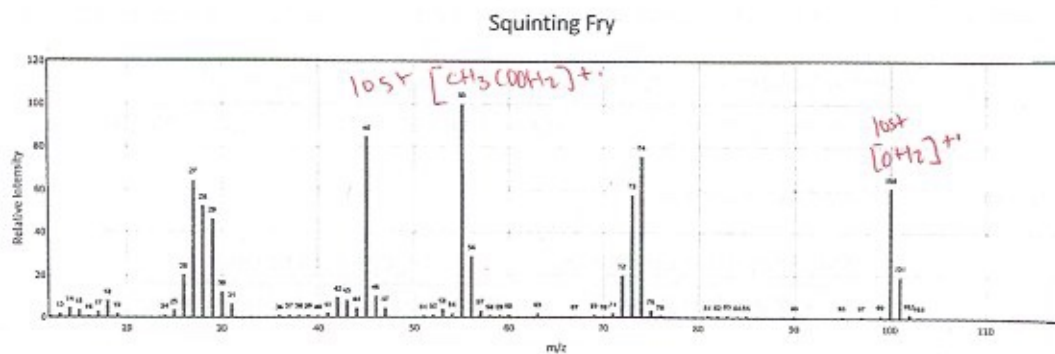
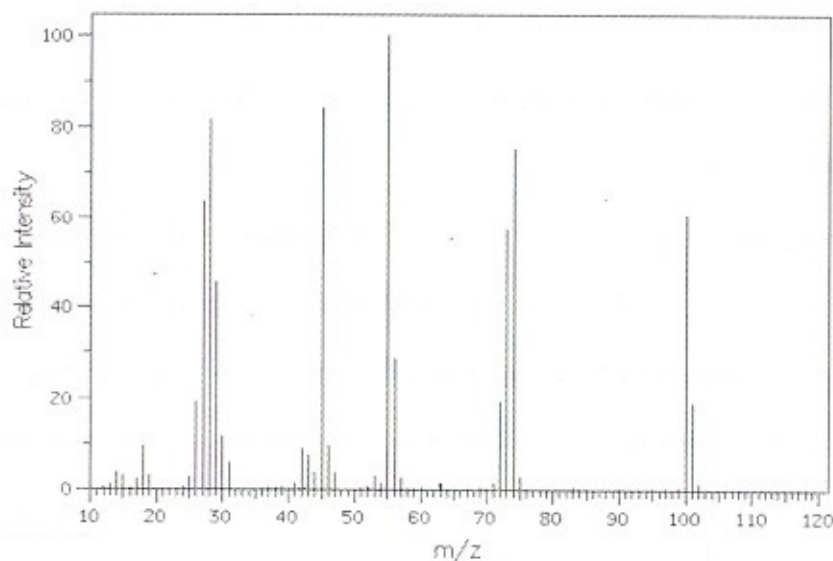


Figure 13: Literature GC-MS Spectrum of Succinic Acid



Peak (m/z)	Fragment Mass	Assignment
-	118 ✓	[M+] (not present)
100	18 ✓	OH <sub>2</sub>
74	44 ✓	COO
55	63 ✓	CH <sub>3</sub> COOH <sub>2</sub>
45	73 ✓	CH <sub>2</sub> CH <sub>2</sub> COOH

Table 5: GC-MS Analysis

A

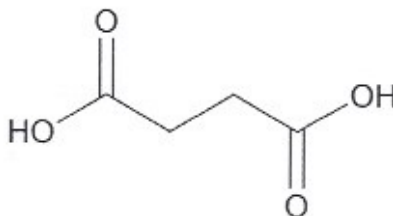
For Squinting Fry, the molecular peak is not displayed on the mass spectrum. Most organic acids have a very weak  $[M^+]$  peak<sup>[12]</sup>, but, for Squinting Fry, the molecular peak is not displayed on the mass spectrum at all. This is likely due to the inevitable dehydration of the molecule every time it goes through the machine, leading to the nonexistent  $[M^+]$  peak. As a result, the peak with the highest  $m/z$  is at 100 and corresponds to a dehydrated version of succinic acid. ✓ *yes.*

The base peak for the molecule is at  $m/z$  55 and gives a fragment mass of about 63. This is essentially due to the molecule breaking in half as it goes through the GC-MS machine and a reading of  $CH_3COOH_2$ , which is heavily protonated. Most of the other fragments are due to additional carbons or oxygens breaking off. Furthermore, the clustering of peaks on the mass spectrum is due to hydrogens being lost easily from either or both of the carboxylic acids and, as such, mostly creating strong peaks which correspond to at least one lost hydrogen. Although McLafferty rearrangements are common in carboxylic acids<sup>[12]</sup>, it does not happen here due to improper structure of the carbon chain.

The Squinting Fry provided GC-MS and literature spectrum match, so it can be concluded that the literature spectrum supports the conclusion that Squinting Fry is succinic acid.

### VIII. Structure and Discussion

Figure 14: Squinting Fry a.k.a. Succinic Acid



Chemical Formula:  $C_4H_6O_4$   
Molecular Weight: 118.09

The compound Squinting Fry was determined to be succinic acid, which has a molecular formula of  $C_4H_6O_4$  and a molecular weight of about 118g/mol. This compound has a melting point

range of 184-186°C<sup>[1]</sup>, which is close to the recorded melting point range of 169-171°C (*Section I*). This depression of melting point is likely due to the benzylic impurity in the substance that was discovered through <sup>1</sup>H NMR. The boiling point of the substance is 235 °C<sup>[1]</sup>, which is well above the capability of the boiling point apparatus and not experimentally determined of the solid unknown.

Solubility tests first indicated that the unknown was a carboxylic acid, since it dissolved in NaOH and NaHCO<sub>3</sub> in small amounts, and in almost nothing else. IR confirmed the presence of the carboxylic acid due to the broad alcohol stretch and the sharp C=O peak. The IR also essentially eliminated most other functional groups but the classification tests were used to confirm a lack of unsaturation, aromaticity, ketones, aldehydes, and halides. No compound had both an amine and a carboxylic acid, so that was also not a possibility.

Although initially GC-MS seemed to indicate that the molecular weight of the compound was 100g/mol, it is known that the [M<sup>+</sup>] peak of a carboxylic acid is extremely weak due to the common fragmentation of an alcohol group or the dehydration of the acid. The discovery that Squinting Fry was a dicarboxylic acid using the neutralization test derivative made it even more probable that the compound would always lose a molecule of alcohol or water and, as such, the true mass of the unknown was determined to be 118g/mol, which matches that of succinic acid. Of the common dicarboxylic acids with a molecular mass of 118g/mol, succinic acid's GC-MS perfectly matched the literature spectrum.

The NMR spectrum for this compound was the least helpful due to succinic acid only displaying one peak in an NMR spectrum, possibly two if the acid hydrogen shows, and also the impure nature of the compound. The main impurity is likely a monosubstituted benzene substance due to the pattern of the splitting and signal peak shifts.

As such, the compound was determined to be succinic acid, and the collected experimental data support the claim. Furthermore, collected literature spectra show close matches to the

experimental spectra collected for ~~the unknown~~ <sup>succinic acid</sup>. This dual confirmation by literature spectra and collected information from classification tests verify that the unknown Squinting Fry is actually the compound succinic acid. ✓

Grade: A

#### Works Cited

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- [12] Syracuse University. Mass Spectrometry: Fragmentation. <http://chemistry.syr.edu/totah/che575/support/3a1/3-3.MS.pdf> (accessed June 6, 2019).



EXP. NUMBER	EXPERIMENT/SUBJECT	DATE	93
	Unknown = Squinting Fry	6/7/2019	
NAME	LAB PARTNER	LOCKER/DESK NO.	COURSE & SECTION NO.
Irika Sinha		8177	CHEM 347 AA

Purpose - determine identity of compound squinting fry.

MP ~~BP~~ 169-171°C, white solid / solubility: dissolves in NaOH & NaHCO<sub>3</sub>  
no D<sub>2</sub>O, DMSO, CCl<sub>4</sub>

classify {  
 Beilstein → no green flame (-)      Ignition → no soot/yellow (-)      sodium bicarb - bubbles (+)  
 Br<sub>2</sub>/DCM → red soln (-)      DNP → no precip (-)  
 KMnO<sub>4</sub> → purple soln (-)      ad soln → red litmus (+)

IR spectrum: broad -OH<sup>cm-1</sup> peak, 1695.9 cm<sup>-1</sup> C=O 1695.9 cm<sup>-1</sup>

NMR  
 - benzylic peaks? ~7 ppm  
 - 3 862 ppm → singlet  
 - 2.562<sup>ppm</sup> → singlet  
 - 2.127<sup>ppm</sup> → singlet  
 - 2.091 ppm - quintet

Derivative: neutralization

ML HCl	Trial 1	Trial 2	Trial 3	Avg
	35	33	28	32

GCMS:  
 [M<sup>+</sup>] = 100  
 Base peak = ~~55~~ 55  
 [M<sup>+</sup>] = 118 not present  
 Base peak = 55  
 highest peak = 100

Conclusion: Succinic acid is the true identity of Squinting Fry.

COPY

SIGNATURE	DATE	WITNESS/TA	DATE
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Unknown Compound Report: **Insanity Wolf** a.k.a. **Tetralin**

I. Physical Properties

Phase: Liquid \*

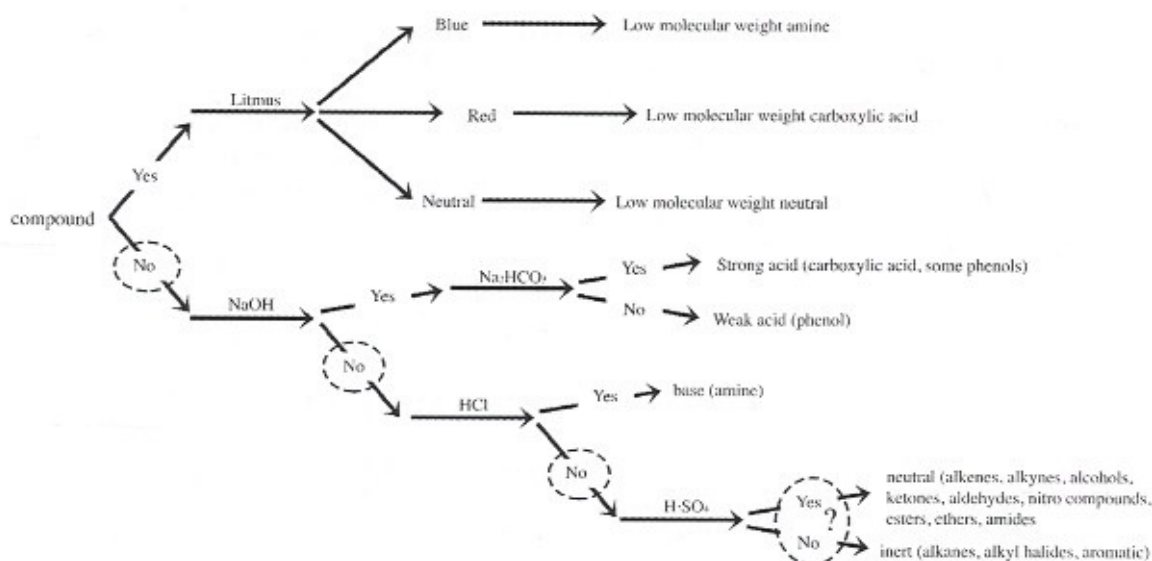
Melting Point: N/A -Liquid so  $< 25^{\circ}\text{C}$  \*

Boiling Point: 212°C *Good!*

Color: Clear \*

II. Solubility

Figure 1: Annotated Solubility Scheme for Unknown - Insanity Wolf



The compound did not dissolve in water, NaOH, or HCl, indicating that it was not a low molecular weight compound, acid, or a base. Whether or not it dissolved in  $\text{H}_2\text{SO}_4$  was ambiguous. When sulfuric acid was added, a dark red/brown precipitate was formed, suggesting that the compound reacts but is not necessarily miscible. The final structure determined implies that it should not have dissolved in  $\text{H}_2\text{SO}_4$  because it is an aromatic compound, with a cyclic alkane. Sources indicate that sulfuric acid is used to purify tetralin, and that the resulting acid layer is colored<sup>[1]</sup>. Since  $\text{H}_2\text{SO}_4$  is used to purify the tetralin, it is unlikely that the tetralin is miscible with the sulfuric acid. Instead,

*OK,  
probably a  
reaction.*

*Interesting find.*

the sulfuric acid reacts with impurities and removes them. Furthermore, commercial grade naphthalene often contains impurities, "especially methylated coumarone and corresponding sulfur compounds, which substances cause a red coloration when commercial naphthalene [is subjected] to treatment with hot concentrated sulfuric acid."<sup>[2]</sup> Although the sulfuric acid was <sup>not</sup> hot in this case, room temperature may still be enough for the sulfuric acid to react with impurities and turn red. - I like the research & thoughts here.

### III. Classification Tests

Test	Tests For	Observations	Positive or Negative
Beilstein	Halide	No green flame but extremely flammable	Negative ✓
Bromine - Methylene Chloride	Unsaturation	Solution remained red, matched negative control	Negative ✓
Potassium Permanganate	Unsaturation	Solution remained purple	Negative ✓
Ignition	Aromaticity	No sooty smoke or yellow flame	Negative ✓
2,4-Dinitrophenylhydrazine (DNP)	Aldehydes/Ketones	No orange precipitate, very different from positive control	Negative ✓

Table 1: Classification Test Results

- often unreliable, so OK.

While there are no positive classification tests, the ignition test result was likely a false negative. This may be due to the fact that the compound, tetralin, is highly flammable and caused the flame on the Bunsen burner to flare up. As the IR spectrum, as mentioned in section IV, contains a prominent peak for a benzene, the possibility of the ignition test giving a false negative is even higher.

The Beilstein test could also have been a false negative due to the flammability of the compound, but the final structure indicates that is not that case. While the Br<sub>2</sub>/DCM and KMnO<sub>4</sub> tests both indicate unsaturation, they do not react with aromatic compounds and, as such, indicate that there are no double or triple bonds, even if there is any degree of unsaturation. The DNP test indicated the lack of a carbonyl group, which was confirmed by IR.

Good!

#### IV. IR Spectrum

Functional Group	Expected Peak (cm <sup>-1</sup> ) <sup>[3][4]</sup>	Observed Peak
C=O	1725–1685 (aldehyde) – often 1725 1780–1665 (ketone) – often 1715 1725-1690 (carboxylic acid) – broad ~1735 (ester)	---
O-H (alcohol)	3600–3200 (alcohol) 3300-2500 (acid)	---
C=C ✓	1600–1450 (aromatic, often four sharp absorptions that occur in pairs near 1600 cm <sup>-1</sup> and 1450 cm <sup>-1</sup> ) 1680-1620 (aliphatic)	1602.5 & 1579.9 ✓ 1492.3 & 1451.3 ✓
C≡C	2250-2100 (sharp) None in symmetrical alkynes	---
C≡N	~2250	---
C-H (saturated) ✓ (sp <sup>3</sup> )	3000-2850 (alkane, stretch) 1465 (CH <sub>3</sub> , bend) 1450, 1375 (CH <sub>3</sub> , bend)	2929.7, 2857.1 ✓
C-H (unsaturated) ✓ (sp <sup>2</sup> )/(sp)	3310-3200 (alkyne terminal) 3150-3000 (vinyl stretch, alkene) 1000-700 (out of plane, alkene) >3000 (stretch, aromatic) 900-690 (out of plane, aromatic), often ~700	3017.3, 3059.9 ✓ 804.0, 864.2, 899.3 742.1
N-H	3500-3300 (two peaks for aliphatic and primary) 1640-1560 (primary) 1580-1490 (secondary) 1600-1450 (aromatic)	---
NO <sub>2</sub>	1560, 1350 (strong)	---
C-X	C—F Stretch, 1350–960 (C-F) 850–500 (C-Cl) right of 667 (C-Br, C-I)	---
C-O	1300-1000 (ether) 1730-1700 (acid) 1320-1210 (acid, strong) 1700-1640 (amide) 1830–1800 & 1775–1740 (anhydride)	---

Table 2: IR Spectrum Analysis

The observed peaks have been assigned, and major peaks include aromatic C-H, unsaturated, (742.1 cm<sup>-1</sup>, 3017.3 cm<sup>-1</sup>, 3059.9 cm<sup>-1</sup>, 804.0 cm<sup>-1</sup>, 864.2 cm<sup>-1</sup>, 899.3 cm<sup>-1</sup>), a major C=C indicator of aromaticity due to the pairs of peaks in the right place (1602.5 cm<sup>-1</sup> & 1579.9 cm<sup>-1</sup>, 1492.3 cm<sup>-1</sup> & 1451.3 cm<sup>-1</sup>), and the C-H, saturated, (2929.7, 2857.1). The lack of almost every functional group

indicated that the compound likely consisted of only hydrogens and carbons, including at least one aromatic ring.

Figure 2: Experimental IR Spectrum of Insanity Wolf

Great  
Spectrum!  
Very clean.

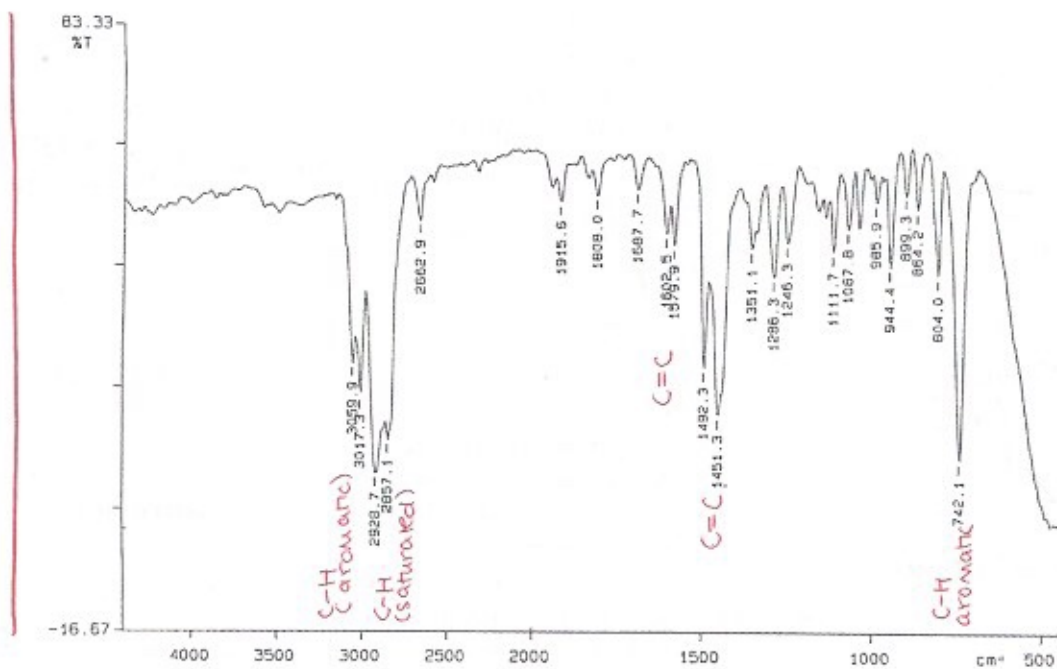
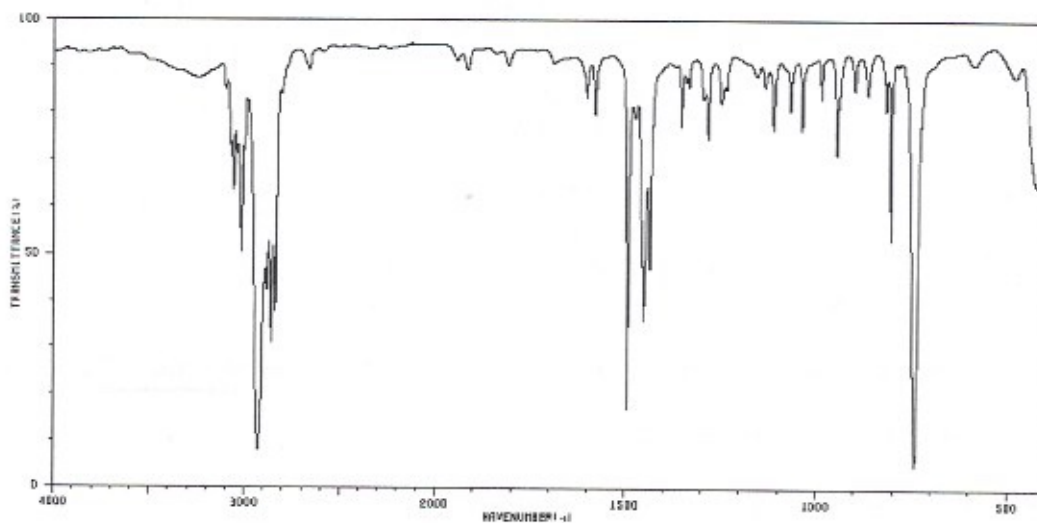


Figure 3: Literature IR Spectrum of Tetralin<sup>(4)</sup>



Both the experimental and literature spectra were taken of the liquid compound, neat. Similar to the literature spectrum, the key IR peaks in for the compound are the C-H unsaturated and aromatic stretches around  $3000\text{cm}^{-1}$ , the double peaks for C=C bonds at around  $1600\text{cm}^{-1}$  and  $1450\text{cm}^{-1}$ , and the C-H unsaturated peak at around  $700\text{cm}^{-1}$ . The rest of the spectrum from literature is simply slightly less noisy than the experimentally collected one, and normalized to a single maximum absorbance. There seems to be a missing peak at about  $1500\text{cm}^{-1}$  but it is thin enough that it could have simply blended into the  $1492.3\text{cm}^{-1}$  peak. The IR spectra match very closely, down to the tiny three peaks at around  $111.7\text{cm}^{-1}$  (Figure 2), and the literature IR spectrum for tetralin confirms the compound as tetralin. *Typo? Unusual for range to go below  $400\text{cm}^{-1}$*

## V. $^1\text{H}$ NMR Spectrum

Figure 4: Full Experimental  $^1\text{H}$  NMR of Insanity Wolf

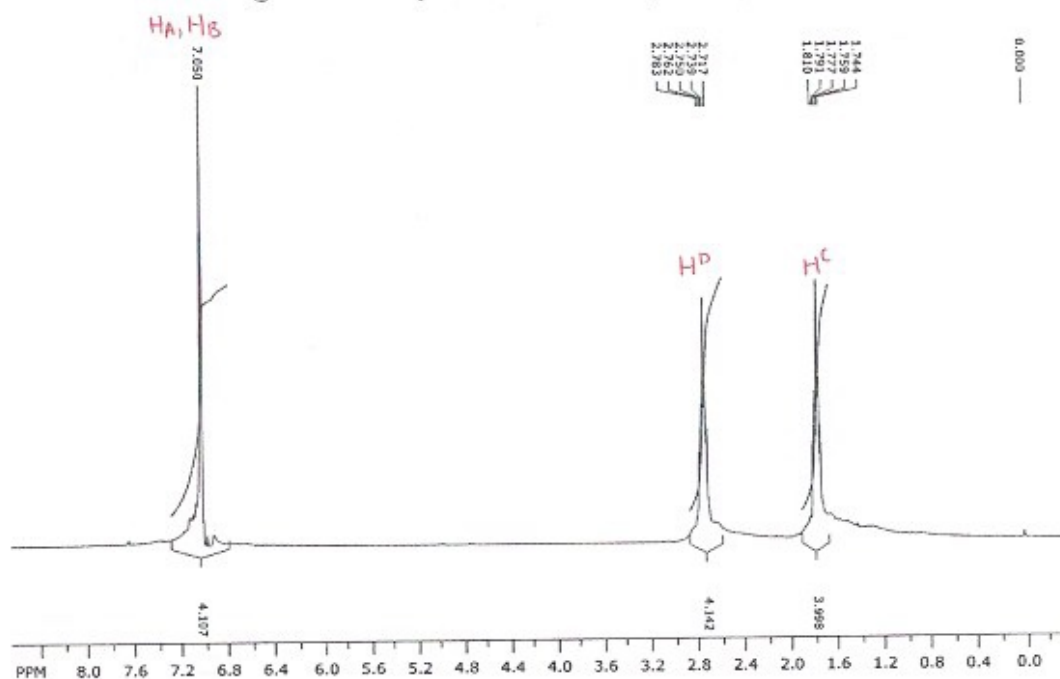


Figure 5: Experimental  $^1\text{H}$  NMR of Insanity Wolf: 1.4-2.90ppm

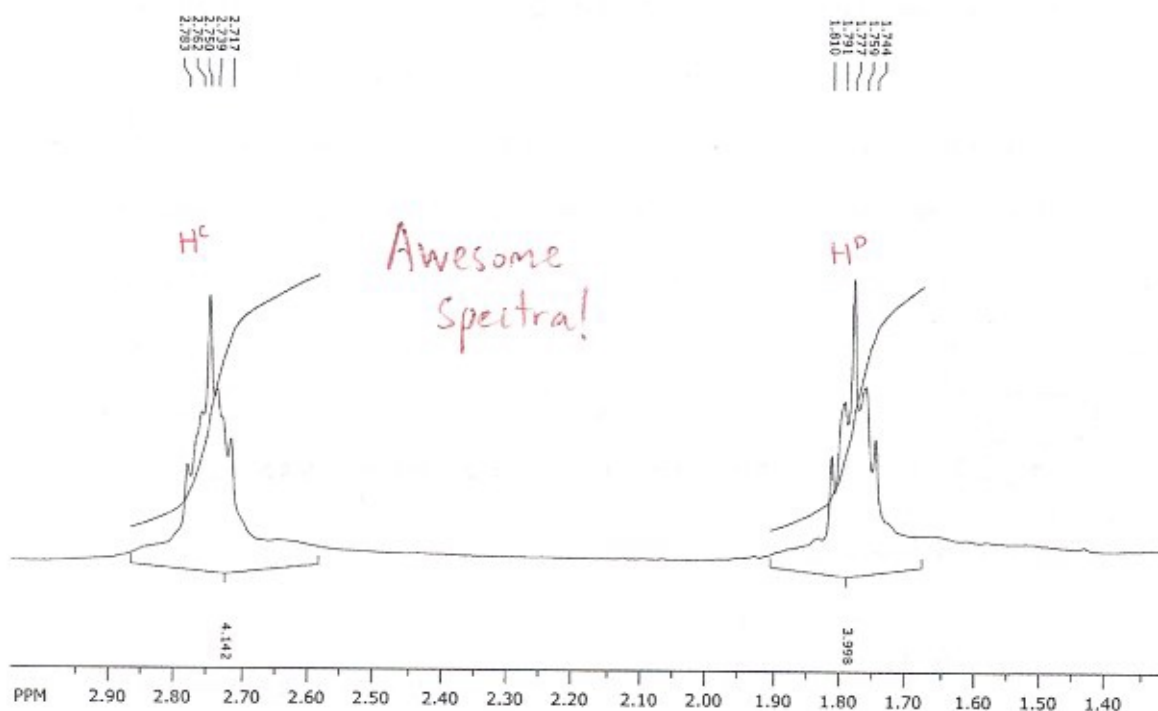


Figure 6: Experimental  $^1\text{H}$  NMR of Insanity Wolf: ~7ppm

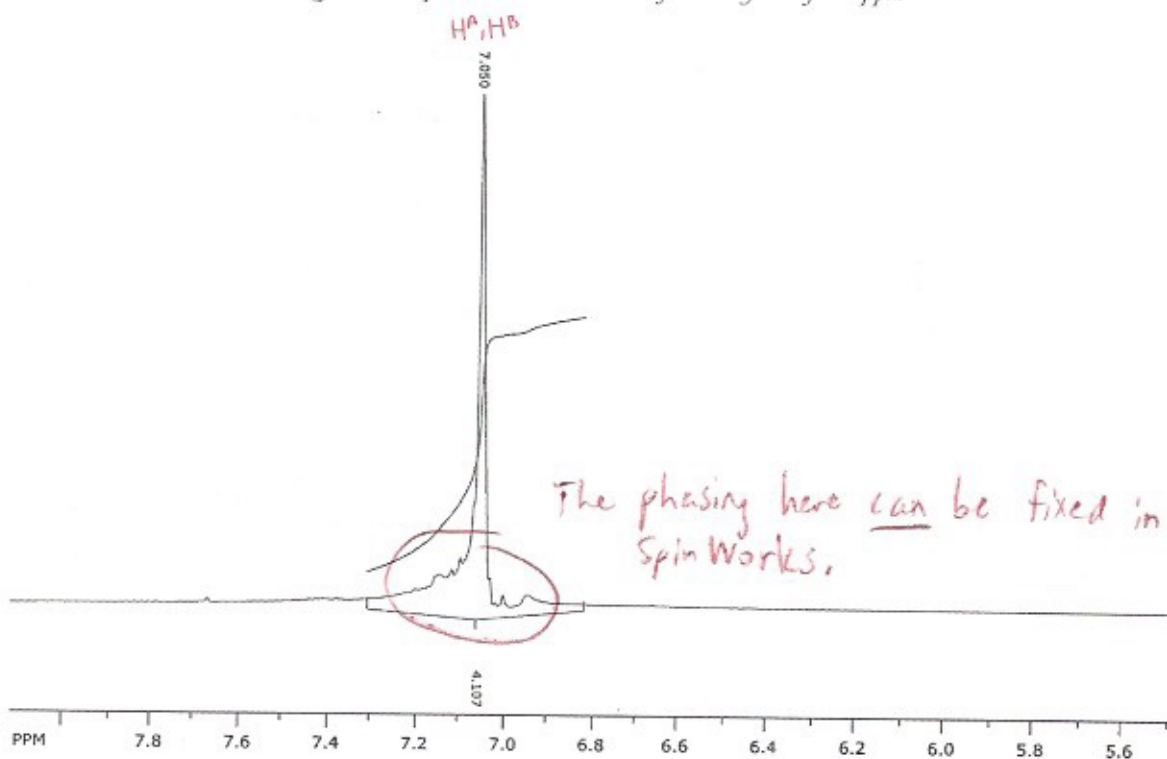
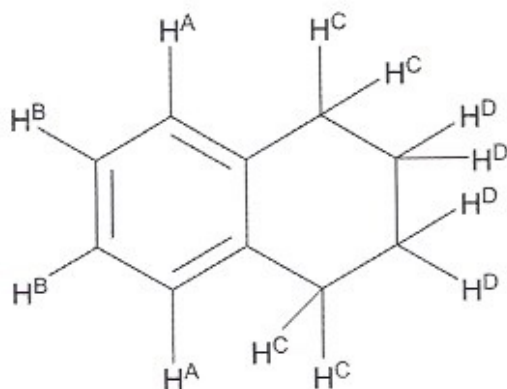


Figure 7: Tetralin with Hydrogen Assignments



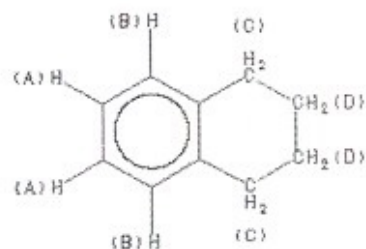
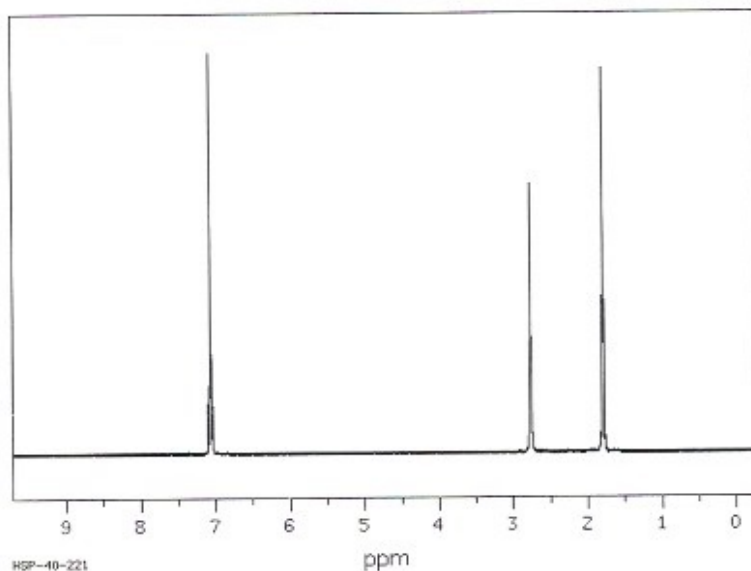
	Chemical Shift	Multiplicity	Integration	J-Value (Hz)	Assignment
Compound	1.777	dt	3.998	4.125	H <sup>D</sup>
	2.750	dt	4.142	4.125	H <sup>C</sup>
	7.050	singlet	4.017	-	H <sup>A</sup> , H <sup>B</sup>

Assignments look good!

Table 3: <sup>1</sup>H NMR Analysis

expected? they overlap, but are likely doublets.

Figure 8: <sup>1</sup>H Literature NMR Spectrum (left) Hydrogen Assignments (right)



Assign.	Shift (ppm)
A	7.077
B	7.060
C	2.767
D	1.795

The NMR spectrum also confirms the compound to be tetralin. Although the splitting of the peaks is not particularly clear in the literature spectrum, the peak values line up almost exactly for the literature and experimental spectra. Furthermore, the integrations match well. The benzylic peak, of



integration 4.017, at 7.050ppm contains overlapping peaks for  $H^A$  and  $H^B$  and, as such, is the signal for four hydrogens. Each of the quintets also signal for four hydrogens each and the integration values are 4.412 and 3.998 for the peaks at 2.750ppm and 1.777ppm, respectively. The quintets occur due to the splitting of  $H^C$  and  $H^D$  by each other.  $H^C$  has two neighbors each, and splits into a doublet of a triplet. The doublet will be due to the other  $H^C$  proton on the same carbon and the triplet is due to neighboring  $H^D$  protons.  $H^D$  is similar and is likely split into a triplet by  $H^C$  and a doublet by the other

$H^D$ .

→ Typically reported by which coupling constant is largest first.  
VI. Derivative (So maybe triplet of doublets). ~~circle~~ In any

No derivative for this compound.

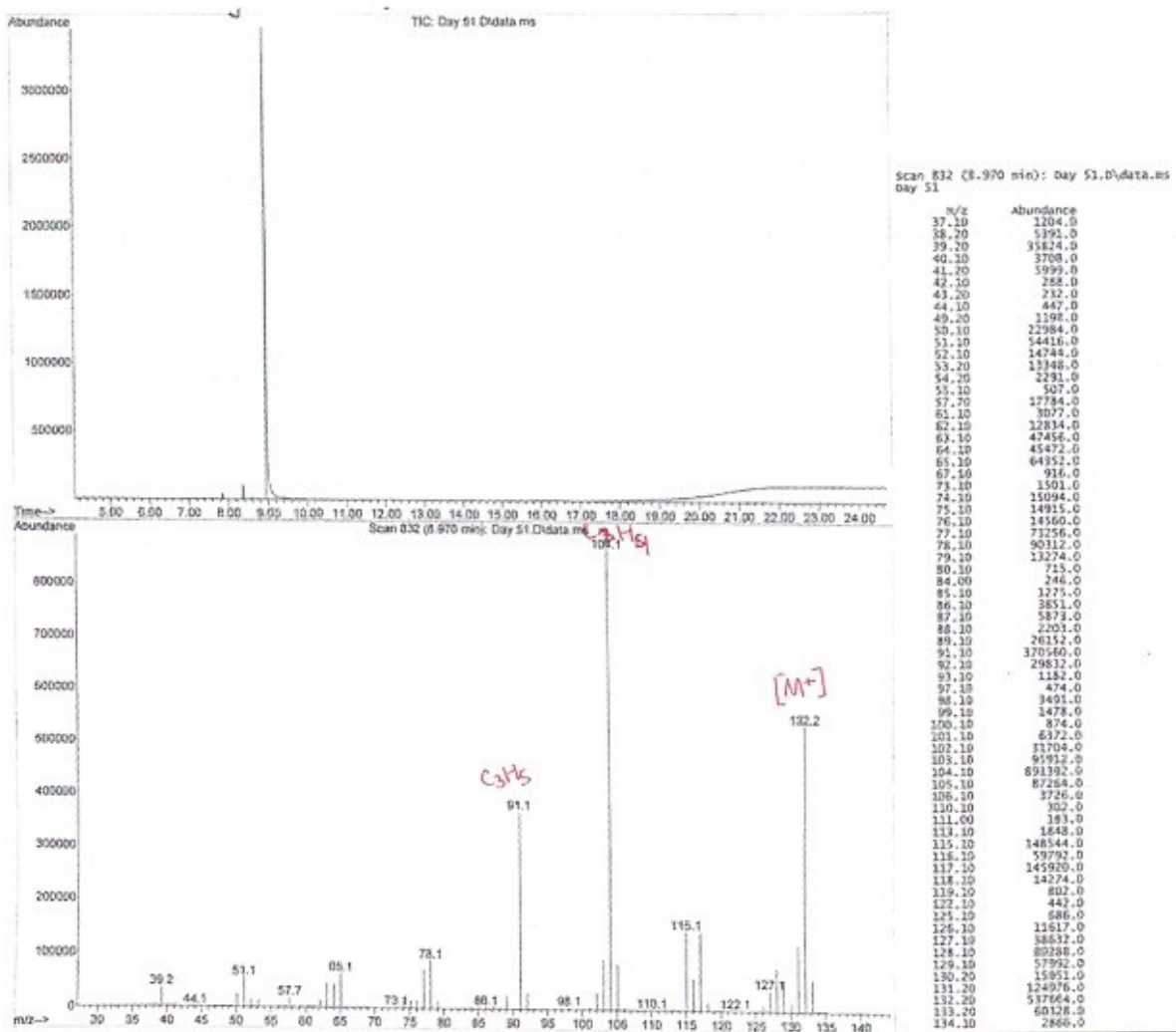
- OK.

case, it's a bit complicated, since coupling constants vary based on

axial-equatorial positions.

VII. GC-MS

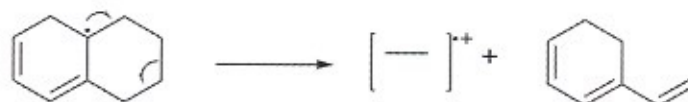
Figure 9: Experimental GC-MS Spectrum (left) and Abundance (right)



Peak (m/z)	Fragment Mass	Assignment <sup>[5]</sup>
132.2	-	[M <sup>+</sup> ] ✓ Good
104.1	28.1	C <sub>2</sub> H <sub>4</sub> ✗
91.1	41.1	C <sub>3</sub> H <sub>5</sub> -
78.1	54.1	C <sub>4</sub> H <sub>6</sub> -
65.1	67	C <sub>5</sub> H <sub>7</sub> -
51.1	81.1	C <sub>6</sub> H <sub>9</sub> -
39.2	93	C <sub>7</sub> H <sub>13</sub> ✗

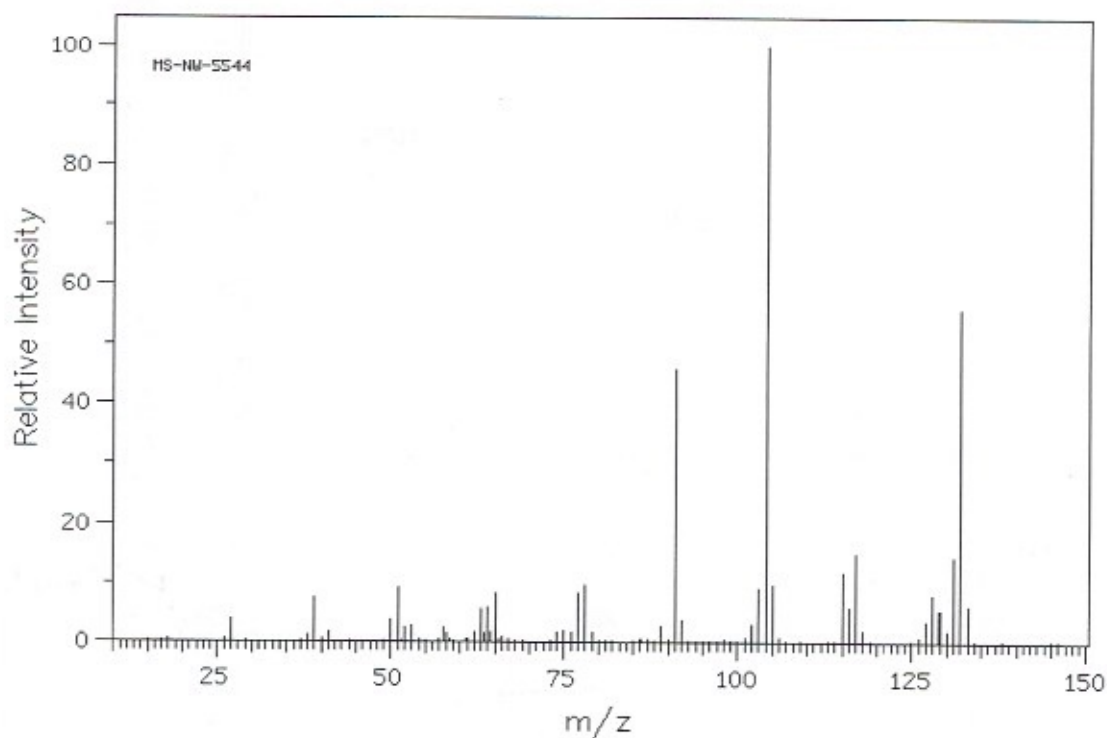
Table 4: GC-MS Analysis

Figure 10: Possible Fragmentation Mechanism for Peak at 104.1



Due to the simple nature of the molecule, it is likely that fragmentation occurred with increasing amounts of carbons and hydrogens breaking off. Figure 10 shows a possible mechanism for the loss of a fragment of  $m/z$  28.1, which often corresponds to  $C_2H_4$ . The GC-MS shows that the molecular peak of the molecule is 132.2, corresponding to its molecular mass, and that the base peak, or most common fragmentation, occurs for the  $C_2H_4$  fragment.

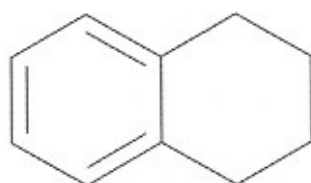
Figure 11: Literature Mass Spectrum of Tetralin<sup>(4)</sup>



The literature mass spectrum and experimental show the same pattern of fragmentation. The molecular peak is at about  $m/z$  132 and the base peak is at  $m/z$  104. Similar patterns of peaks can also be seen at  $m/z$  115 and  $m/z$  91.

### VIII. Structure and Discussion

Figure 12: Insanity Wolf a.k.a. Tetralin Structure



CORRECT!  
Good job.

The compound Insanity Wolf was determined to be tetralin, or 1,2,3,4-tetrahydronaphthalene, which has a molecular formula of  $C_{10}H_{12}$  and a molecular weight of about 132g/mol. This compound has a boiling point of 200-209°C<sup>[6]</sup>, which is close to the recorded boiling point of 212°C (Section I). The melting point of the substance is -30 °C<sup>[6]</sup>, which is well below room temperature and, as such the compound is a liquid, which was also true of the experimental substance.

The first indication that the compound was non-derivatizable was due to the solubility test. The compound precipitated a red substance when treated  $H_2SO_4$  rather than truly mixing, indicating an alkane, alkyl halide, or aromatic compound (Figure 1). The color of the precipitate was reddish-brown, which matches the color which occurs when naphthalene compounds are treated with  $H_2SO_4$ <sup>[2]</sup>. The compound's inability to dissolve in either HCl or NaOH indicates that it is not an acid or a base (Figure 1). Furthermore, the compound tested negative for the DNP test, Beilstein test, and both unsaturation tests, indicating that there was no carbonyl, halide, or unsaturation in the form of a double or triple bond in the compound (Section III). The ignition test also failed, and indicated a lack of aromaticity, but that was likely due to the high flammability of the compound.

The IR spectrum of the compound also gave important clues about the structure of the compound. The lack of anything but aromatic C=C and C-H, along with aliphatic C-H peaks indicated

that there was no nitro group, cyano group, alcohol, carbonyl, or amine in the compound. This essentially meant that the only elements in the compound were carbon and hydrogen, and the compound had a benzene ring.

The GC-MS indicated that the  $m/z$  of the compound was 132.2 (*Table 4*). Since there was no alcohol, carboxylic acid, and amine in the compound, as indicated by IR and various classification tests, this molecular weight was likely correct and not missing hydrogens or any fragments. If the weight of six carbons, or a benzene ring's carbons, are removed from the mass, there is only 60g left, which is equivalent to five carbons or four carbons and twelve hydrogens. As it would be impossible to have a compound with eleven carbons and absolutely nothing else, the molecular formula of the compound can be confirmed to  $C_{10}H_{12}$ .

This molecular formula signifies that the molecule has five degrees of unsaturation as calculated by the degree of unsaturation formula,  $\frac{2C+2+N-H-X}{2} = \text{DOS}$ . Since a benzene ring leads to four degrees of unsaturation, and there are no double bonds in the molecule as indicated by the negative results on the unsaturation tests (*Section III*), the molecule must create a ring structure. For the benzene ring and the new ring to be attached, there must be a nonaromatic ring of  $C_4H_8$  attached to the benzene ring. The substitution pattern can be determined from the IR spectrum. For an ortho-substituted compound there is one peak at  $\sim 750 \text{ cm}^{-1}$ , for a meta-substituted compound there are three peaks at  $\sim 850\text{-}900 \text{ cm}^{-1}$ ,  $\sim 750\text{-}800 \text{ cm}^{-1}$ , and  $\sim 700 \text{ cm}^{-1}$ , and a para-substituted compound has a single peak at  $\sim 800\text{-}850 \text{ cm}^{-1}$  [3]. The IR spectrum of Insanity Wolf shows only a peak at 742.1, indicating that the compound is ortho-substituted (*Table 2*). ? usually I call it IHD.

As such, the compound was determined to be tetralin, and the collected experimental data support the claim. Furthermore, collected literature spectra show close matches to the experimental spectra collected for tetralin. This dual confirmation by literature spectra and collected information from classification tests verify that the unknown Insanity Wolf is actually the compound tetralin.

### Works Cited

- <sup>[1]</sup> Armarego, W.L.F.; Christina, C. Tetralin (1,2,3,4-tetrahydronaphthalene). Purification of Organic Chemicals – Organic Compounds. In *Purification of Laboratory Chemicals*. Butterworth-Heinemann: Burlington, MA, 2009; pp 340.
- <sup>[2]</sup> Schroeter, Georg (Newport Manufacturing Company). Purification of naphthalene. US Patent 1763410, Jun 10, 1930.
- <sup>[3]</sup> Pavia, D. L.; Kriz, G. S.; Lampman, G. M.; Engel, R. G. *A Microscale Approach to Organic Laboratory Techniques*; Cengage Learning: Boston, 2018.
- <sup>[4]</sup> Spectral Database for Organic Compounds SDBS. SDBS No.: 907.  
<https://sdb.sdb.aist.go.jp/sdb/cgi-bin/landingpage?sdbno=907> (accessed May 30, 2019).
- <sup>[5]</sup> King, A. Wake Forest University. Table of Common Fragment Ions.  
<https://users.wfu.edu/~kingag/223L/procedures-handouts/common%20MS%20fragment%20ions.pdf> (accessed May 30, 2019).
- <sup>[6]</sup> Sigma-Aldrich. 1,2,3,4-Tetrahydronaphthalene.  
<https://www.sigmaaldrich.com/catalog/product/sial/522651> (accessed May 30, 2019).

Awesome job! Great start. Nice spectra & analysis. -*WJ*

Grade: A

**Purpose** Determine unknown organic compound using chemical classification, spectroscopy, and preparation of derivatives

**IR**

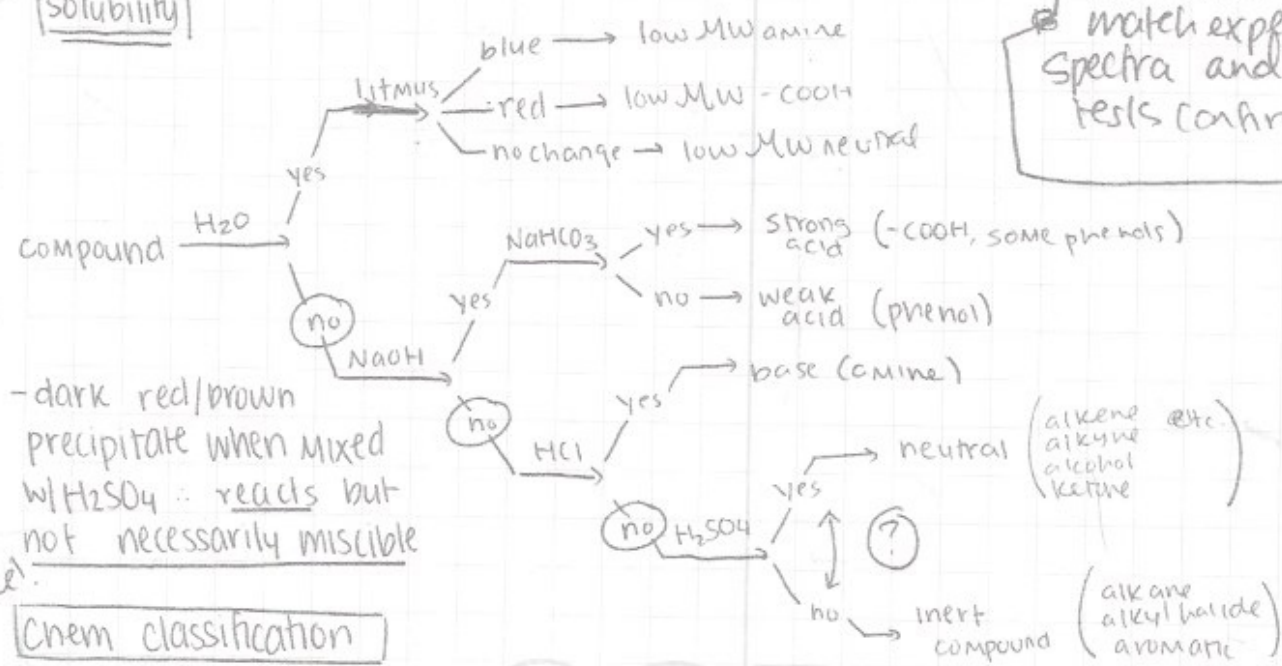
- no 2250 cm<sup>-1</sup> C≡N
- no 2250-2100 cm<sup>-1</sup> C≡C stretch
- no 742 cm<sup>-1</sup> = benzene
- 2928 cm<sup>-1</sup> 2857.1 cm<sup>-1</sup> C-H stretch
- no OH (3600-3200 cm<sup>-1</sup>)
- no NH (3500-3300 cm<sup>-1</sup>)
- no C=O 1850-1630 (too small)

✓ OK

looks like only benzene & C-H

**Conclusion:** The unknown "Insanity Wolf" is tetralin. Literature spectra match experimental spectra and experimental tests confirm

**Solubility**



-dark red/brown precipitate when mixed w/ H<sub>2</sub>SO<sub>4</sub>: reacts but not necessarily miscible

flammable!

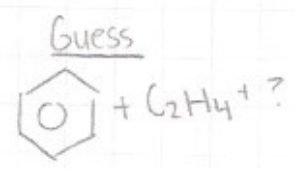
**Chem classification**

- ignition (aromatic) - (-) but inconclusive (IR, NMR)
- DNP (carbonyl) - (-)
- unsaturation (Br<sub>2</sub>/methylene chloride, KMnO<sub>4</sub>) - both (-) → Br<sub>2</sub> alone w/ (+), (-) control
- Failed Beilstein = no halide

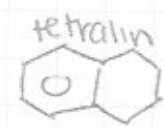
**Physical + GCMS**

B.P.: 212°C  
[M<sup>+</sup>]: 132  
base peak: 104.1 } M-28 → C<sub>2</sub>H<sub>4</sub>

NMR δ	m	I
1.778	g	~1
2.750	g	~1
7.050	s?	~1



132 - 72 = 60 →  $\frac{60}{12} = 5$ ? but not 5 so probably 4 → C<sub>10</sub>H<sub>12</sub>  
 $D_{0s} = \frac{20 + 2 - 12}{2} = 5$  (three from benzene) → no double bond so



Unknown Compound Report: Grumpy Cat a.k.a. 1-Butylamine ✓

CORRECT!

I. Physical Properties

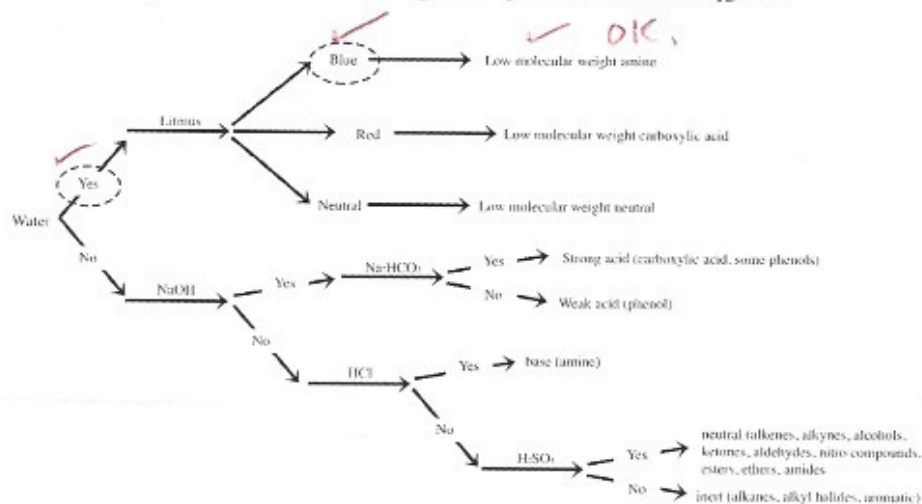
	Experimental	Theoretical <sup>[1][2]</sup>
Phase	Liquid ✓	Liquid *
Melting Point	N/A - Liquid so < 25°C	-49 °C
Boiling Point	75 °C ✓	78 °C *
Color	Clear with dull yellow tinge ✓	Colorless; turns yellow on standing
Smell	Fishy ✓	Fishy, ammonia-like odor
Vapor Pressure	Less than atmospheric pressure - it fumes	68 mmHg (20°C)

Table 1: Physical Properties

The compound is a volatile liquid with a propensity to fume whenever uncapped. The inside of the bottle shows condensation, and, at the time of this report, the compound is clear with a dull yellow tinge, which matches the theoretical explanation that it turns yellow when exposed to air. Melting point data is unavailable experimentally due to the liquid nature of the compound, but the boiling point was measured to be 75 °C, which has only a percent error of 3.87% from the theoretical compound. Good!

II. Solubility

Figure 1: Annotated Solubility Chart for Unknown Grumpy Cat





The solubility data for Grumpy Cat suggests that it is a low molecular weight amine. When it is mixed with water and a litmus paper held over the fumes, the litmus paper slowly turns blue, as if spattered with spray paint. This supports the idea that the compound has a low vapor pressure.

### III. Classification Tests

Test	Tests For	Observations	Positive or Negative
Beilstein	Halide	No green flame, but compound is highly flammable.	negative ✓
Bromine – Methylene Chloride	Unsaturation	Solution remained clear but created a white gas - HBr <i>Yes.</i>	negative ✓
Ignition	Aromaticity	No soot or yellow color, but compound is highly flammable.	negative ✓
2,4-Dinitrophenylhydrazine (DNP)	Aldehydes/Ketones	No orange precipitate, very different from positive control	negative ✓
pH of an aqueous solution	Amine	pH is basic	positive ✓
Cerium (IV) test	Alcohol	Red/white precipitate formed, yellow-orange liquid did not turn red – different from positive control	negative ✓

Table 2: Classification Test Results

This compound failed almost every classification test, but often in ways that tended to create fumes or gas. It did not fail the pH of an aqueous solution test, which indicated that it was an amine. The nitrous acid test was not done since the compound's volatility made classification tests difficult and the positive result of nitrogen gas bubbles may not have been noticeable anyway. Both flame tests showed that the compound was highly flammable and suggested that there were no halides or aromaticity in the compound. While they could have been false negatives, the IR spectrum indicates that that the test results were correct.

When the compound was subjected to the Br<sub>2</sub>/DCM test, the solution remained clear but a white gas was also evolved. When hydrogen bromide is created, the reaction is likely a substitution reaction, rather than an addition reaction with a double or triple bond, and there is likely no unsaturation. The negative DNP test also indicated that there are no aldehydes or ketones in the

substance. The cerium (IV) test for alcohols was done due to the broad  $>3000\text{cm}^{-1}$  peak in the IR spectrum, but the precipitate formed was indicative of an amine, rather than an alcohol.

#### IV. IR Spectrum

Figure 2: Experimental IR Spectrum

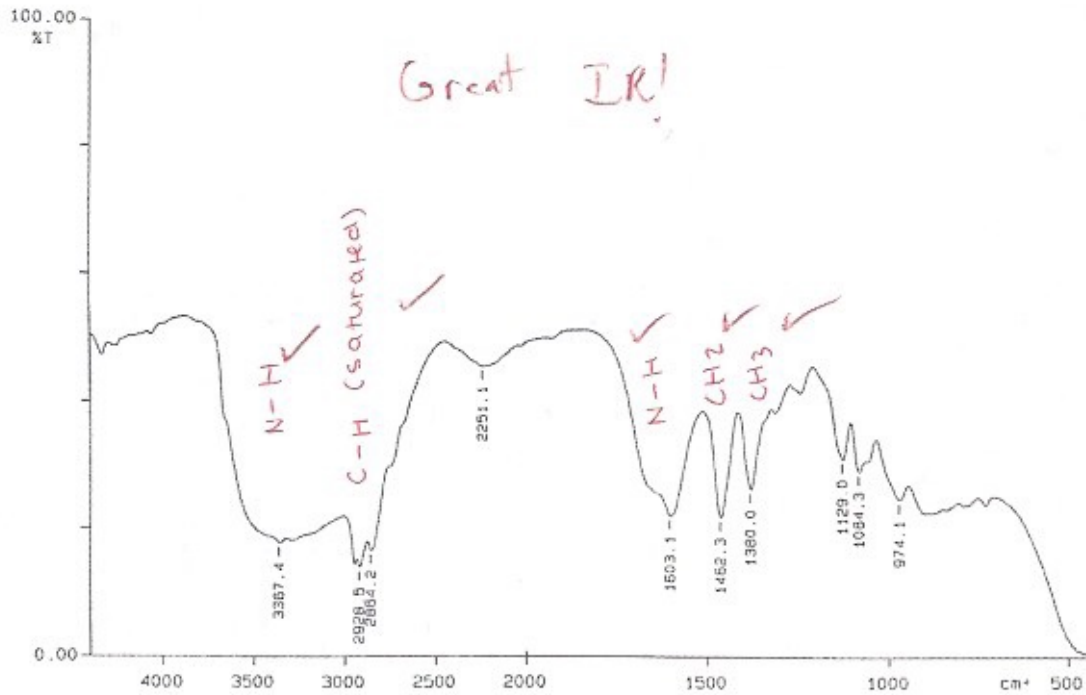


Figure 3: Literature Spectrum 1<sup>1</sup>

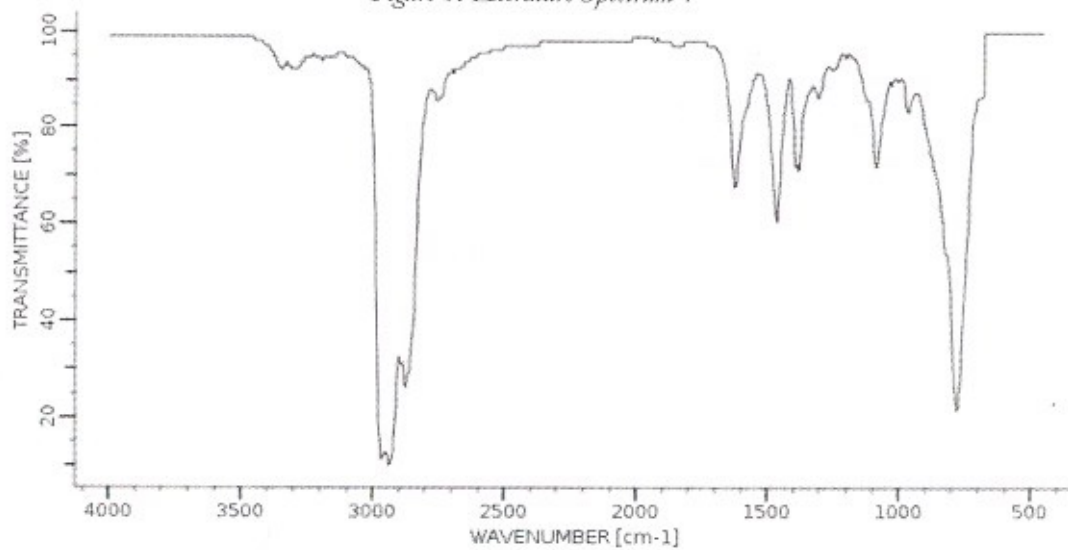


Figure 4: Literature Spectrum 2<sup>5</sup>

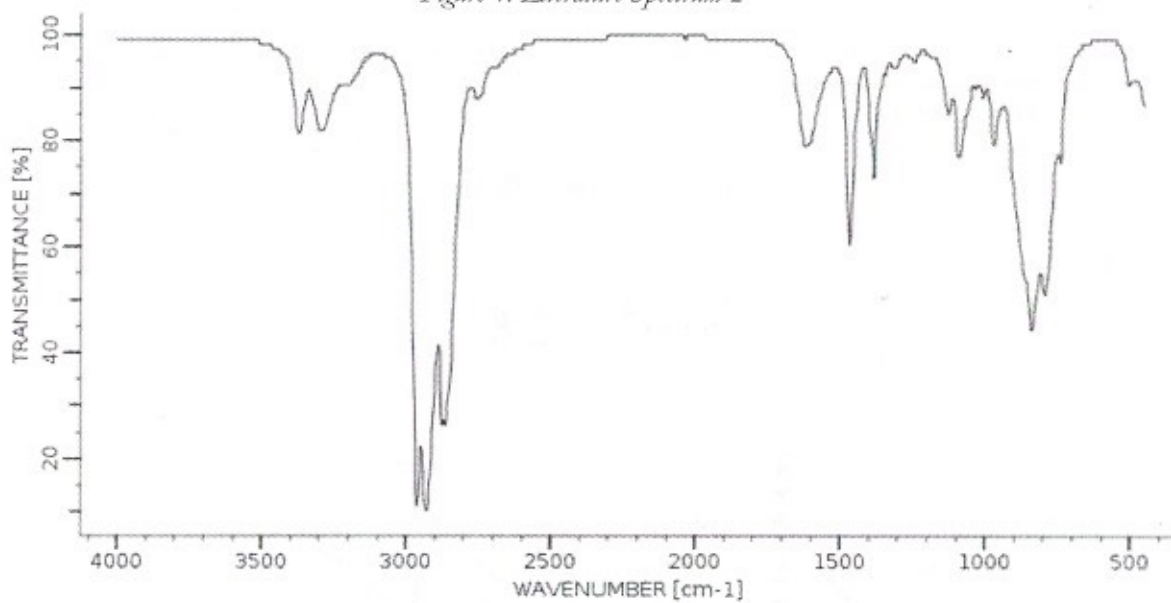
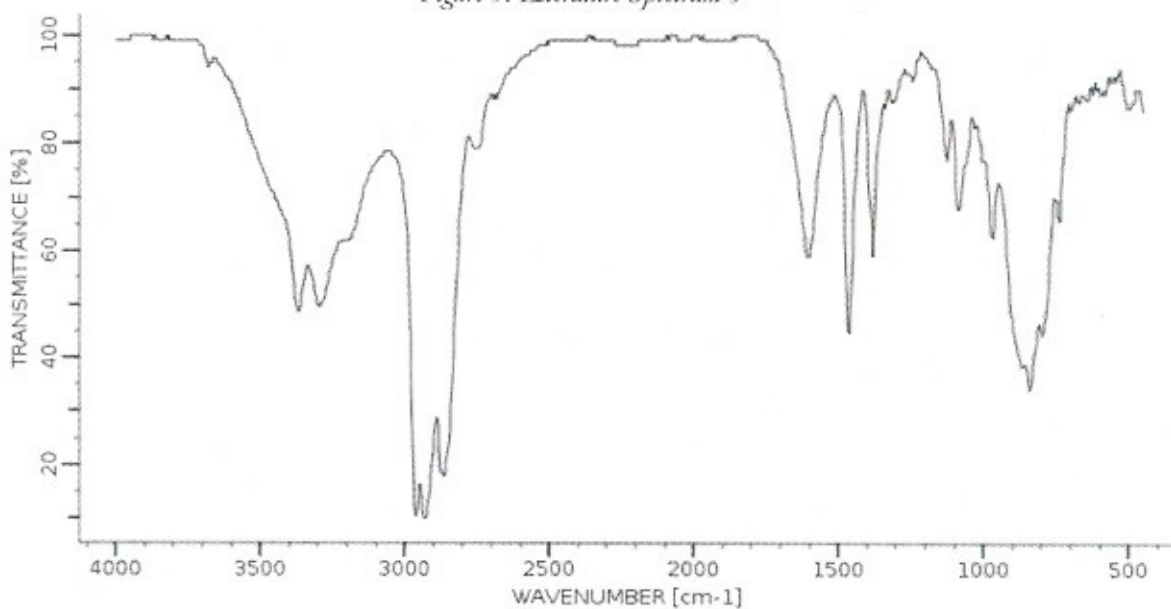


Figure 5: Literature Spectrum 3<sup>6</sup>



Functional Group	Expected Peak (cm <sup>-1</sup> ) <sup>[1][4]</sup>	Observed Peak
C=O	1725–1685 (aldehyde) – often 1725 1780–1665 (ketone) – often 1715 1725-1690 (carboxylic acid) – broad ~1735 (ester)	---
O-H (alcohol)	3600–3200 (alcohol) 3300-2500 (acid)	---
C=C	1680-1620 (aliphatic) 1600–1450 (aromatic, often four sharp absorptions that occur in pairs near 1600 cm <sup>-1</sup> and 1450 cm <sup>-1</sup> )	---
C≡C	2250-2100 (sharp) None in symmetrical alkynes	---
C≡N	~2250	---
✓ C-H (saturated)	3000-2850 (alkane, stretch) 1465 (CH <sub>2</sub> , bend) 1450, 1375 (CH <sub>3</sub> , bend)	2928.5, 2864.2 1462.3 1380.0
C-H (unsaturated)	3310-3200 (alkyne terminal) 3150-3000 (vinyl stretch, alkene) 1000-700 (out of plane, alkene) >3000 (stretch, aromatic) 900-690 (out of plane, aromatic), often ~700	---
✓ N-H	3500-3300 (two peaks for aliphatic and primary) 1640-1560 (primary) 1580-1490 (secondary) 1600-1450 (aromatic)	3367.4 (broad) 1603.1
NO <sub>2</sub>	1560, 1350 (strong)	---
C-X	C—F Stretch, 1350–960 (C-F) 850–500 (C-Cl) right of 667 (C-Br, C-I)	---
C-O	1300-1000 (ether) 1730-1700 (acid) 1320-1210 (acid, strong) 1700-1640 (amide) 1830–1800 & 1775–1740 (anhydride)	---

Table 3: IR Spectrum Analysis

The IR spectrum for this compound is almost empty – indicating nothing more than saturated C-H bonds and a primary amine. While the broad stretch at 3367.4cm<sup>-1</sup> could be indicative of an alcohol, the cerium (IV) test indicates that that is not the case. Instead, it is more likely that the broad stretch is due to free N-H bonds and hydrogen bonding, which is usually what makes the alcohol peak broad. Although nitrogen is less electronegative than oxygen and, as such, forms weaker hydrogen

bonds, the neat form of amine and its high concentration allows for many hydrogen bonds and, as such, the broad peak at  $>3000\text{cm}^{-1}$ . Furthermore, the peak at  $1603\text{cm}^{-1}$  is suggestive of a primary aliphatic amine.

In the literature spectrum of 1-butylamine, the peak with two fangs at  $>3000\text{cm}^{-1}$  goes from almost not present (Figure 3), to somewhat present (Figure 4), to somewhat present with a broad peak (Figure 5). These are official literature spectra and should be extremely clean, However, it appears that the volatility of the compound leads to variable peaks in that region and my spectra is likely a step past Figure 5 in terms of how broad that peak is and covers up the two peaks meant for an aliphatic, primary amine. Beyond those two peaks, the experimental spectrum is almost identical to the literature spectra. It is missing a peak at  $800\text{cm}^{-1}$  but that is likely because the machine became saturated at that time point and the peak is covered by the transmittance going to zero.

### V. $^1\text{H}$ NMR Spectrum

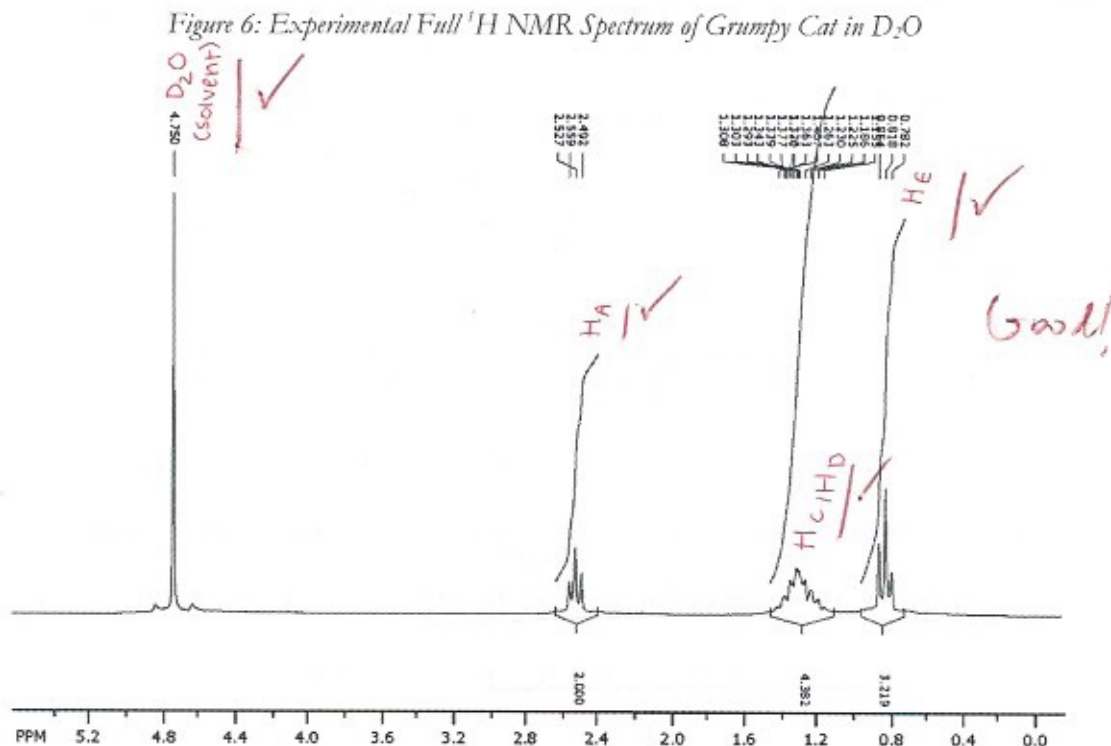


Figure 7: Experimental  $^1\text{H}$  NMR Spectrum of Grumpy Cat in  $\text{D}_2\text{O}$ : Expanded View

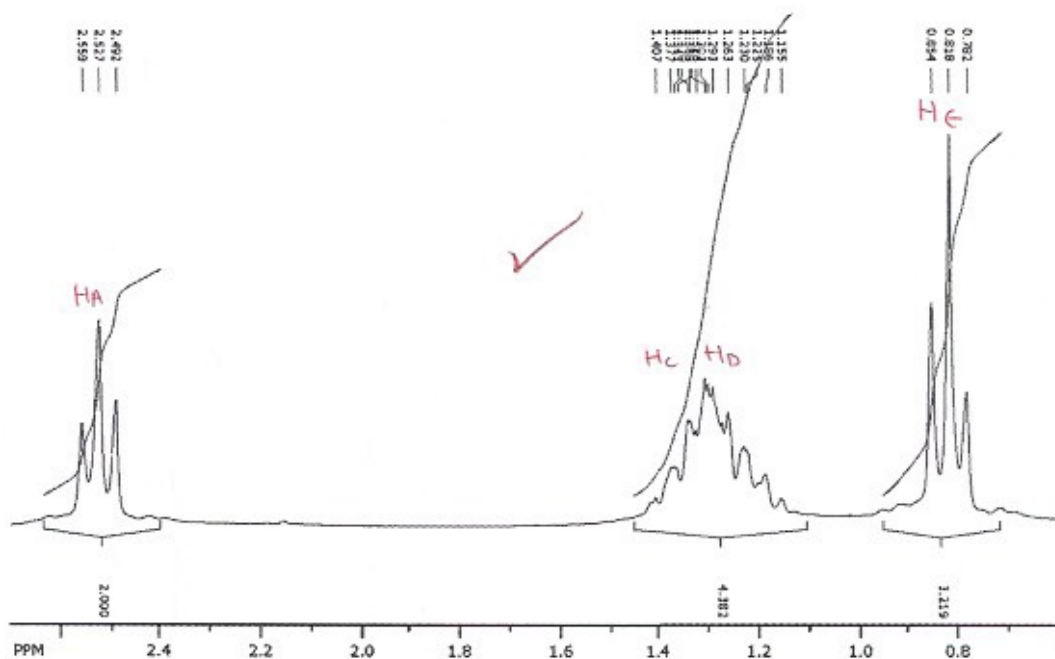


Figure 8: Literature 90 MHz  $^1\text{H}$  NMR Spectrum of 1-Butylamine in  $\text{CDCl}_3$  (left) Hydrogen Assignments (right)

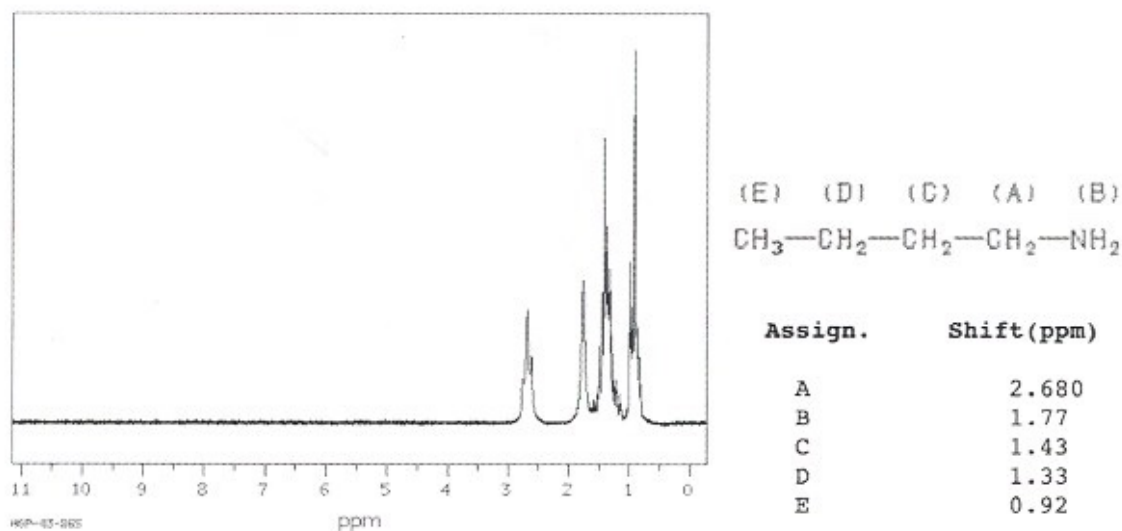


Figure 9: Literature 300MHz  $^1\text{H}$  NMR Spectrum of 1-Butylamine in  $\text{CDCl}_3$

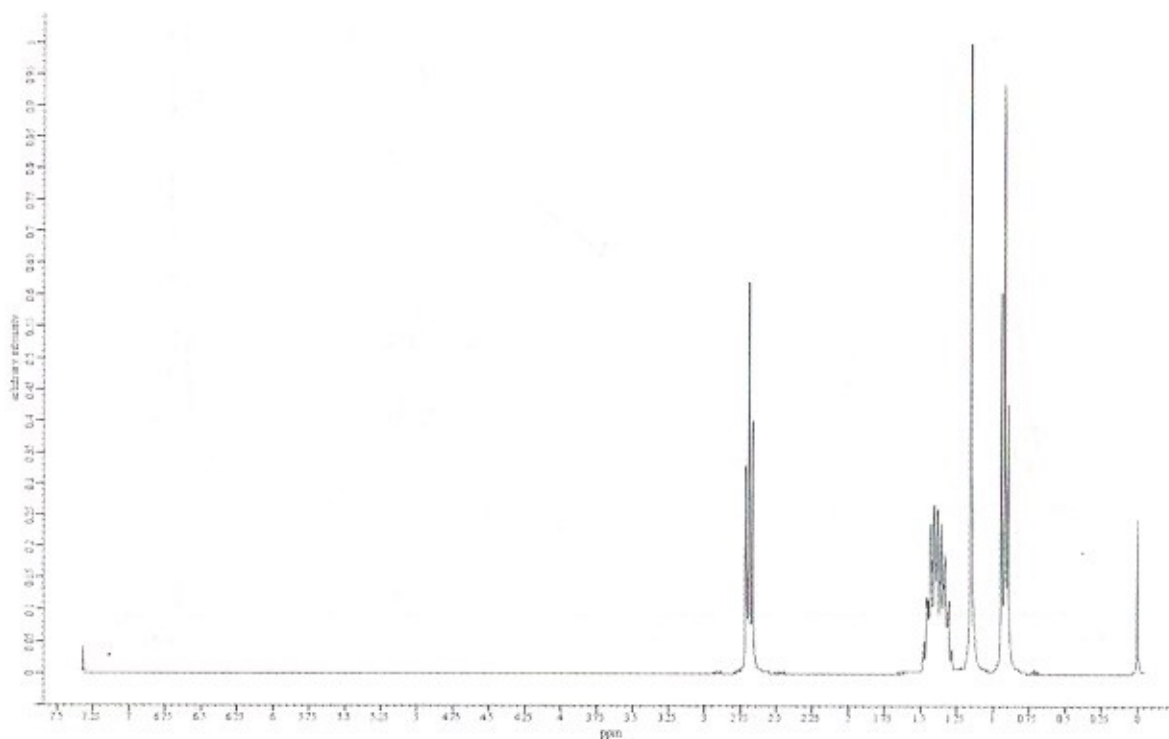
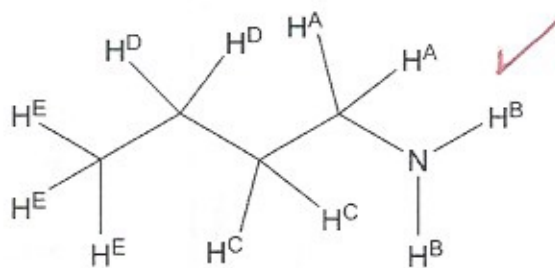


Figure 10: Hydrogen Assignments for 1-Butylamine



	Experimental				Literature <sup>[8]</sup>		Assignment
	Chemical Shift(ppm)	Integration	Multiplicity	J-Value (Hz)	Chemical Shift(ppm)	Multiplicity	
Compound	2.527	2.000	t	$J_{AC}=9$	2.680	t	H <sup>A</sup>
	-	-	-	-	1.77	s	H <sup>B</sup>
	1.293	4.382	m	-	1.43	m	H <sup>C</sup>
					1.33		H <sup>D</sup>
0.818	3.219	t	$J_{ED}=8.75$	0.92	t	H <sup>E</sup>	
Solvent	4.750	-	s	-	-	-	D <sub>2</sub> O <sup>[9]</sup>

Table 4: NMR Spectrum Analysis

The NMR spectrum for the experimental Grumpy Cat sample and literature 1-butylamine match closely, although the 90MHz NMR spectrum from SBDS (Figure 8) is not particularly well resolved. H<sup>A</sup> is  $\alpha$  to nitrogen, and so has a shift from 2-3ppm<sup>[10]</sup>. The shift found in the NMR spectrum at 2.527ppm integrates to 2.000, which is correct. At 1.293ppm there is a multiplet integrating for 4.382 hydrogens which is likely the overlapping signals for four hydrogens of H<sup>C</sup> and H<sup>D</sup>. H<sup>C</sup> is split by the other H<sup>C</sup> hydrogen, H<sup>D</sup>, and H<sup>A</sup> into what is likely a doublet of a doublet of a doublet, while H<sup>D</sup> is split by the other H<sup>D</sup> hydrogen, H<sup>C</sup>, and H<sup>E</sup> into what is likely a doublet of a doublet of a triplet. These peaks overlap and create the messy peak seen at 1.293ppm. At 0.818ppm is a triplet which integrates to approximately 3 hydrogens and is due to H<sup>E</sup>. Furthest away from the amine, it is the most shielded.

The only discrepancy is for the H<sup>B</sup> hydrogen. The literature spectrum also has a singlet for H<sup>B</sup> but, due to the experimental spectrum using D<sub>2</sub>O as a solvent, the peak for the amine hydrogens is absent since the hydrogens exchange in the solvent.

## VI. Derivative

Amount Compound (g)	Amount 0.1 M HCl (mL)	Observations	NE (g/mol)	Error for NE
0.230	30.1	When the parafilm was removed from the top, there was liquid on the parafilm. pH paper held above the flask turned blue	76.41	4.47%

Table 5: Neutralization Equivalent Data



For the derivative test, the neutralization equivalent test was used. The neutralization equivalence is usually calculated for carboxylic acids, but essentially the neutralization equivalent is used to either determine the molar mass of a compound per each mole of the functional group that reacts. Since 1-butylamine is a primary amine, 0.230g was dissolved in about 50mL of deionized water, and then the solution was titrated with 0.1M HCl. The equation for the neutralization equivalent for a base is as follows:

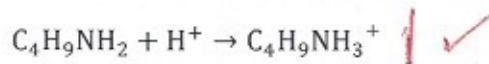
$$\frac{\text{Neutralization equivalent}}{\# \text{ amine groups}} = \frac{\text{mg base compound}}{(\text{molarity of HCl}) \times (\text{mL of HCl})}$$

This equation can also be written as:

$$\frac{\text{Neutralization equivalent}}{\# \text{ amine groups}} = \frac{\text{g base compound}}{\text{moles of HCl to neutralize}}$$

$$\therefore \text{Neutralization equivalent} = \frac{(\# \text{ amine groups})(\text{g base compound})}{\text{moles of HCl to neutralize}}$$

Logically it can be understood that if the compound only has one amine group per mole of compound, then the number of moles of HCl required to protonate it is equal to the number of moles of compound. This can also be seen in the neutralization equation below:



As such, the equation can be rewritten as:

$$\text{Neutralization equivalent} = \text{Molar weight of compound} = \frac{\text{mg base compound}}{(\text{molarity of HCl}) \times (\text{mL of HCl})} \quad | \quad \checkmark$$

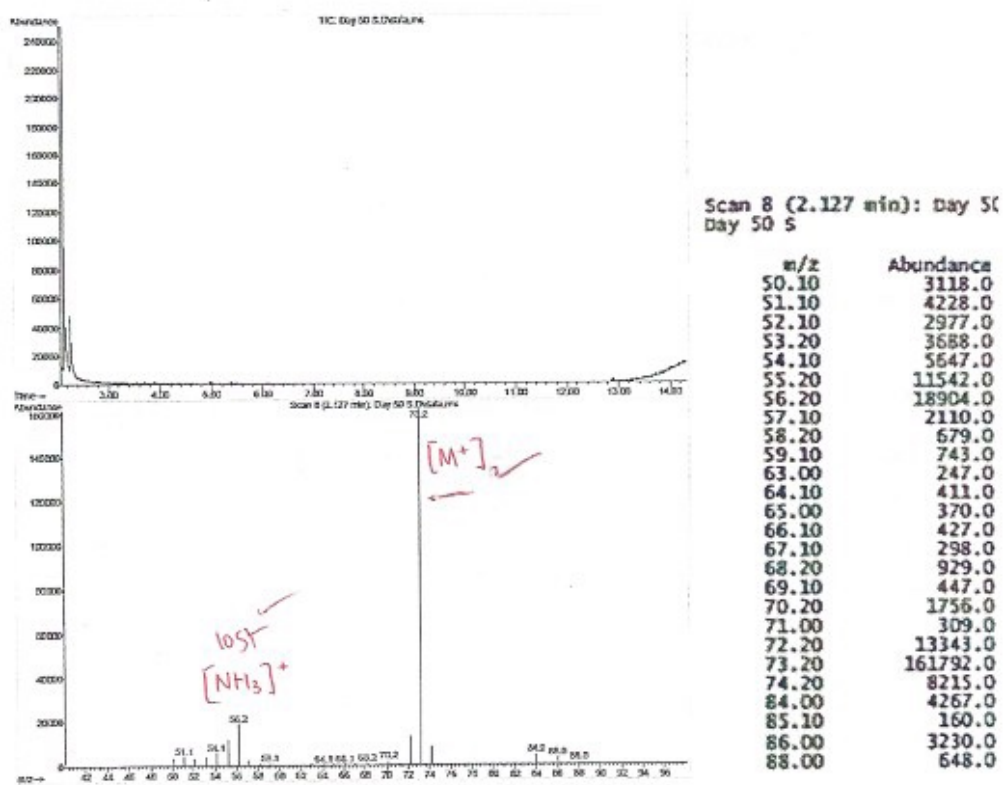
Ideally, since the molecular mass of the compound is known to be 73.14 g/mol, it can be calculated that 31.45mL of 0.1 M HCl should be added to 0.230g of compound to neutralize it. The amount actually used experimentally was 30.1mL, which indicated a neutralization equivalent of 76.41 g/mol, which is 4.47% lower than it should ideally have been. This likely occurred due to the volatility of the compound.

The compound evaporated as it was being titrated, and less of the compound was titrated than weighed out, even when parafilm was used to cover the top of the compound-containing Erlenmeyer flask. Since the equivalence point of a weak base titrated with a strong acid is usually slightly less than 7, bromothymol blue, which has a pH range of 6.0 - 7.6 for indication<sup>[1]</sup>, was used as a pH indicator. Methyl red, with a pH range of 4.8-6.0 might have been a better indicator for equivalence, but it was not available.

However, the neutralization equivalence test confirmed the molecular weight of the compound, since it was already known that there was only one amine group and the test was used to confirm the compound.

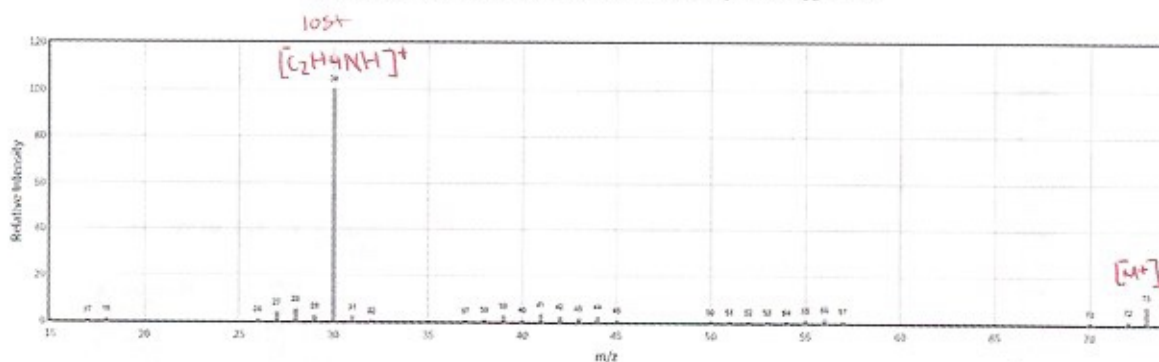
## VII. GC-MS

Figure 11: Initial Experimentally Recorded GC-MS of Grumpy Cat (left) Percentage Report (right)



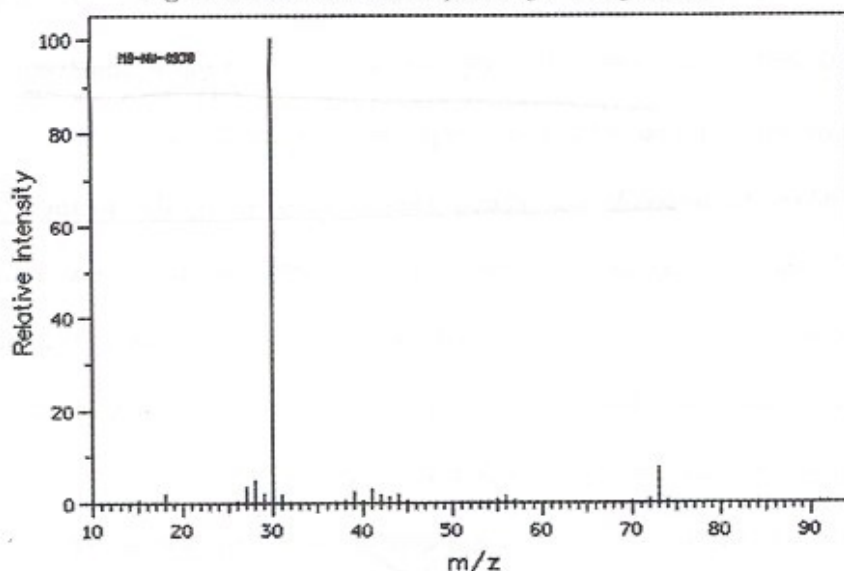
From the initially received GC-MS spectrum, it appeared that the mass peak for the molecule was at  $m/z$  73.2 and the base peak was at  $m/z$  56.2. The odd mass value indicated the presence of a nitrogen but there were no recorded peaks below  $m/z$  50.10. However, information from IR spectrum suggested that the molecule was a primary amine with no other functional groups, and, literature searches indicated that all primary amines with a molecular formula of  $C_4H_{11}N$  had a base peak at around  $m/z$  30. Even if the peaks were cut off at  $m/z$  50, only isobutylamine and butylamine had a peak at  $m/z$  56. Most had peaks at  $m/z$  58 and peaks occurred in the  $m/z$  40 range as well. Furthermore, for the spectrum, the maximum abundance was at less than a minute and only 8 scans were taken overall. Most of the other spectra taken had between 500 and 1000 scans. All of this was odd and pointed to an inaccurate mass spectrum in terms of fragmentation.

Figure 12: Literature Spectrum Provided of Grumpy Cat



Peak (m/z)	Fragment Mass	Assignment
73	-	$[M^+]$
56	<del>17</del>	$NH_3$
41	32	$CH_3NH_2$
30	43	$C_2H_4NH$

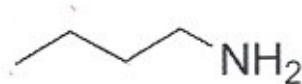
Table 6: Mass Spectrum Analysis

Figure 13: Literature Mass Spectrum for 1-Butylamine<sup>[2]</sup>

The new provided spectrum provided a more accurate depiction of the molecule and showed a base peak at  $m/z$  30, as expected. This base peak occurred due to  $C_2H_5NH$  breaking off from the molecule. Due to the ease with which a nitrogen could pick up or lose a hydrogen during electron mass spectrometry, other fragments likely occurred due to fragmentation by the nitrogen with some carbons.

### VIII. Structure and Discussion

Figure 14: Grumpy Cat a.k.a. 1-Butylamine Structure

Chemical Formula:  $C_4H_{11}N$ 

Exact Mass: 73.09

Molecular Weight: 73.14

The compound Grumpy Cat was determined to be 1-Butylamine, which has a molecular weight of about 73g/mol and a molecular formula of  $C_4H_{11}N$ . The compound had an experimental boiling point of 75°C, which is close to the theoretical boiling point of 78°C. It was a liquid, which was supported by the theoretical melting point of -49 °C. Furthermore, its ability to fume was

Low vapor pressure means  
↑ less volatile.

Irika Sinha 14  
CHEM 347 AA  
Unknown Compound: Grumpy Cat

supported by the low vapor pressure of 1-butylamine. 68 mmHg is lower than the atmospheric pressure in Seattle and, as such, allows the compound to vaporize at room temperature. ✓

Some difficulties with identifying the compound occurred due to the common occurrence of a gas forming when the molecule was treated with anything. Likely due to the volatility of the compound, it also did not successfully pass through the GC-MS machine for any significant amount of time. The mass spectrum accurately indicated the molecular peak at  $m/z$  73.2 but inaccurately indicated the rest of the peaks. This mass was used to confirm the existence of only one amine by using the neutralization equivalence test as a derivatization.

The initial solubility test about this compound was useful and immediately suggested that the compound was a low molecular weight organic base and, therefore, an amine. The IR indicated a lack of anything but an amine, although the broad peak at above  $3000\text{cm}^{-1}$  suggested a possible alcohol group in the molecule. However, the cerium (IV) test ruled out the probability of an alcohol in the solution, and the IR only indicates an aliphatic amine beyond that.

The IR spectrum and classification tests confirmed an aliphatic amine with no other functional groups. As such, a molecular weight of 73 g/mol indicated  $\text{C}_4\text{H}_{11}\text{N}$ , which has a few different permutations. The NMR spectrum indicated two sets of hydrogens with two neighbors, due to the triplets formed. The roofing effect indicated that they were either split by the messy quintet hydrogens or the other set of hydrogens. The structure of 1-butylamine indicates that the triplet hydrogens were split by the hydrogens in between them. The NMR spectrum peaks and integrations are correct for 1-Butylamine. Due to the solvent  $\text{D}_2\text{O}$ , the nitrogen hydrogens were exchanged and do not show a peak in the  $^1\text{H}$  NMR spectrum.

As such the unknown compound "Grumpy Cat" was determined to be 1-butylamine. Collected experimental data supported the claim and the literature spectra match what was collected, even though the compound reacts with air and changes color and, possibly, composition. This dual

confirmation by literature spectra and classification tests verify that the unknown Grumpy Cat is actually the compound 1-butylamine. ✓

Grade: A

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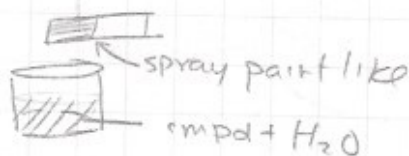
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EXP. NUMBER	EXPERIMENT/SUBJECT Unknown: Grumpy Cat	DATE
NAME Irika Sinha	LAB PARTNER	COURSE & SECTION NO. CHEM 337
		LOCKER/DESK NO. 8177

Purpose: Determine identity of compound Grumpy Cat

Physical: liq, bp:  $75^{\circ}\text{C}$ , clear + yellow tinge, fishy strong smell, FUMES

Solubility: dissolves in  $\text{H}_2\text{O}$   $\rightarrow$  litmus blue



class.

Beilstein: (-)  
 Br<sub>2</sub>/DCM: (-) gas evolved  
 Ignition: (-) very flammable  
 DNP: (-)  
 PhAg soln - blue litmus (+)  
 cerium (IV) - (-)

IR: broad  $>3000$ ,  $\text{CH}$ ,  $1603, 1462, 3, 1380, 3$



NMR: 2.527 (t), 1.263 (messy g-ish), 0.818 (t)

Derivative:  
 bromo thymol blue - 30.1, 0.230g  
~~bromo~~ thymol blue - 0.20g  $\rightarrow$  26.2, 24  
 - para film collects liq.



GC/MS:  $[M^+] = 73$  base peak: 30

Conclusion: Grumpy Cat is 1-butylamine

COPY

SIGNATURE	DATE	WITNESS/TA	DATE
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