

# Multistep Synthesis of Fluorene Derivatives

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## Executive Summary

Starting with the tricyclic aromatic hydrocarbon, fluorene, a series of labs were performed over several weeks to obtain primary, secondary, and tertiary product derivatives. Specific derivatives include: an amine, an amide, an imine, an alkyl amine, an iodo, an oxidation, and a Suzuki. Each product was verified with melting point, TLC, and IR spectroscopy. When available, literature experimental values were compared to values obtained. Instructor provided NMR spectra of the target products were analyzed, but not used to verify product identity. Overall product yields are provided in [Annex 2](#) near the end of this report.

## Acknowledgments

Very seldom is a large project accomplished solely on one's own merits. Therefore, great appreciation is extended to fellow student, Kassandra Willoughby, for the numerous small helps that made these experiments run more smoothly. Also (*of course!*) to the excellent mentorship of the supervising instructor.

**Supervising Instructor:** Chad Stephens  
*College of Math and Sciences, Augusta University*



# Table of Contents

<b>Section</b>	<b>Page</b>
<b>Synthesis of 2-Nitrofluorene from Fluorene</b> .....	<b>7</b>
Summary	7
Experimental Methods and Observations	7
Discussion and Results	7
Conclusion	8
Appendix A: Final Product Photographs	9
Appendix B: IR Spectra	10
Appendix C: Proton NMR Spectra	11
Appendix D: Experiment Laboratory Notes	13
<b>Synthesis of 2-Nitrofluorenone from 2-Nitrofluorene</b> .....	<b>15</b>
Summary	15
Experimental Methods and Observations	15
Discussion and Results	15
Conclusion	16
Appendix A: Final Product Photographs	17
Appendix B: IR Spectra	18
Appendix C: Carbon NMR Spectra	19
Appendix D: Experiment Laboratory Notes	21
<b>Synthesis of 2-Aminofluorene from 2-Nitrofluorene</b> .....	<b>23</b>
Summary	23
Experimental Methods and Observations	23
Discussion and Results	24
Conclusion	24
Appendix A: Final Product Photographs	25
Appendix B: IR Spectra	26
Appendix C: Proton NMR Spectra	27
Appendix D: Experiment Laboratory Notes	29
<b>Synthesis of Imine from 2-Aminofluorene</b> .....	<b>31</b>
Summary	31
Experimental Methods and Observations	31
Discussion and Results	31
Conclusion	32
Appendix A: Final Product Photographs	33
Appendix B: IR Spectra	34
Appendix C: Proton NMR Spectra	35
Appendix D: Mechanism	36
Appendix E: Experiment Laboratory Notes	37

Synthesis of Acetamide from 2-Aminofluorene .....	<b>39</b>
Summary	39
Experimental Methods and Observations	39
Discussion and Results	39
Conclusion	40
Appendix A: Crude Product Photographs	41
Appendix B: IR Spectra	42
Appendix C: Proton NMR Spectra	43
Appendix D: Mechanism	44
Appendix E: Experiment Laboratory Notes	45
Synthesis of Benzylamide from 2-Aminofluorene .....	<b>47</b>
Summary	47
Experimental Methods and Observations	47
Discussion and Results	47
Conclusion	48
Appendix A: Final Product Photographs	49
Appendix B: IR Spectra	50
Appendix C: Proton NMR Spectra	51
Appendix D: Fluorine NMR Spectra	52
Appendix E: Mechanism	53
Appendix F: Experiment Laboratory Notes	55
Synthesis of 2-Iodofluorene from 2-Aminofluorene .....	<b>57</b>
Summary	57
Experimental Methods and Observations	57
Discussion and Results	58
Conclusion	59
Appendix A: IR Spectra	60
Appendix B: Proton NMR Spectra	61
Appendix C: Carbon NMR Spectra	62
Appendix D: Experiment Laboratory Notes	63
Synthesis of Benzyl Amine from Imine .....	<b>65</b>
Summary	65
Experimental Methods and Observations	65
Discussion and Results	65
Conclusion	66
Appendix A: Final Product Photographs	67
Appendix B: IR Spectra	68
Appendix C: Proton NMR Spectra	69
Appendix D: Mechanism	70
Appendix E: Experiment Laboratory Notes	71

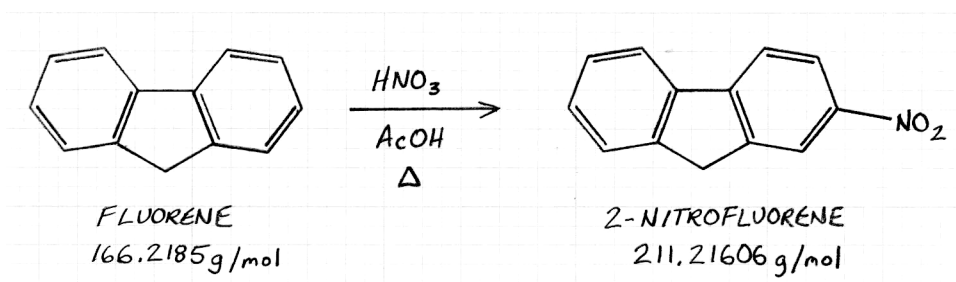
Synthesis of Suzuki Product from 2-Iodofluorene .....	<b>73</b>
Summary .....	73
Experimental Methods and Observations .....	73
Discussion and Results .....	73
Conclusion .....	74
Appendix A: Final Product Photographs .....	75
Appendix B: IR Spectra .....	76
Appendix C: Experiment Laboratory Notes .....	77
<i>Annex 1: General Chemical and Safety Data</i> .....	<b>81</b>
<i>Annex 2: Experimental Results Summary</i> .....	<b>91</b>
<i>Annex 3: General Laboratory Notes</i> .....	<b>93</b>



# Synthesis of 2-Nitrofluorene from Fluorene

## Summary

Fluorene was nitrated with nitric acid to produce 2-nitrofluorene.



**Fig 1:** Fluorene nitration reaction to yield 2-nitrofluorene

## Experimental Method and Observations

To an Erlenmeyer flask 3.0 g (18 mmol) of fluorene was added with 25 mL of acetic acid. The mixture was stirred in a hot water bath while temperature was raised to 70° C. Stirring continued while 4 mL of nitric acid was added. The temperature was increased to 80° C and held steady for 6 minutes. The mixture was removed from the bath.

The mixture was diluted with 19.8 mL of water and cooled to room temperature. The precipitate was vacuum filtered and washed with water. The filtrate was discarded.

The crude product was transferred to a clean beaker. Methanol was added and the crude was ground to small particles to remove impurities soluble in methanol. The crude was vacuum filtered and rinsed with methanol. Recrystallation was performed with isopropanol to yield final product.

The product was dried in open atmosphere for one full week. A yellow precipitate formed (*Appendix A: Final Product Photograph*). TLC was performed with 10% ethyl acetate in hexanes.

## Discussion and Results

Thin layer chromatography results depict a product that differs from the original. The  $R_f$  for fluorene was 0.90 while the  $R_f$  for 2-nitrofluorene was 0.38.

The melting point obtained 157.2 – 157.6° C agrees with experimental results 158° C for 2-nitrofluorene as reported in literature[2]. The range obtained suggests a purity better than 99%. The obtained value differs from the melting point of the reagent, fluorene, which is reported as 115 – 119° C in literature[1].

The IR spectrum of the product (*Appendix B: IR Spectra*) shows standard SP3 C–H and SP2 C–H peaks and standard benzene peaks. Two peaks of interest are displayed at the edge of the fingerprint region that can substantiate product identification: a possible N–O peak at wavenumber 1516.91, and a possible C–N peak at wavenumber 1332.57.

The proton NMR spectrum of the product (*Appendix C: Proton NMR Spectra*) shows a number of unremarkable peaks indicative of benzene rings. There is also a peak that accords with the central hydrogens between the two rings.

The molar quantity of the primary reagent fluorene ( $C_{13}H_{10}$ ) was:

$$mol\ C_{13}H_{10} = 3.0\ g\ C_{13}H_{10} * \frac{1\ mol\ C_{13}H_{10}}{166.2185\ g\ C_{13}H_{10}} = 0.018\ mol\ C_{13}H_{10}$$

The molar mass of the yielded product (2-nitrofluorene) is 211.21606 g per mole. The number of moles yielded is:

$$mol\ C_{13}H_9NO_2 = 2.338\ g\ C_{13}H_9NO_2\ (collected) * \frac{1\ mol\ C_{13}H_9NO_2}{211.21606\ g\ C_{13}H_9NO_2} = 0.011\ mol\ C_{13}H_9NO_2$$

The percent yield is:

$$\begin{aligned} \% - yield\ C_{13}H_9NO_2 &= 0.011\ mol\ C_{13}H_9NO_2 * \frac{1\ mol\ C_{13}H_{10}}{1\ mol\ C_{13}H_9NO_2} * \frac{1}{0.018\ mol\ C_{13}H_{10}} * 100\% \\ &= 61.5\% \end{aligned}$$

The yield is realistic for laboratory conditions.

## Conclusion

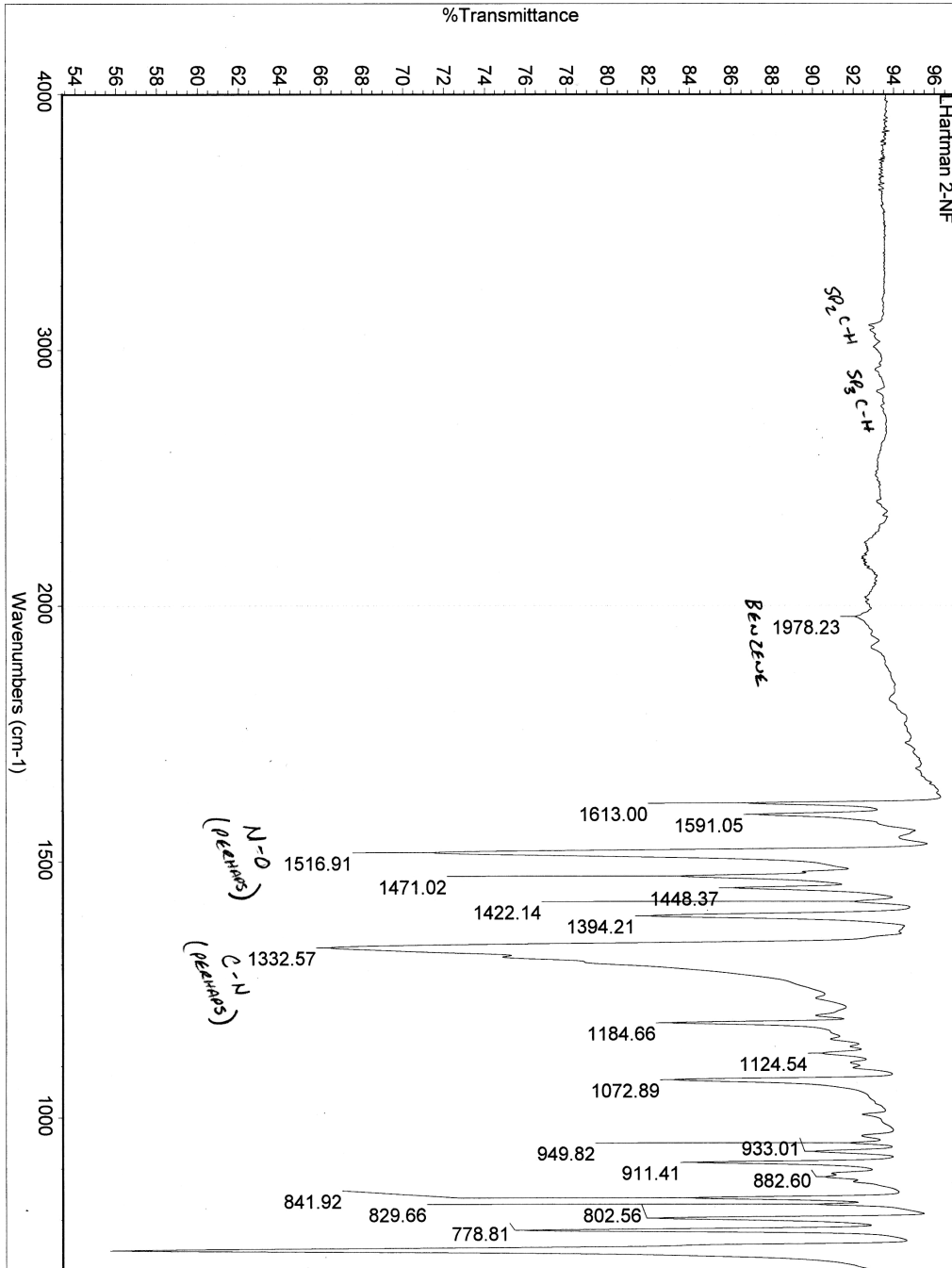
The product formed was 2-nitrofluorene, with a reasonable yield. Purity of excellent quality was confirmed by an obtained melting point range that encompassed the experimental value reported in literature[2]. IR analysis also confirms product identity.



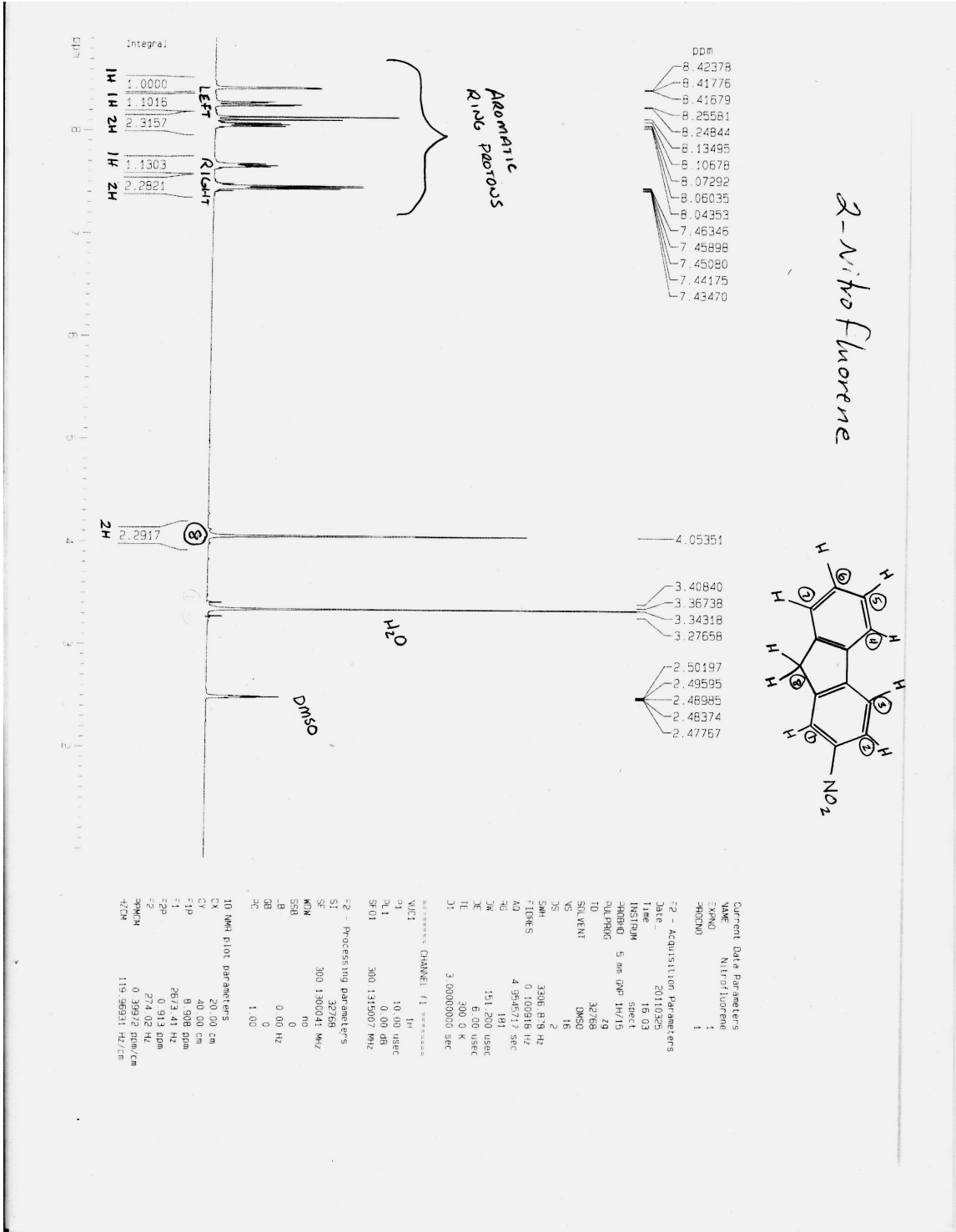
**Appendix A: Final Product Photograph**  
Source: Student Notes



Appendix B: IR Spectra  
Source: Student Notes



Appendix C: Proton NMR Spectra  
Source: Laboratory Instructor





**Appendix D: Experiment Laboratory Notes**  
Source: Student Notes



# Synthesis of 2-Nitrofluorenone from 2-Nitrofluorene

## Summary

The central CH<sub>2</sub> group on the 2-nitrofluorene was oxidized by oxygen in open atmosphere for one week to produce 2-nitrofluorenone.

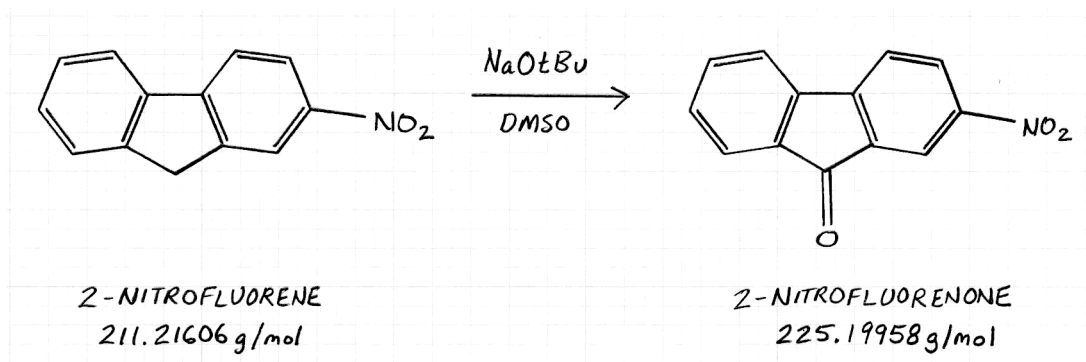


Fig 1: 2-Nitrofluorene oxidation reaction to yield 2-nitrofluorenone

## Experimental Method and Observations

To a 50 mL Erlenmeyer flask was added 0.158 g (0.748 mmol) of 2-nitrofluorene and 4.1 mL of dimethyl sulfoxide. The mixture was dissolved with mild heat. After dissolution 0.012 g (0.124 mmol) of sodium tert-butoxide was added while swirling by hand. The mixture turned opaque and deep purple.

The mixture was placed in open-air storage for one full week. At the end of the week it was retrieved and swirled. It turned to opaque dark green and showed solids. It was diluted with 4 mL of methanol, placed in ice bath, vacuum filtered, and washed with methanol. Recrystallization was performed with isopropanol to yield a deep yellow final product ([Appendix A: Final Product Photograph](#)).

The product was dried in open atmosphere for another full week. TLC was performed with 50% ethyl acetate in hexanes.

## Discussion and Results

Thin layer chromatography results depict a product that differs from the original. The  $R_f$  for 2-nitrofluorene was 0.70 while the  $R_f$  for 2-nitrofluorenone was 0.40.

The melting point obtained 219–223° C agrees with experimental results 220–223° C for 2-nitrofluorenone as reported in literature[3]. The range obtained suggests a purity better than 99%. The obtained value differs from the melting point of the reagent, 2-nitrofluorene, which was reported as 157.2 – 157.6° C above.

The IR spectrum of the product ([Appendix B: IR Spectra](#)) shows standard SP2 C–H peaks and standard benzene peaks. Three peaks of interest are displayed at the edge of the fingerprint region that can substantiate product identification: a C=O peak at wavenumber 1710.98, a possible C=C peak at wavenumber 1610.18, and a possible N–O peak at wavenumber 1517.14.

The carbon NMR spectrum of the product (*Appendix C: Carbon NMR Spectra*) shows the required 13 peaks. The only confirmed peak is the central carbon between the rings. The remainder are predictions based on calculations given in the following table (carbon positions are assigned in *Appendix C*). The predictions are not certain due to the number of peaks and close proximity of values:

**Table 1: Benzene Carbon NMR Predictive Values vs Experimental Values (given in ppm)**

Position	Benzene	NO <sub>2</sub>	Ph	COPh	Predicted	Experimental
'1'	128.5	20.0 (i)	-1.2 (p)	-0.2 (m)	147.1	148.23
'2'	128.5	-4.8 (o)	0.4 (m)	3.6 (p)	127.7	124.42
'3'	128.5	0.9 (m)	-1.1 (o)	-0.2 (m)	128.1	131.09
'4'	128.5	5.8 (p)	13.1 (i)	1.7 (o)	149.1	149.22
'5'	128.5	-	13.1 (i)	1.7 (o)	143.3	141.91
'6'	128.5	-	-1.1 (o)	-0.2 (m)	127.2	122.81
'7'	128.5	-	0.4 (m)	3.6 (p)	132.5	134.07
'8'	128.5	-	-1.2 (p)	-0.2 (m)	127.1	122.05
'9'	128.5	-	0.4 (m)	1.7 (o)	130.6	130.04
'10'	128.5	-	-1.1 (o)	9.4 (i)	136.8	134.16
'12'	128.5	0.9 (m)	-1.1 (o)	9.4 (i)	137.7	135.86
'13'	128.5	-4.8 (o)	0.4 (m)	1.7 (o)	125.8	118.31

The molar quantity of the primary reagent 2-nitrofluorene (C<sub>13</sub>H<sub>9</sub>NO<sub>2</sub>) was:

$$\text{mol C}_{13}\text{H}_9\text{NO}_2 = 0.158 \text{ g C}_{13}\text{H}_9\text{NO}_2 * \frac{1 \text{ mol C}_{13}\text{H}_9\text{NO}_2}{211.21606 \text{ g C}_{13}\text{H}_9\text{NO}_2} = 0.000748 \text{ mol C}_{13}\text{H}_9\text{NO}_2$$

The molar mass of the yielded product (2-nitrofluorenone) is 225.19958 g per mole. The number of moles yielded is:

$$\text{mol C}_{13}\text{H}_7\text{NO}_3 = 0.082 \text{ g C}_{13}\text{H}_7\text{NO}_3 \text{ (collected)} * \frac{1 \text{ mol C}_{13}\text{H}_7\text{NO}_3}{225.19958 \text{ g C}_{13}\text{H}_7\text{NO}_3} = 0.000364 \text{ mol C}_{13}\text{H}_7\text{NO}_3$$

The percent yield is:

$$\begin{aligned} \% \text{ - yield C}_{13}\text{H}_7\text{NO}_3 &= 0.000364 \text{ mol C}_{13}\text{H}_7\text{NO}_3 * \frac{1 \text{ mol C}_{13}\text{H}_9\text{NO}_2}{1 \text{ mol C}_{13}\text{H}_7\text{NO}_3} * \frac{1}{0.000748 \text{ mol C}_{13}\text{H}_9\text{NO}_2} * 100\% \\ &= 48.7\% \end{aligned}$$

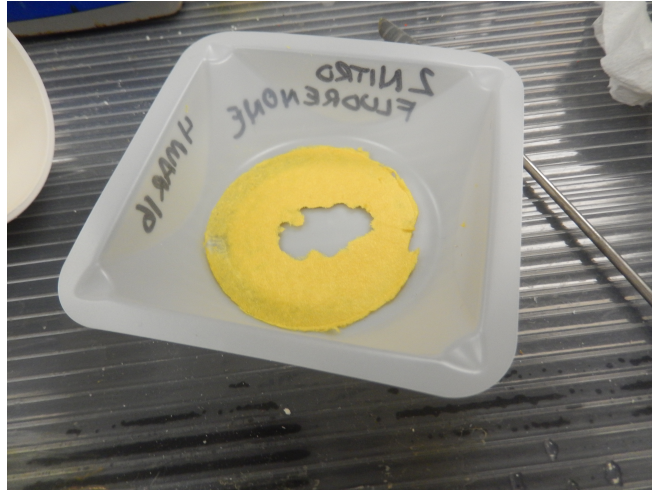
The yield is realistic for laboratory conditions.

## Conclusion

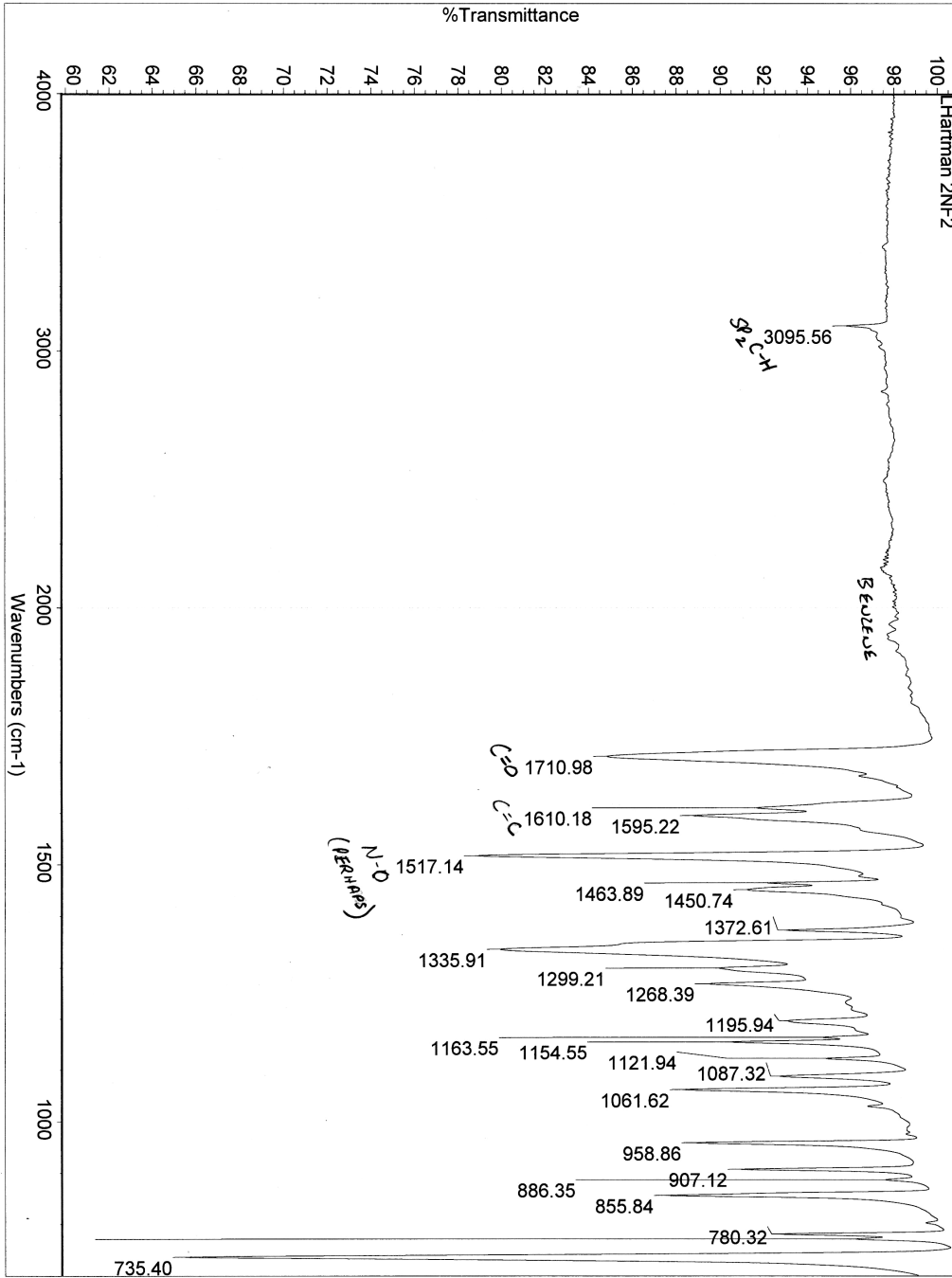
The product formed was 2-nitrofluorenone, with a reasonable yield. Purity of excellent quality was confirmed by an obtained melting point range that encompassed the experimental value reported in literature[3]. IR analysis also confirms product identity.



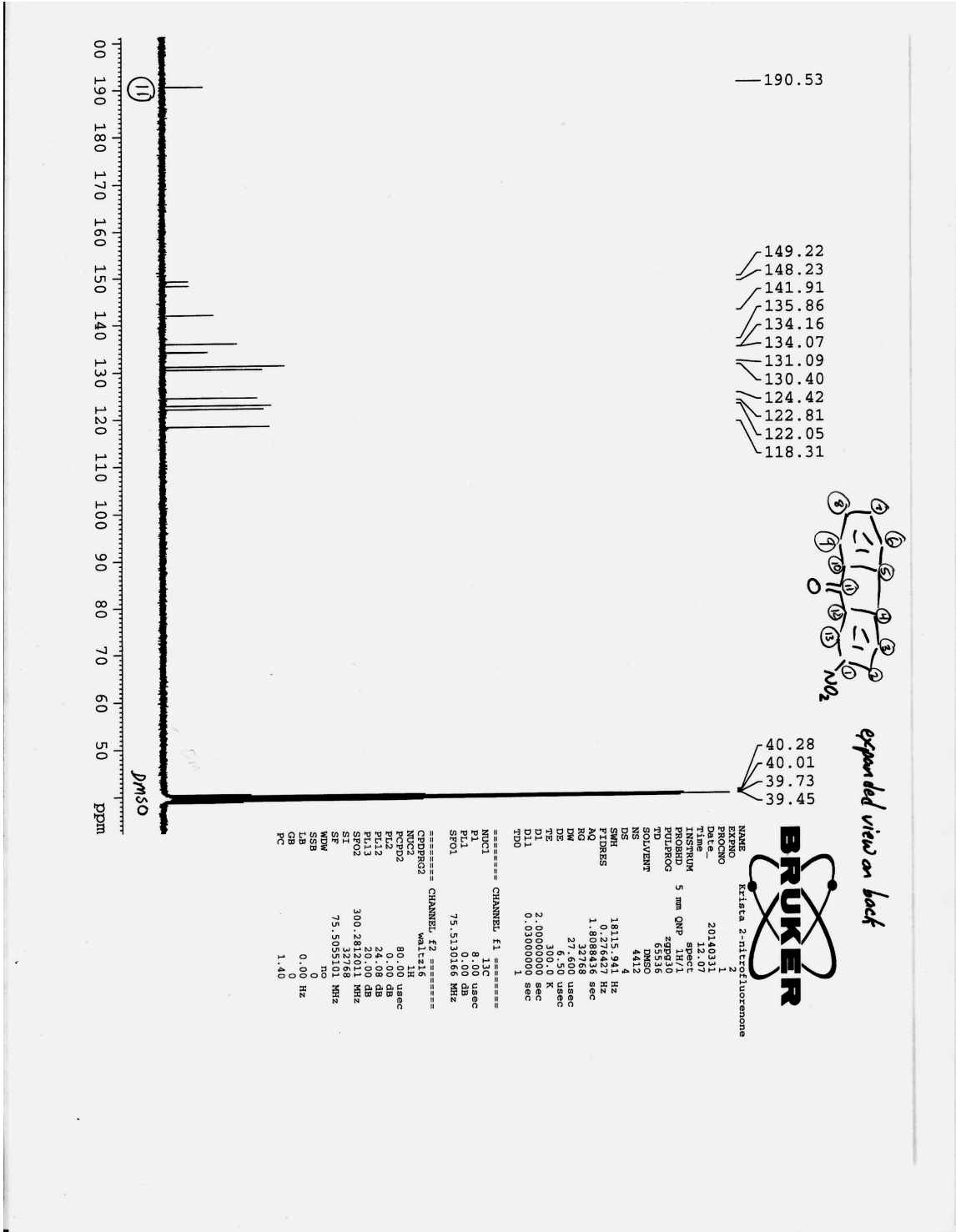
**Appendix A: Final Product Photograph**  
Source: Student Notes



Appendix B: IR Spectra  
Source: Student Notes



Appendix C: Carbon NMR Spectra  
Source: Laboratory Instructor



— 149.22  
— 148.23

— 141.91

— 135.86

— 134.16

— 134.07

— 131.09

— 130.40

— 124.42

— 122.81

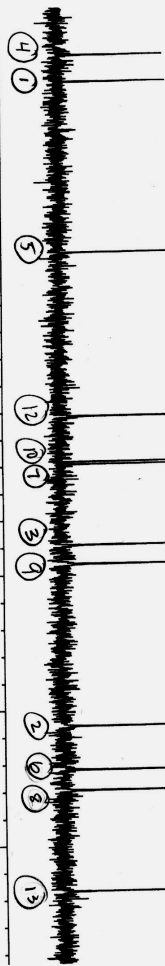
— 122.05

— 118.31

ASSIGNMENTS  
AMONG THESE  
VALUES UNCERTAIN

ASSIGNMENTS  
AMONG THESE  
VALUES UNCERTAIN

150  
145  
140  
135  
130  
125  
120  
ppm



**BRUKER**  
NAME Kelsita 2-nitrofluorenone  
EXPNO 1  
PROCNO 2  
Date\_ 20140331  
Time 12.07  
INSTRUM spect  
PROBHD 5 mm QNP 1H/1  
PULPROG zgpg30  
TD 65536  
F2 481.4  
SOLVENT DMSO  
NS 4  
DS 4  
SWH 18115.941 Hz  
FIDRES 0.276437 Hz  
AQ 1.8088436 sec  
RG 327.5  
SFO 27.600 usec  
DE 6.50 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.0300000 sec  
TD0 1

=====  
CHANNEL F1  
NUC1 13C  
P1 130 usec  
PL1 0.00 dB  
SFO1 75.5130166 MHz

=====  
CHANNEL F2  
NAME waltz16  
NUC1 13C  
PCPD2 80.00 usec  
PL2 0.00 dB  
PL12 24.08 dB  
PL13 24.08 dB  
PCPD3 300.2812001 MHz  
SFO2 32768  
SFO3 75.5055101 MHz  
SF 75.5055101 MHz  
WDW NO  
SSB 0  
GB 0  
PC 1.40

*expanded view*

**Appendix D: Experiment Laboratory Notes**  
Source: Student Notes



# Synthesis of 2-Aminofluorene from 2-Nitrofluorene

## Summary

The nitro group on 2-nitrofluorene was reduced with zinc and ammonium chloride to an amino group to produce 2-aminofluorene.

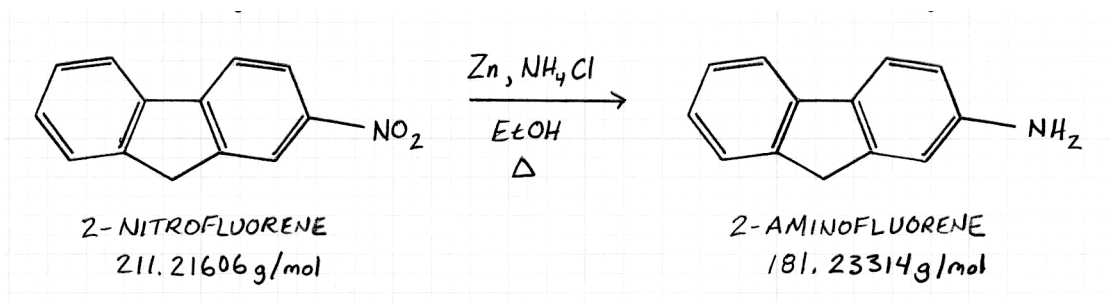


Fig 1: 2-Nitrofluorene reduction reaction to yield 2-aminofluorene

## Experimental Method and Observations

To a 125 mL Erlenmeyer flask was added 1.265 g (5.98 mmol) of 2-nitrofluorene and 20 mL of ethanol. In a separate container 1.926 g (36.0 mmol) of ammonium chloride was mixed with 6.8 mL of water. After mixing, the mixture was added to the Erlenmeyer flask. After further mixing for uniform consistency, 1.567 g (23.9 mmol) of zinc was added to the Erlenmeyer flask. The mixture was then heated with reflux for 30 minutes with a test tube condenser. A test tube filled with ice was inserted into the flask (but not touching the reaction mixture). During the reflux the melted ice was periodically discarded and replaced with more ice. The reflux resulted in a suspension of heavy grey metal salts.

TLC was performed with 10% ethyl acetate in hexanes to confirm that all of the 2-nitrofluorene was consumed. The suspension was cooled in an ice bath to room temperature. Afterwards 20 mL of 2N NaOH was added. Then the mixture was diluted with 24 mL of diethyl ether. The suspension was gravity filtered with a watch glass covering to prevent diethyl ether from evaporating. The mixture was comprised of an orange liquid and grey slurry. The remaining solid was rinsed with diethyl ether to obtain more product. The resulting solution was clear yellow.

The filtrate (clear, yellow) was separated in a separatory funnel. The aqueous layer was drained and checked for basicity with pH paper to ensure that the NaOH was separated. The reading was 12-13 pH. The remaining organic layer was washed with brine, drained and dried with anhydrous sodium sulfate. The diethyl ether was removed via rotary evaporation. The final product was a brown crystalline powder ([Appendix A: Final Product Photograph](#)).

The product was dried in open atmosphere for one full week. TLC was performed with 25% ethyl acetate in hexanes.

## Discussion and Results

Thin layer chromatography results depict a product that differs from the original. The  $R_f$  for 2-nitrofluorene was 0.38 while the  $R_f$  for 2-aminofluorene was 0.07.

The melting point obtained 123–124° C agrees with experimental results 128–129° C for 2-aminofluorene as reported in literature[4]. The range obtained suggests a purity better than 95%. The obtained value differs from the melting point of the reagent, 2-nitrofluorene, which was reported as 157.2 – 157.6° C above.

The IR spectrum of the product (*Appendix B: IR Spectra*) shows standard SP3 C–H peaks, standard SP2 C–H peaks and standard benzene peaks. Three peaks of interest are displayed on the plot that can substantiate product identification: a twin RNH<sub>2</sub> peak at wavenumbers 3445.63 and 3358.90, and a possible C=C peak at wavenumber 1612.95.

The proton NMR spectrum of the product (*Appendix C: Proton NMR Spectra*) shows a number of unremarkable peaks indicative of benzene rings. There are also two peaks that accord with the two central hydrogens between the two rings and the two hydrogens attached to the nitrogen.

The molar quantity of the primary reagent 2-nitrofluorene (C<sub>13</sub>H<sub>9</sub>NO<sub>2</sub>) was:

$$\text{mol C}_{13}\text{H}_9\text{NO}_2 = 1.265 \text{ g C}_{13}\text{H}_9\text{NO}_2 * \frac{1 \text{ mol C}_{13}\text{H}_9\text{NO}_2}{211.21606 \text{ g C}_{13}\text{H}_9\text{NO}_2} = 0.00599 \text{ mol C}_{13}\text{H}_9\text{NO}_2$$

The molar mass of the yielded product (2-aminofluorene) is 181.23314 g per mole. The number of moles yielded is:

$$\text{mol C}_{13}\text{H}_{11}\text{N} = 0.685 \text{ g C}_{13}\text{H}_{11}\text{N (collected)} * \frac{1 \text{ mol C}_{13}\text{H}_{11}\text{N}}{181.23314 \text{ g C}_{13}\text{H}_{11}\text{N}} = 0.00378 \text{ mol C}_{13}\text{H}_{11}\text{N}$$

The percent yield is:

$$\begin{aligned} \% \text{ - yield C}_{13}\text{H}_{11}\text{N} &= 0.00378 \text{ mol C}_{13}\text{H}_{11}\text{N} * \frac{1 \text{ mol C}_{13}\text{H}_9\text{NO}_2}{1 \text{ mol C}_{13}\text{H}_{11}\text{N}} * \frac{1}{0.00599 \text{ mol C}_{13}\text{H}_9\text{NO}_2} * 100\% \\ &= 63.1\% \end{aligned}$$

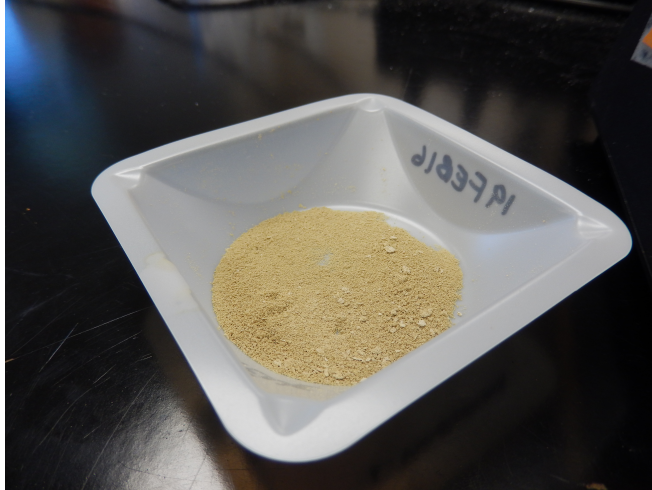
The yield is realistic for laboratory conditions.

## Conclusion

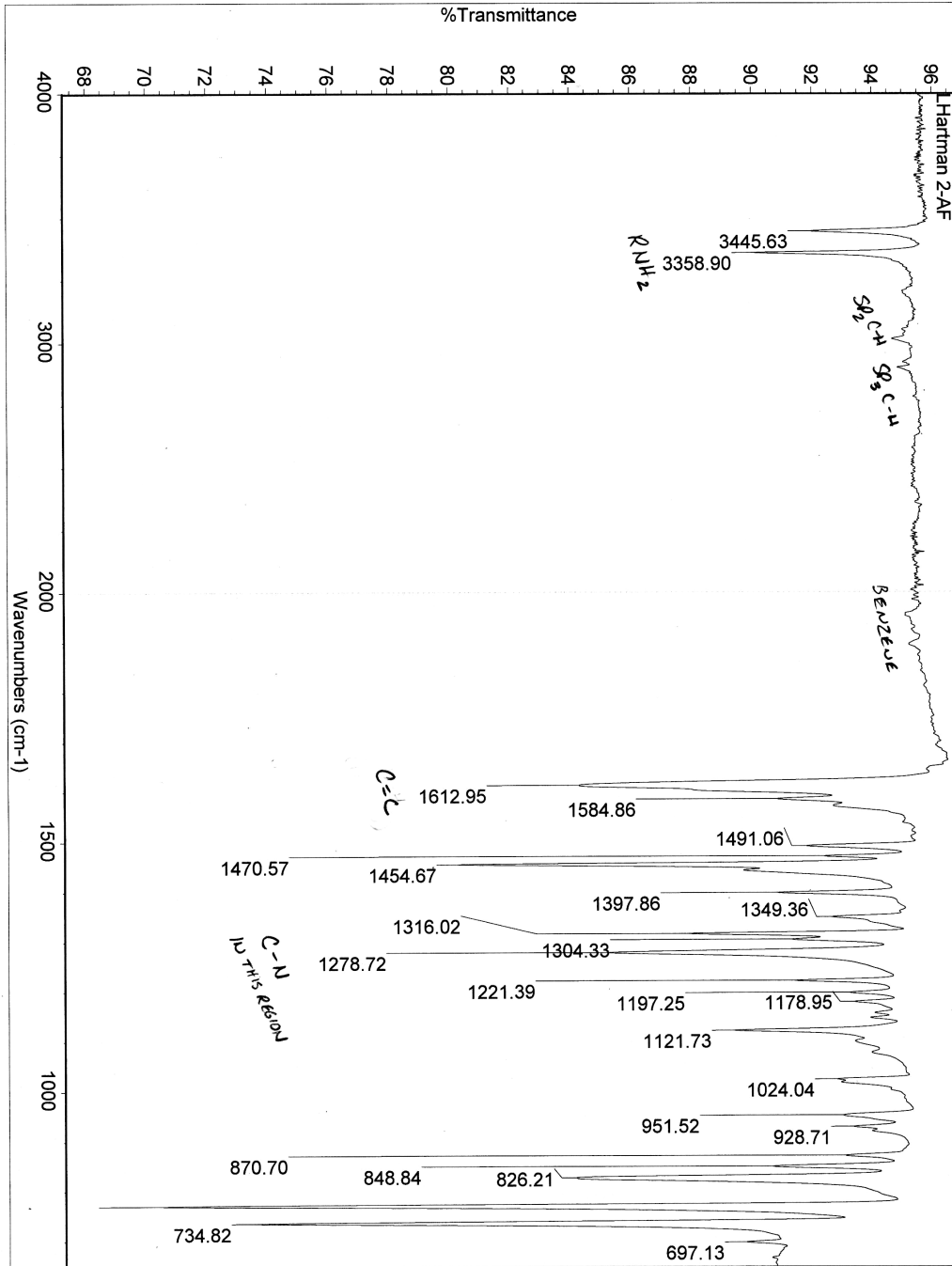
The product formed was 2-aminofluorene, with a reasonable yield. Purity of excellent quality was confirmed by an obtained melting point range that encompassed the experimental value reported in literature[4]. IR analysis also confirms product identity.



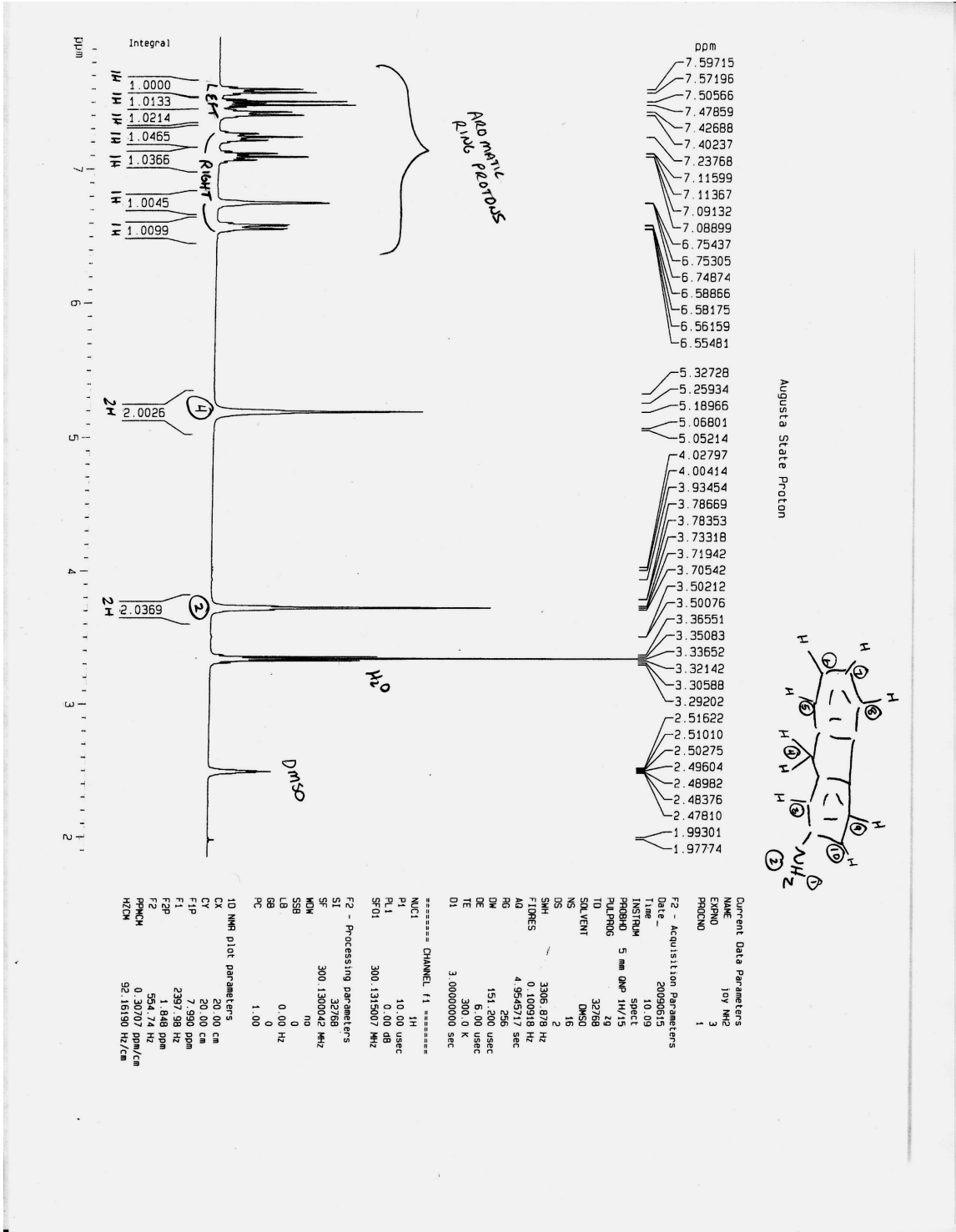
**Appendix A: Final Product Photograph**  
Source: Student Notes



Appendix B: IR Spectra  
Source: Student Notes



Appendix C: Proton NMR Spectra  
Source: Laboratory Instructor





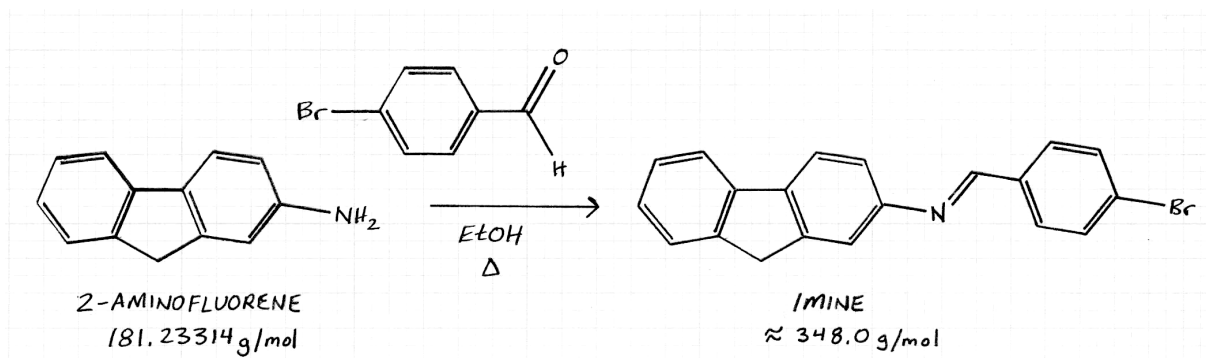
**Appendix D: Experiment Laboratory Notes**  
Source: Student Notes



# Synthesis of Imine from 2-Aminofluorene

## Summary

The tertiary amine group on 2-aminofluorene reacted with the carbonyl group on 4-bromobenzaldehyde to form N-[(4-bromophenyl)methyl]-9H-fluor-2-imine.



**Fig 1:** 2-aminofluorene reaction to yield N-[(4-bromophenyl)methyl]-9H-fluor-2-imine

## Experimental Method and Observations

The following compounds were transferred into a clean, dry round bottom flask: 0.273 g (1.50 mmol) of 2-aminofluorene, 9 mL of ethanol, and 0.326 g (1.76 mmol) of 4-bromobenzaldehyde. The mixture was stirred and heated with reflux for a total of 30 minutes. The mixture was removed from heat and placed in an ice bath. After several minutes of cooling the precipitate was collected via vacuum filtration and rinsed with methanol.

The crude was recrystallized with isopropanol using hot filtration to obtain purified final product. Hot filtration was used to keep product dissolved due to the likelihood the product would recrystallize on the filter paper. The product was pale yellow in color ([Appendix A: Final Product Photograph](#)).

The product was dried in open atmosphere for one full week. TLC was performed with 25% ethyl acetate in hexanes.

## Discussion and Results

Thin layer chromatography results depict a product that differs from the original. The  $R_f$  for 2-aminofluorene was 0.29 while the  $R_f$  for N-[(4-bromophenyl)methyl]-9H-fluor-2-imine was 0.74.

The melting point obtained 184 – 185.2° C. No experimental values stemming from literature searches were discovered to corroborate obtained value. The obtained value differs from the melting point of the reagent, 2-aminofluorene, which was reported as 123 – 124° C above.

The IR spectrum of the product ([Appendix B: IR Spectra](#)) shows standard SP3 C–H peaks, standard SP2 C–H peaks and standard benzene peaks. One peak of interest was displayed at the edge of the fingerprint region that may substantiate product identification: a possible C=N peak at wavenumber 1646.00.

The proton NMR spectrum of the product (*Appendix C: Proton NMR Spectra*) shows a number of unremarkable peaks indicative of benzene rings. There are also two peaks that accord with the two central hydrogens between the two rings and the SP<sup>2</sup> hydrogen attached to the carbon double bonded to the nitrogen.

The proposed mechanism for N-[(4-bromophenyl)methyl]-9H-fluor-2-imine is presented in *Appendix D: Mechanism*. The amine group of 2-aminofluorene is a strong nucleophile that attacks the carbonyl of the aldehyde. Water is formed as a byproduct of the reaction.

The molar quantity of the primary reagent 2-aminofluorene (C<sub>13</sub>H<sub>11</sub>N) was:

$$\text{mol C}_{13}\text{H}_{11}\text{N} = 0.273 \text{ g C}_{13}\text{H}_{11}\text{N} * \frac{1 \text{ mol C}_{13}\text{H}_{11}\text{N}}{181.23314 \text{ g C}_{13}\text{H}_{11}\text{N}} = 0.00151 \text{ mol C}_{13}\text{H}_{11}\text{N}$$

The molar mass of the yielded product (N-[(4-bromophenyl)methyl]-9H-fluor-2-imine) is about 348.0 g per mole. The number of moles yielded is:

$$\text{mol C}_{20}\text{H}_{14}\text{BrN} = 0.218 \text{ g C}_{20}\text{H}_{14}\text{BrN (collected)} * \frac{1 \text{ mol C}_{20}\text{H}_{14}\text{BrN}}{348.0 \text{ g C}_{20}\text{H}_{14}\text{BrN}} = 0.000626 \text{ mol C}_{20}\text{H}_{14}\text{BrN}$$

The percent yield is:

$$\begin{aligned} \% \text{ - yield C}_{20}\text{H}_{14}\text{BrN} &= 0.000946 \text{ mol C}_{20}\text{H}_{14}\text{BrN} * \frac{1 \text{ mol C}_{13}\text{H}_{11}\text{N}}{1 \text{ mol C}_{20}\text{H}_{14}\text{BrN}} * \frac{1}{0.00148 \text{ mol C}_{13}\text{H}_{11}\text{N}} * 100\% \\ &= 41.6\% \end{aligned}$$

The yield is realistic for laboratory conditions.

## Conclusion

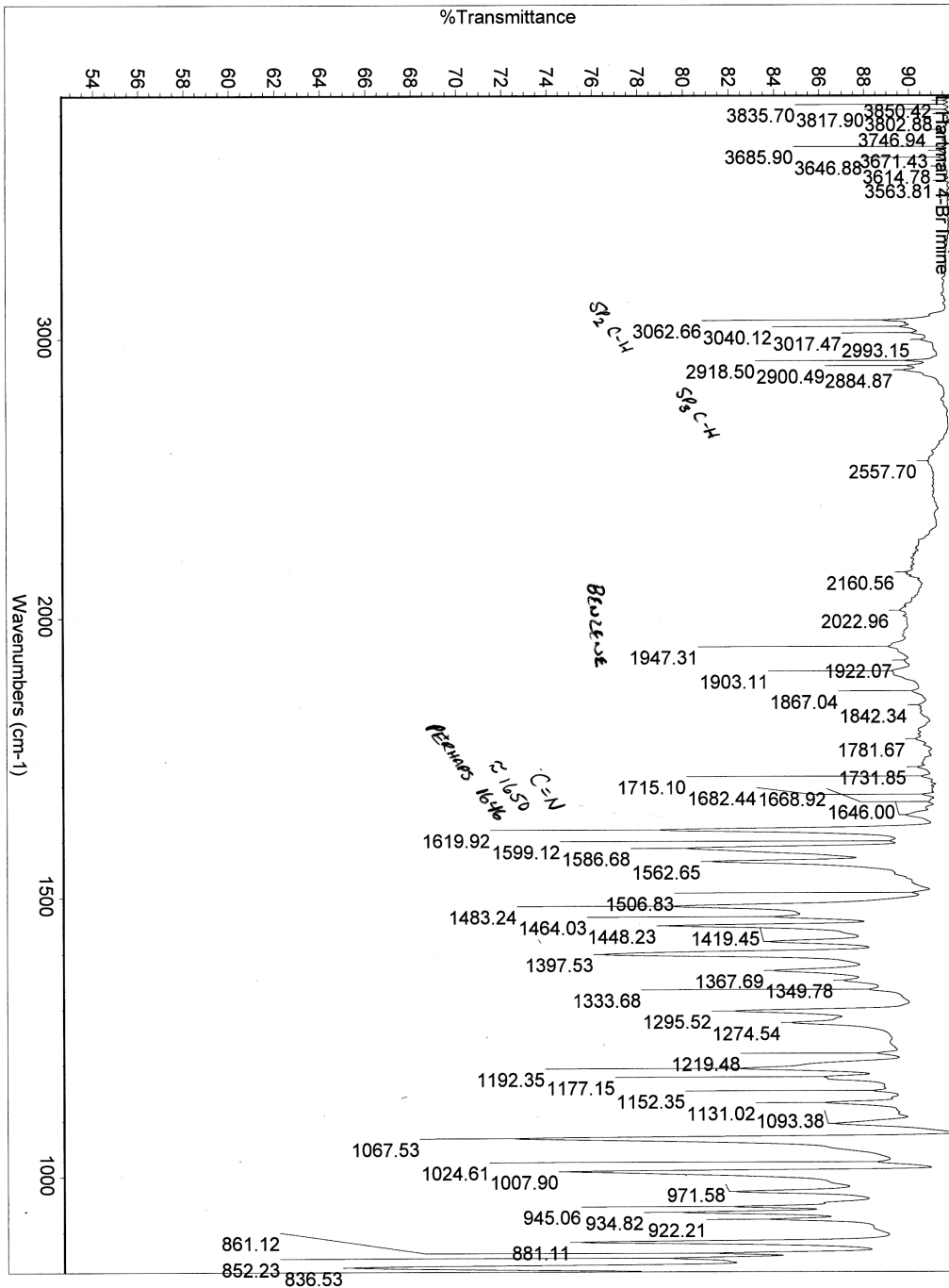
The product formed was likely N-[(4-bromophenyl)methyl]-9H-fluor-2-imine, with a reasonable yield. IR spectrum analysis does not refute this conclusion. Without further data—an experimental melting point from literature would be useful—certainty can not be ascertained.



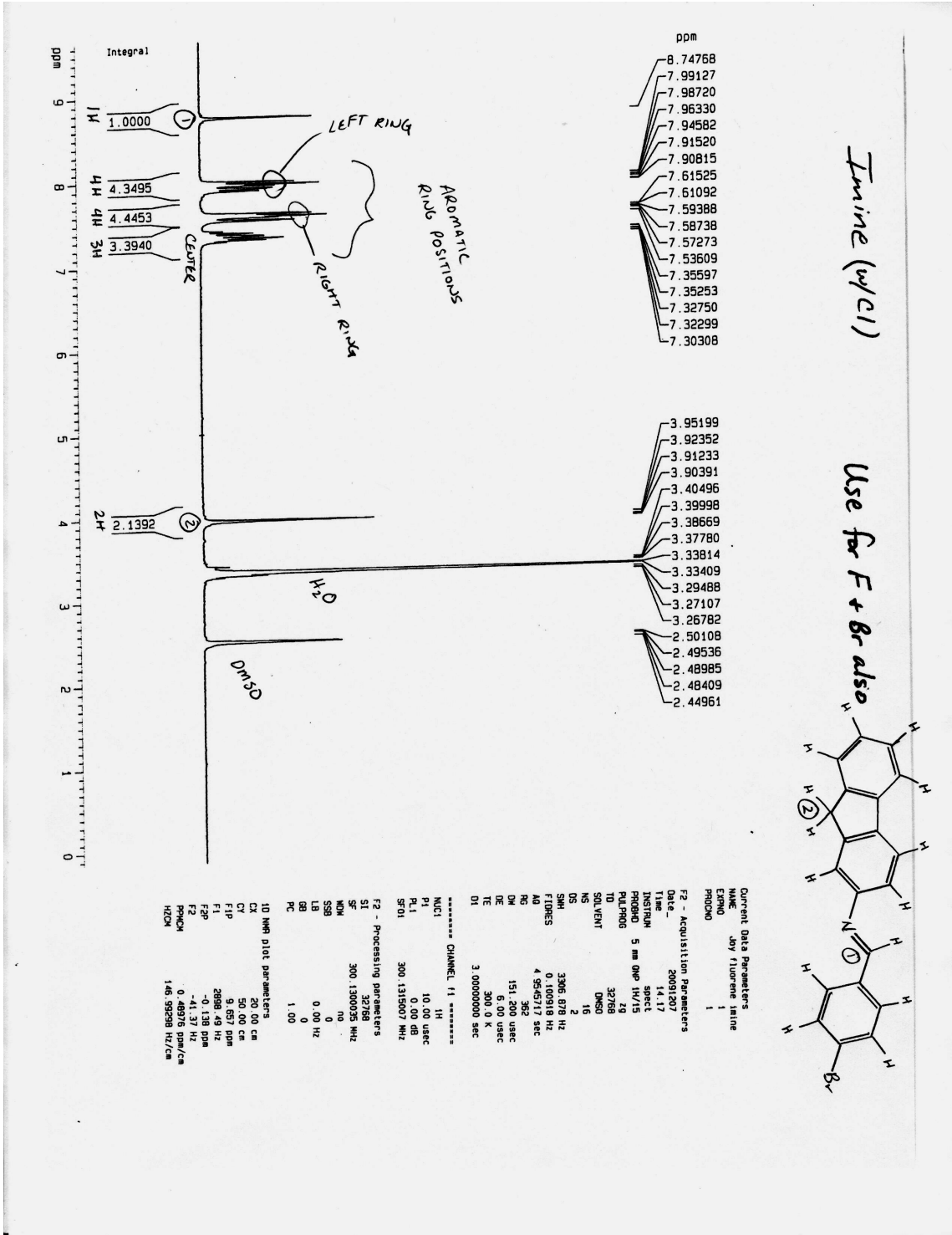
**Appendix A: Final Product Photograph**  
Source: Student Notes



Appendix B: IR Spectra  
 Source: Student Notes

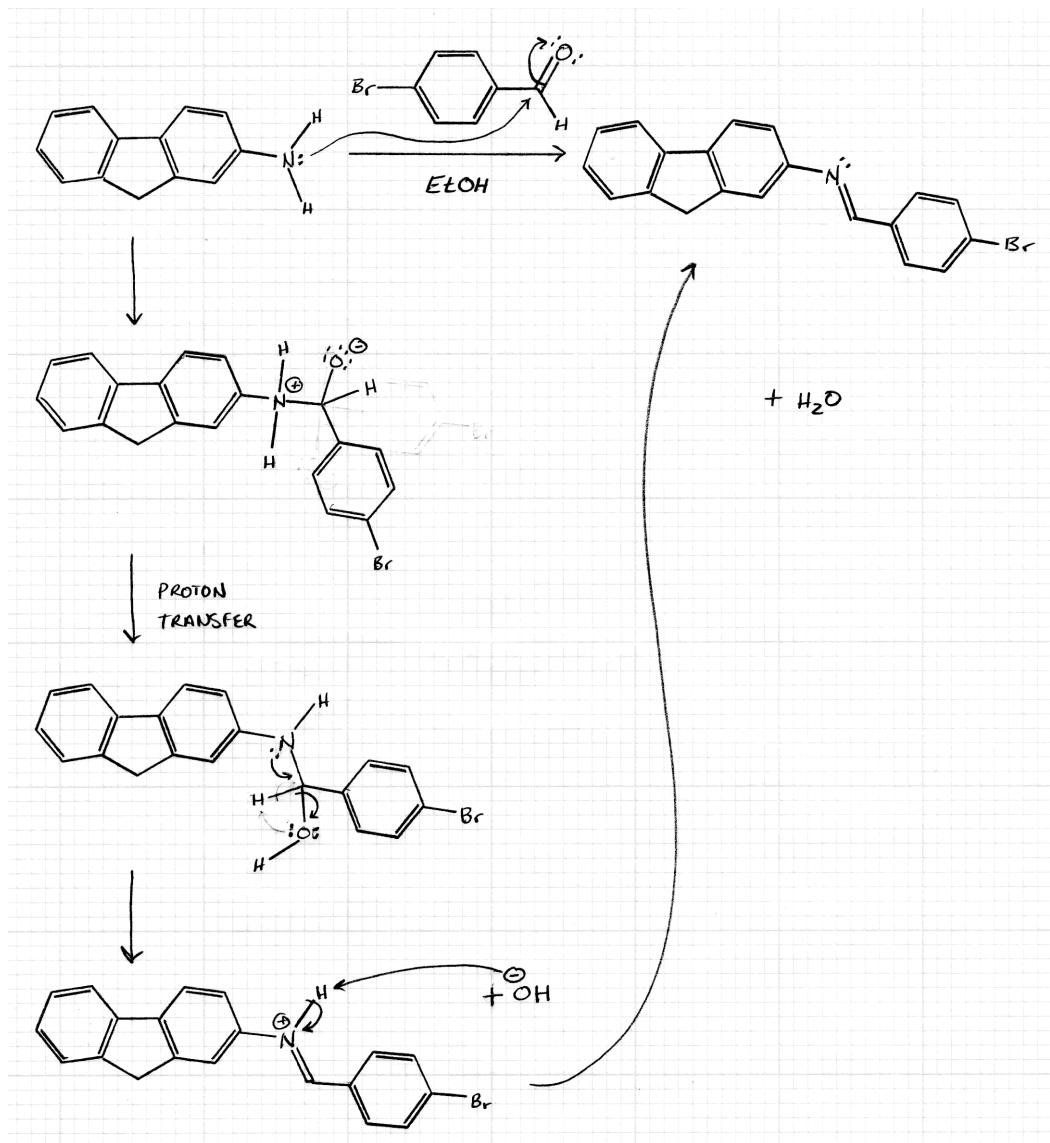


Appendix C: Proton NMR Spectra  
Source: Laboratory Instructor



# Appendix D: Mechanism

Source: Student Notes



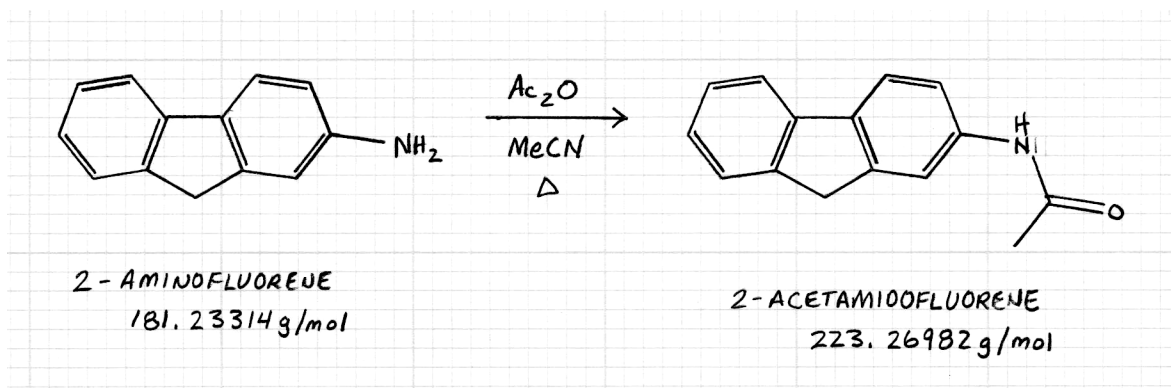
**Appendix E: Experiment Laboratory Notes**  
Source: Student Notes



# Synthesis of Acetamide from 2-Aminofluorene

## Summary

2-Aminofluorene was acetylated with acetic anhydride to produce 2-acetamidofluorene. Acetonitrile was used as the solvent because its aprotic property was important to prevent unwanted protonation from occurring.



**Fig 1:** 2-Aminofluorene acetylation reaction to yield 2-acetamidofluorene

## Experimental Method and Observations

The following compounds were transferred into a clean, dry round bottom flask: 0.136 g (0.750 mmol) of 2-aminofluorene, 0.136 g (1.33 mmol) of acetic anhydride, and 3 mL acetonitrile. The mixture was stirred and heated with reflux for a total of 30 minutes. At the end of reflux water was added to the mixture to promote crystallization. It was then placed in an ice bath for several minutes. After crystallization, the mixture was vacuum filtered and the solid washed with water. The crude product was a green clay consistency ([Appendix A: Crude Product Photograph](#)).

After the crude was dried in open atmosphere for one full week, it was recrystallized from toluene. The final product was a white solid.

TLC was performed with 25% ethyl acetate in hexanes; however, no distinction between original reactant and product could be determined. TLC was reperformed with 50% ethyl acetate in hexanes with better results.

## Discussion and Results

Thin layer chromatography results depict a product that differs from the original. The  $R_f$  for 2-aminofluorene was 0.60 while the  $R_f$  for 2-acetamidofluorene was 0.29.

The melting point obtained 186.5–189° C agrees with experimental results 192–193° C for 2-acetamidofluorene as reported in literature[6]. The range obtained suggests a purity better than 95%. The obtained value differs from the melting point of the reagent, 2-aminofluorene, which was reported as 123 – 124° C above.

The IR spectrum of the product (*Appendix B: IR Spectra*) shows standard SP3 C–H peaks, standard SP2 C–H peaks and standard benzene peaks. Two peaks of interest are displayed on the plot that can substantiate product identification: an RNH<sub>2</sub> peak at wavenumber 3297.45 and a C=O peak at wavenumber 1660.91.

The proton NMR spectrum of the product (*Appendix C: Proton NMR Spectra*) shows a number of unremarkable peaks indicative of benzene rings. There are also three peaks that accords with the two central hydrogens between the two rings, the hydrogen attached to the nitrogen, and the hydrogens belonging to the methyl group.

The proposed mechanism for 2-acetamidofluorene is presented in *Appendix D: Mechanism*). The amine group of 2-aminofluorene is a strong nucleophile that attacks the carbonyl of the symmetric anhydride. Acetic acid is formed as a byproduct of the reaction.

The molar quantity of the primary reagent 2-aminofluorene (C<sub>13</sub>H<sub>11</sub>N) was:

$$\text{mol C}_{13}\text{H}_{11}\text{N} = 0.136 \text{ g C}_{13}\text{H}_{11}\text{N} * \frac{1 \text{ mol C}_{13}\text{H}_{11}\text{N}}{181.23314 \text{ g C}_{13}\text{H}_{11}\text{N}} = 0.00750 \text{ mol C}_{13}\text{H}_{11}\text{N}$$

The molar mass of the yielded product (2-acetamidofluorene) is 223.26982 g per mole. The number of moles yielded is:

$$\text{mol C}_{15}\text{H}_{13}\text{NO} = 0.175 \text{ g C}_{15}\text{H}_{13}\text{NO (collected)} * \frac{1 \text{ mol C}_{15}\text{H}_{13}\text{NO}}{223.26982 \text{ g C}_{15}\text{H}_{13}\text{NO}} = 0.000784 \text{ mol C}_{15}\text{H}_{13}\text{NO}$$

The percent yield is:

$$\begin{aligned} \% \text{ - yield C}_{15}\text{H}_{13}\text{NO} &= 0.000784 \text{ mol C}_{15}\text{H}_{13}\text{NO} * \frac{1 \text{ mol C}_{13}\text{H}_{11}\text{N}}{1 \text{ mol C}_{15}\text{H}_{13}\text{NO}} * \frac{1}{0.00750 \text{ mol C}_{13}\text{H}_{11}\text{N}} * 100\% \\ &= 104\% \end{aligned}$$

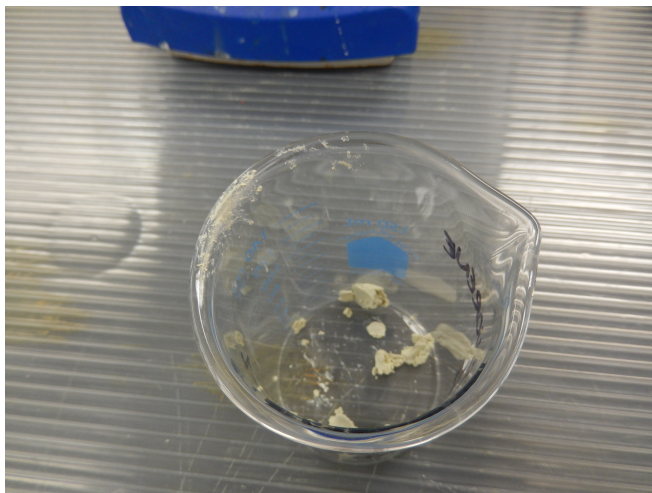
The yield is not realistic. It was discovered later in the sequence of these experiments that the scale used to obtain many of the reported masses was subject to a large error if the plastic shielding surrounding the sample plate was in any manner disturbed. This measurement may have been subject to this error.

## Conclusion

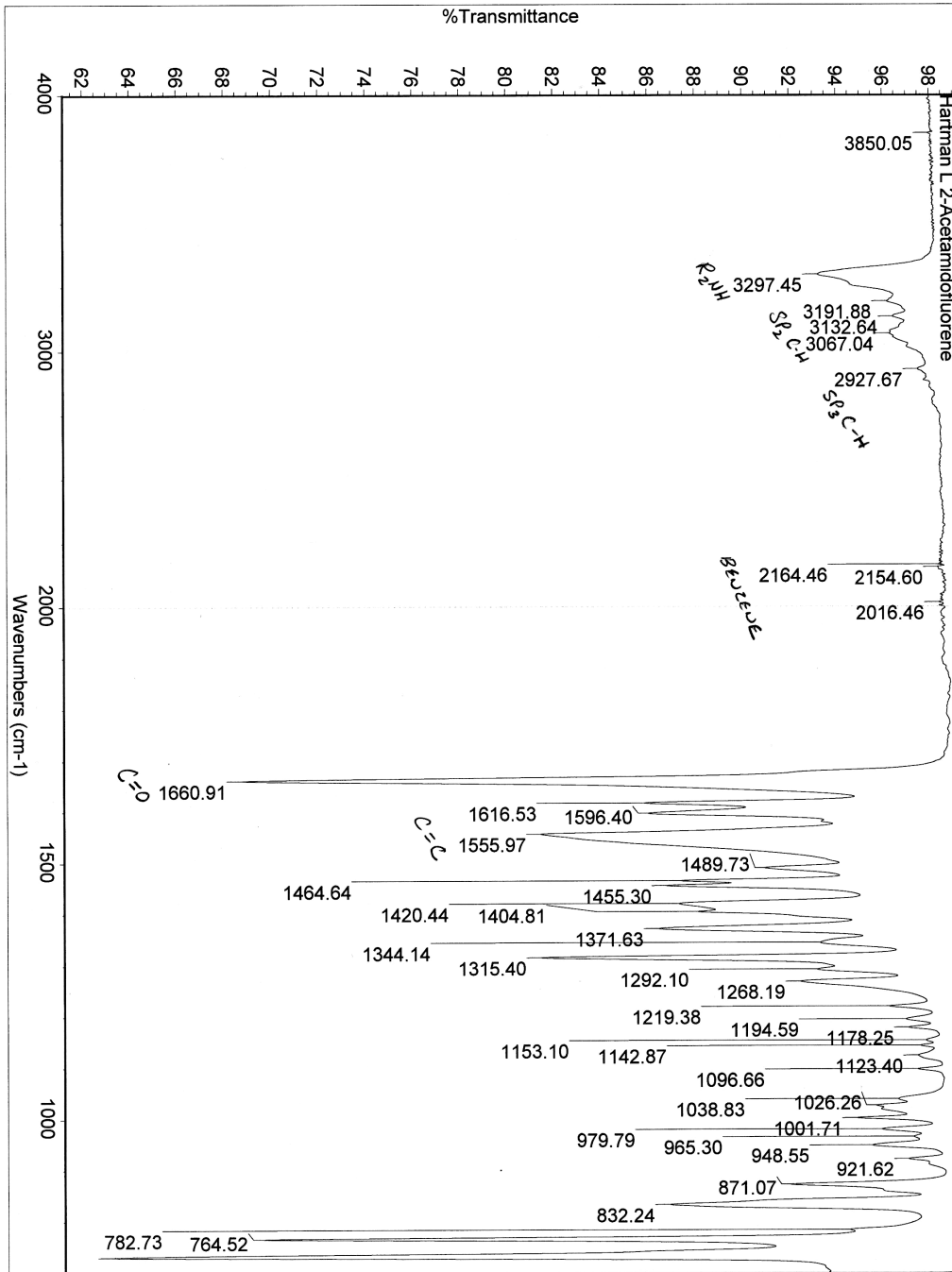
The product formed was 2-acetamidofluorene, with an erroneous yield value. Purity of excellent quality was confirmed by an obtained melting point range that encompassed the experimental value reported in literature[6]. IR analysis also confirms product identity.



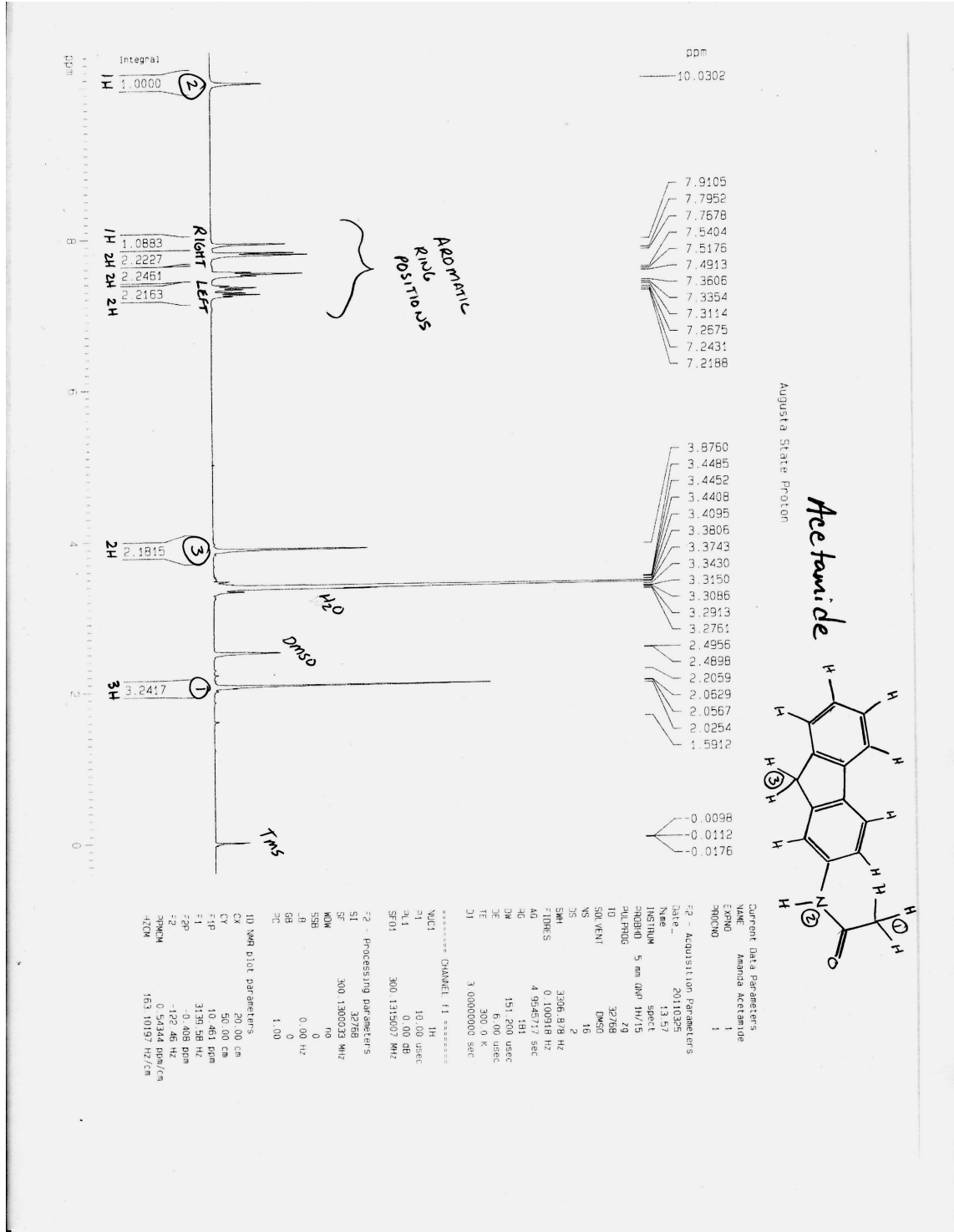
**Appendix A: Crude Product Photograph**  
Source: Student Notes



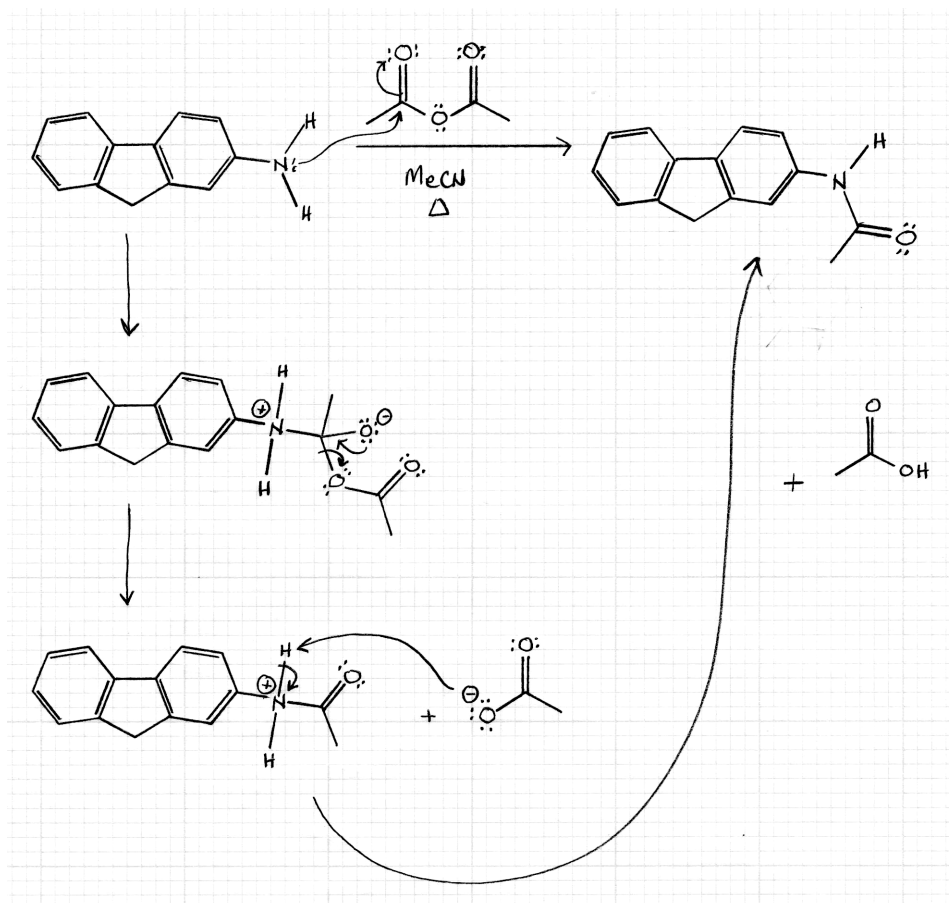
**Appendix B: IR Spectra**  
 Source: Student Notes



Appendix C: Proton NMR Spectra  
Source: Laboratory Instructor



Appendix D: Mechanism  
Source: Student Notes



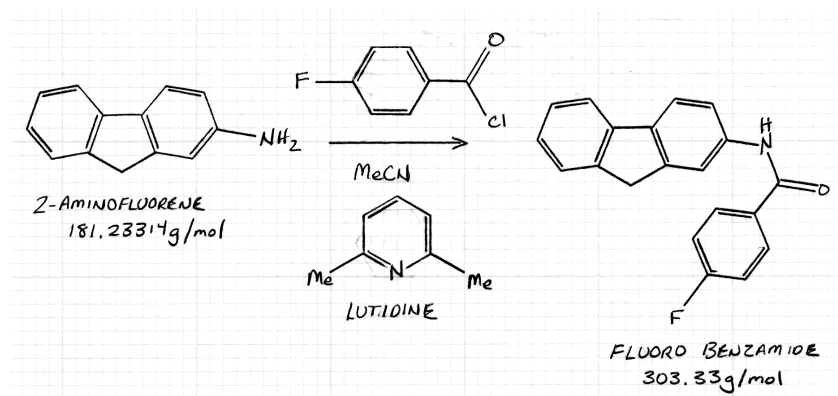
**Appendix E: Experiment Laboratory Notes**  
Source: Student Notes



# Synthesis of Benzylamide from 2-Aminofluorene

## Summary

2-Aminofluorene was added to 4-fluorobenzoyl chloride (acid chloride) to produce N-9H-fluoren-3-yl-4-fluoro-benzamide.



**Fig 1:** 2-Aminofluorene reaction to yield N-9H-fluoren-3-yl-4-fluoro-benzamide

## Experimental Method and Observations

This reaction was scaled to 83% of lab manual specifications due to inadequate quantity of 2-aminofluorene. To a 50 mL Erlenmeyer flask was added: 0.075 g (0.414 mmol) of 2-aminofluorene and 4 mL acetonitrile. The mixture was stirred until the solid was dissolved. A graduated cylinder was tared with a few drops of acetonitrile. To the cylinder was added 0.080 g (0.504 mmol) of 4-fluorobenzoyl chloride (liquid phase), then contents were transferred to the flask where an immediate reaction took place. Afterwards, 6 drops of lutidine was added directly to the flask. The mixture was stirred at room temperature for 35 minutes. When stirring was complete 3 mL of water was added to promote crystallization. The crude was vacuum filtered.

The crude was recrystallized with 35 mL of propanol to obtain purified final product. The quantity of propanol added was far more than necessary and caused significant problems. The product would not recrystallize after 15 minutes of ice bath until the bottom of the beaker was scraped with a glass stirring rod and an additional 10 mL of water was added. Product recovery after two rounds of vacuum filtration was very poor. The product obtained was a tan powder ([Appendix A: Final Product Photograph](#)).

The product was dried in open atmosphere for one full week. TLC was performed with 50% ethyl acetate in hexanes.

## Discussion and Results

Thin layer chromatography results depict a product that differs from the original. The  $R_f$  for 2-aminofluorene was 0.65 while the  $R_f$  for N-9H-fluoren-3-yl-4-fluoro-benzamide was 0.79.

The melting point obtained 227 – 230° C. No experimental values within literature probes were discovered to corroborate obtained value. The obtained value differs from the melting point of the reagent, 2-aminofluorene, which was reported as 123 – 124° C above.

The IR spectrum of the product (*Appendix B: IR Spectra*) shows standard SP3 C–H peaks, standard SP2 C–H peaks and standard benzene peaks. Three peaks of interest are displayed on the plot that can substantiate product identification: an RNH<sub>2</sub> peak at wavenumber 3290, a C=O peak at wavenumber 1644, and a C=C peak at wavenumber 1590 or 1604.

The proton NMR spectrum of the product (*Appendix C: Proton NMR Spectra*) shows a number of unremarkable peaks indicative of benzene rings. There are also two peaks that accords with the two central hydrogens between the two rings and the hydrogen attached to the nitrogen. The fluorine NMR spectrum of the product (*Appendix D: Fluorine NMR Spectra*) shows a single peak with a multiplet signal. The fluorine is located in an assymmetric position on the proposed product. Given fluorine NMR sensitivity to reaching across further bonds, this position would yield a complex signal.

The proposed mechanism for N-9H-fluoren-3-yl-4-fluoro-benzamide is presented in *Appendix E: Mechanism*). The amine group of 2-aminofluorene is a strong nucleophile that attacks the carbonyl of the acid chloride. Hydrochloric acid is formed as a byproduct of the reaction.

The molar quantity of the primary reagent 2-aminofluorene (C<sub>13</sub>H<sub>11</sub>N) was:

$$\text{mol C}_{13}\text{H}_{11}\text{N} = 0.075 \text{ g C}_{13}\text{H}_{11}\text{N} * \frac{1 \text{ mol C}_{13}\text{H}_{11}\text{N}}{181.23314 \text{ g C}_{13}\text{H}_{11}\text{N}} = 0.000414 \text{ mol C}_{13}\text{H}_{11}\text{N}$$

The molar mass of the yielded product (N-9H-fluoren-3-yl-4-fluoro-benzamide) is 303.33 g per mole. The number of moles yielded is:

$$\text{mol C}_{20}\text{H}_{14}\text{FNO} = 0.015 \text{ g C}_{20}\text{H}_{14}\text{FNO (collected)} * \frac{1 \text{ mol C}_{20}\text{H}_{14}\text{FNO}}{303.33 \text{ g C}_{20}\text{H}_{14}\text{FNO}} = 0.000049 \text{ mol C}_{20}\text{H}_{14}\text{FNO}$$

The percent yield is:

$$\begin{aligned} \% \text{ - yield C}_{20}\text{H}_{14}\text{FNO} &= 0.000049 \text{ mol C}_{20}\text{H}_{14}\text{FNO} * \frac{1 \text{ mol C}_{13}\text{H}_{11}\text{N}}{1 \text{ mol C}_{20}\text{H}_{14}\text{FNO}} * \frac{1}{0.000414 \text{ mol C}_{13}\text{H}_{11}\text{N}} * 100\% \\ &= 11.9\% \end{aligned}$$

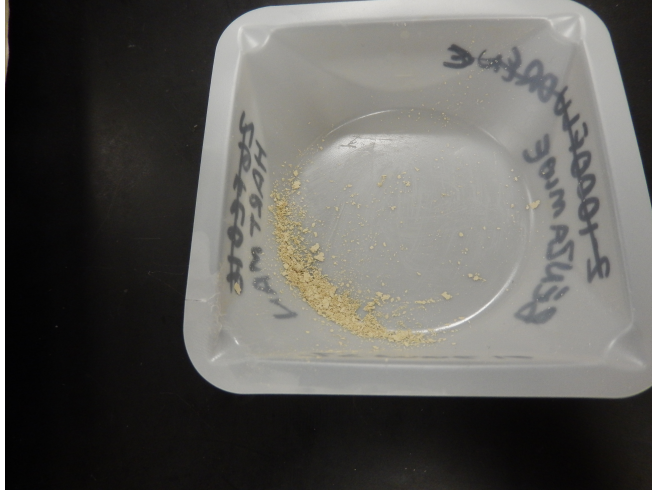
The yield obtained was very minimal. See above for mistake made during recrystallization procedure.

## Conclusion

The product formed was likely N-9H-fluoren-3-yl-4-fluoro-benzamide, with a low yield. IR spectrum analysis does not refute this conclusion. Without further data—an experimental melting point from literature would be useful—certainty can not be ascertained.

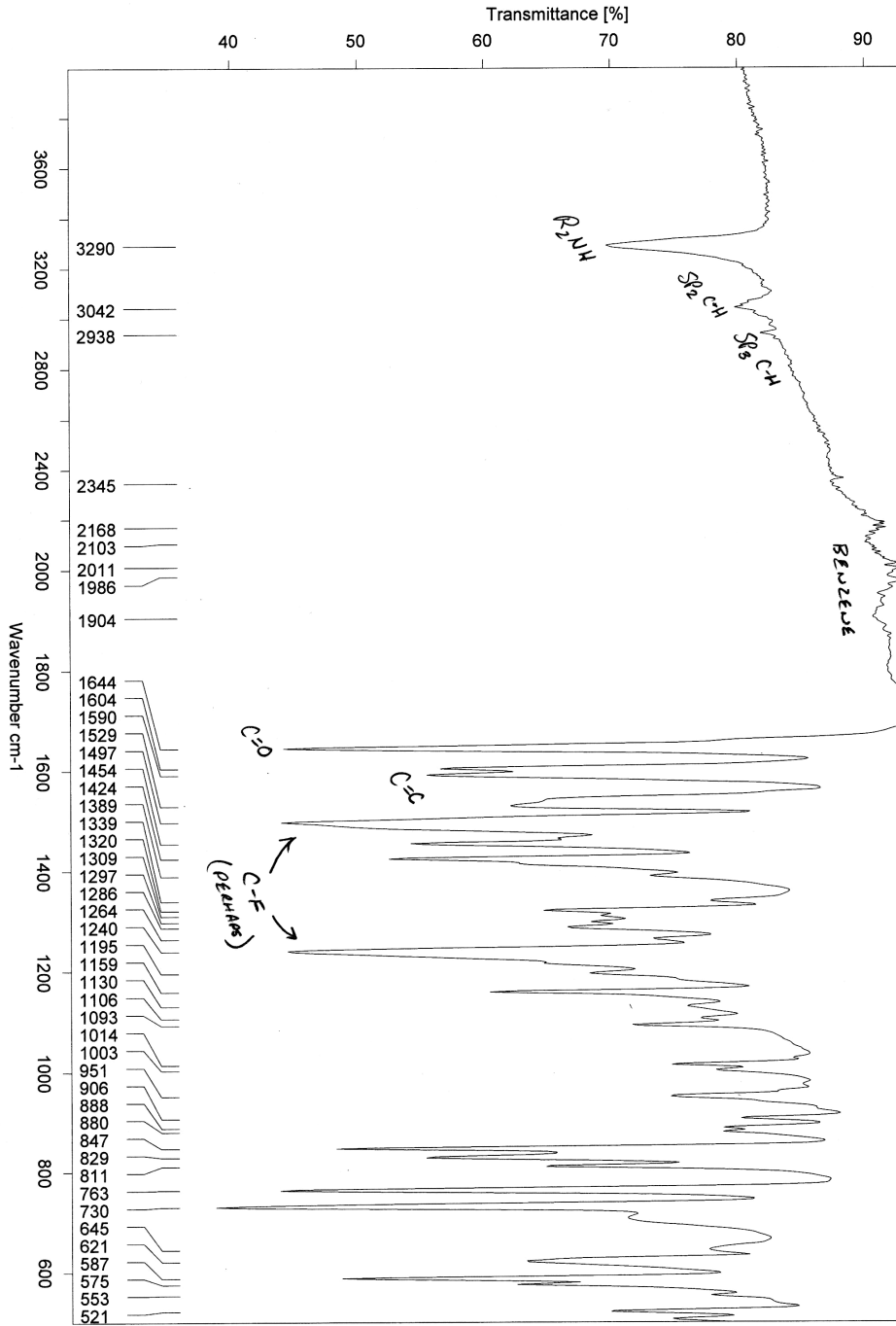


**Appendix A: Final Product Photograph**  
Source: Student Notes



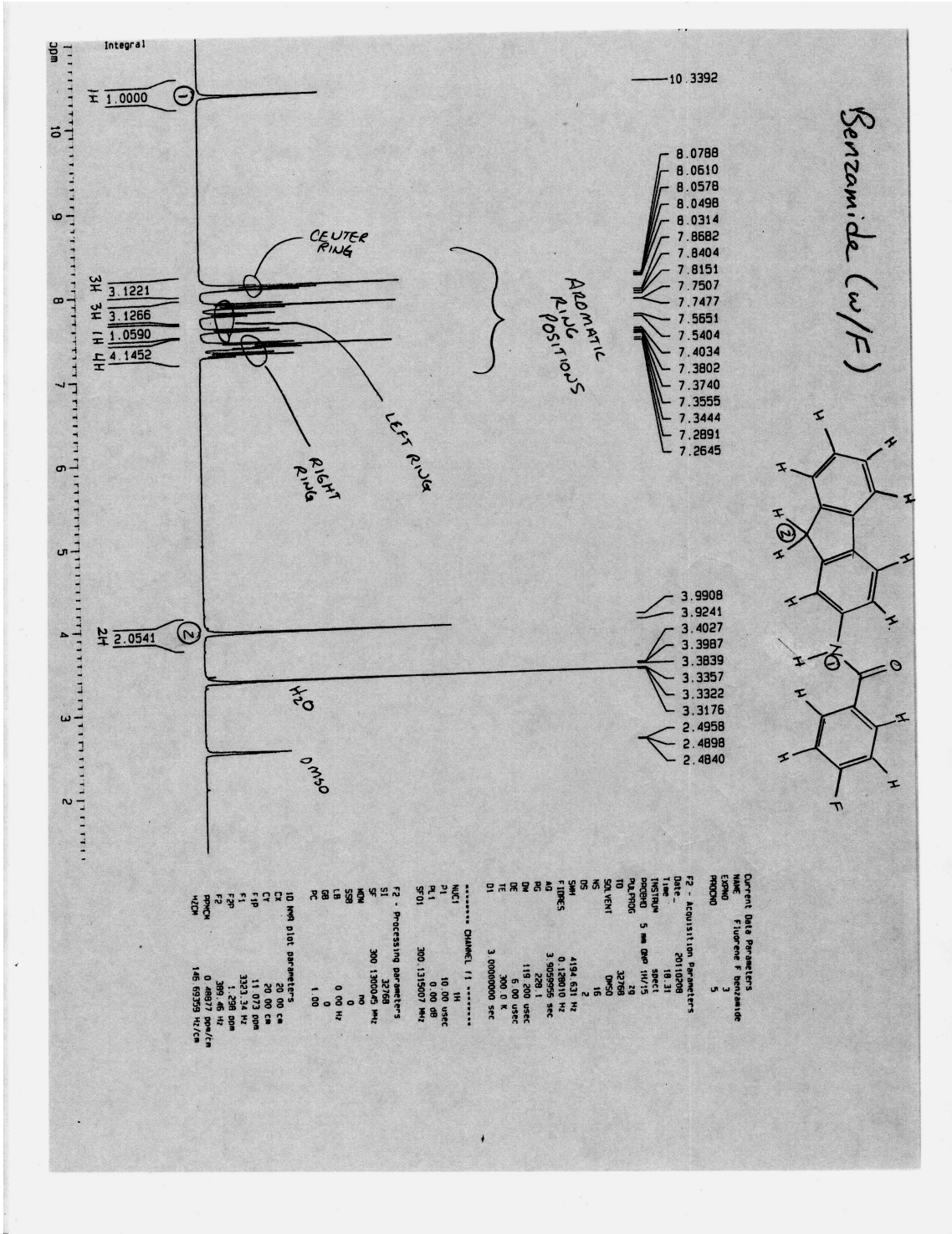
Appendix B: IR Spectra  
Source: Student Notes

4/1/2016



Fluoro Benzamide

Appendix C: Proton NMR Spectra  
Source: Laboratory Instructor

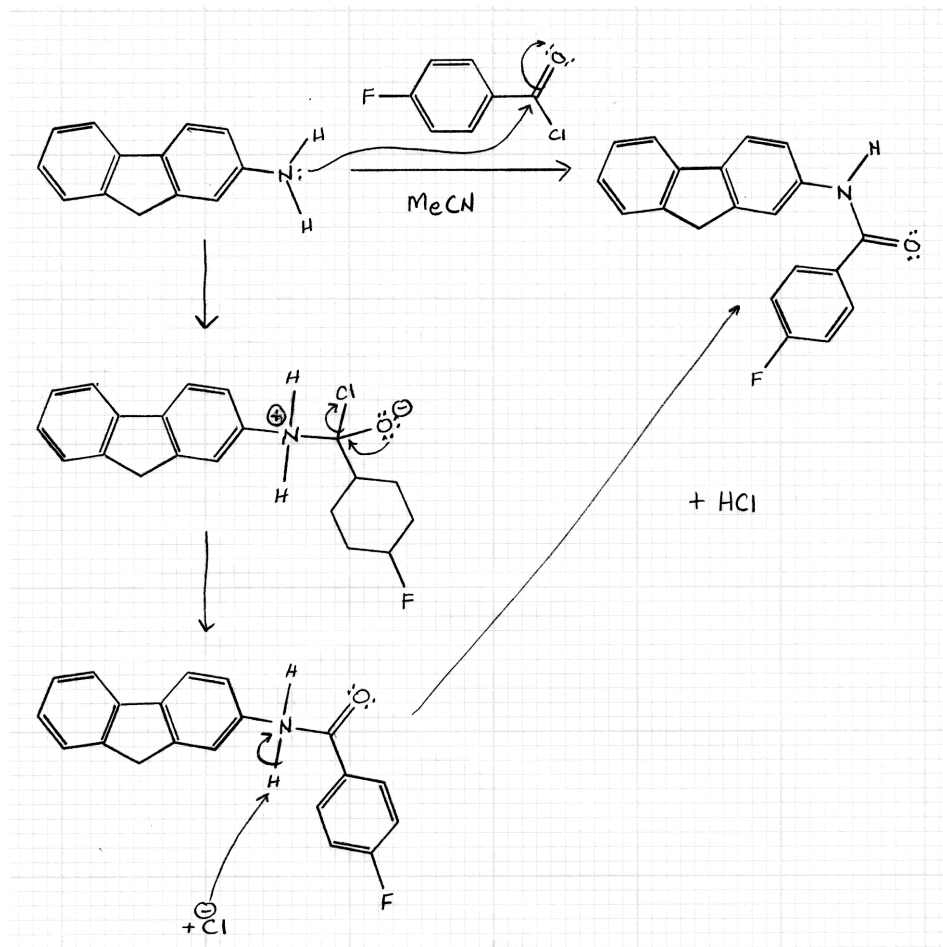


Appendix D: Fluorine NMR Spectra  
 Source: Laboratory Instructor

*Benzamide (w/F) <sup>19</sup>F NMR (expanded)*



Appendix E: Mechanism  
Source: Student Notes





**Appendix F: Experiment Laboratory Notes**  
Source: Student Notes





# Synthesis of 2-Iodofluorene from 2-Aminofluorene

## Summary

Sodium nitrite was reacted with p-toluenesulfonic acid to produce nitrous acid in situ. The amine group attached to the fluorene was attacked by the  $^+NO$  of nitrous acid to ultimately form a diazonium ion. The iodide of potassium iodide then displaced the diazonium group on the fluorene to produce 2-iodofluorene.

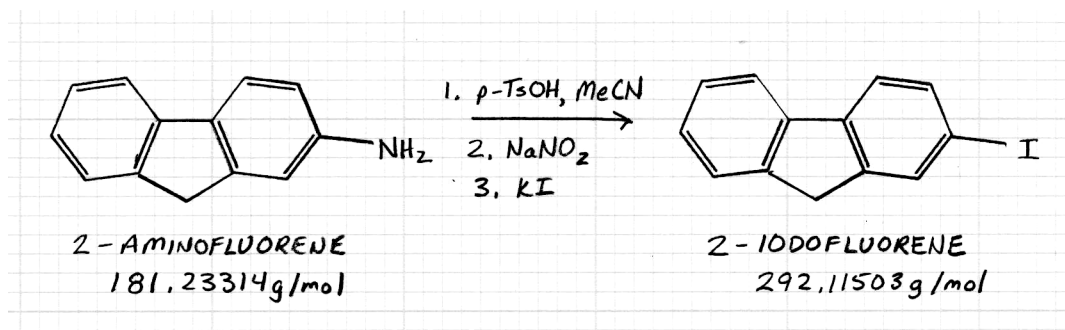


Fig 1: 2-Aminofluorene diazonium reaction to yield 2-iodofluorene

## Experimental Method and Observations

To a small Erlenmeyer flask was added 0.270 g (1.49 mmol) of 2-aminofluorene and 3 mL acetonitrile. The mixture was placed in an ice bath for a few minutes. To a small beaker was added 0.856 g (4.5 mmol) of p-toluenesulfonic acid (solid) and 3 mL acetonitrile. The beaker was then placed on mild heat to assist dissolution of the acid. The mixture in the Erlenmeyer flask was transferred to the beaker and stirred. The resulting mixture was placed in an ice bath. A reaction took place that gave an opaque, purple product.

A third mixture was prepared in a beaker with 0.205 g (2.97 mmol) of sodium nitrite with 2 mL of water. This mixture was placed in an ice bath. A fourth mixture was prepared in a beaker with 0.747 g (4.50 mmol) of potassium iodide and 2 mL of water. This was also placed in an ice bath. After a few minutes of cooling the sodium nitrite solution was added drop-wise to the 2-aminofluorene/p-toluenesulfonic acid mixture while stirring and while the acid mixture remained in the ice bath. The resulting reaction cause the product to turn dark red-brown color. Stirring continued for 10 minutes. The potassium iodide solution was then added to the mixture. The resulting suspension was placed on mild heat (held at 50 – 60° C) for 15 minutes with vigorous stirring. During this time N<sub>2</sub> gas was released.

A fifth solution was prepared in a beaker with 25 mL of water, 15.5 mL of 2M sodium sulfate, and 15.5 mL of 2N sodium hydroxide. After the N<sub>2</sub> gas release completed the 2-aminofluorene/p-toluenesulfonic acid suspension was added to this fifth mixture. The resulting product turned to a more brown color. The new mixture was stirred vigorously for a few minutes to form more precipitate.

The crude was boiled in 30 – 40 mL of distilled hexanes to dissolve the product then filtered through a 4 – 5 cm silica gel plug with vacuum filtration. The resulting organic solution was clear violet. The organic layer was separated in a separatory funnel with 18 mL of aqueous sodium sulfite. Afterwards, it was dried with anhydrous sodium sulfate. The organic layer was rotary evaporated to remove the distilled hexanes.

The product was dried in open atmosphere for one full week. A TLC was performed with pure hexanes. No crude or final product photographs were obtained.

## Discussion and Results

Thin layer chromatography results depict a product that differs from the original. The  $R_f$  for 2-aminofluorene was 0.60 while the  $R_f$  for 2-iodofluorene was 0.29.

The melting point obtained 125.6 – 127° C agrees with experimental results 128° C for 2-iodofluorene as reported in literature[5]. The range obtained suggests a purity better than 95%. The obtained value marginally differs from the melting point of the reagent, 2-aminofluorene, which was reported as 123 – 124° C above. Melting point in this instance is not sufficient alone to differentiate product from reagent.

The IR spectrum of the product ([Appendix A: IR Spectra](#)) shows standard SP3 C–H peaks, standard SP2 C–H peaks and standard benzene peaks. Two peaks of interest are displayed on the plot: a C=C peak at wavenumber 1660.99 and an unidentified peak at 3298.22. The unknown peak has characteristics similar to an OH group. A great deal of water was used in this experiment. A second possibility is that unreacted residue (containing an RNH<sub>2</sub> group) from the reagent caused the peak; however, an RNH<sub>2</sub> group would have showed as two more narrow peaks, rather than a broad peak. The most plausible explanation is the presence of water. While this author's procedure was to dry each compound for a week in open atmosphere prior to obtaining experimental data, perhaps the IR was obtained before the drying period in this instance.

The proton NMR spectrum of the product ([Appendix B: Proton NMR Spectra](#)) shows a number of unremarkable peaks indicative of benzene rings. There is also one peak that accords with the two central hydrogens between the two rings.

The carbon NMR spectrum of the product ([Appendix C: Carbon NMR Spectra](#)) shows the required 13 peaks. There are two confirmed peaks: one is the central carbon between the rings, the other is the carbon to which is attached the iodine. The remainder are predictions based on calculations given in the following table (carbon positions are assigned in [Appendix C](#)). The predictions are not certain due to the number of peaks and close proximity of values:

**Table 1: Benzene Carbon NMR Predictive Values vs Experimental Values (given in ppm)**

Position	Benzene	I	Ph	COPh	Predicted	Experimental
'1'	128.5	–32.0 (i)	–1.2 (p)	–0.2 (m)	95.1	92.48
'2'	128.5	10.2 (o)	0.4 (m)	3.6 (p)	142.7	142.63
'3'	128.5	2.9 (m)	–1.1 (o)	–0.2 (m)	130.1	125.13
'4'	128.5	–1.1 (p)	13.1 (i)	1.7 (o)	142.2	149.22
'5'	128.5	–	13.1 (i)	1.7 (o)	143.3	140.76
'6'	128.5	–	–1.1 (o)	–0.2 (m)	127.2	122.06
'7'	128.5	–	0.4 (m)	3.6 (p)	132.5	127.38
'8'	128.5	–	–1.2 (p)	–0.2 (m)	127.1	120.26
'9'	128.5	–	0.4 (m)	1.7 (o)	130.6	130.04
'10'	128.5	–	–1.1 (o)	9.4 (i)	136.8	133.95
'12'	128.5	2.9 (m)	–1.1 (o)	9.4 (i)	139.7	135.38
'13'	128.5	10.2 (o)	0.4 (m)	1.7 (o)	140.8	140.15

The molar quantity of the primary reagent 2-aminofluorene ( $C_{13}H_{11}N$ ) was:

$$mol\ C_{13}H_{11}N = 0.270\ g\ C_{13}H_{11}N * \frac{1\ mol\ C_{13}H_{11}N}{181.23314\ g\ C_{13}H_{11}N} = 0.00149\ mol\ C_{13}H_{11}N$$

The molar mass of the yielded product (2-iodofluorene) is 292.11503 g per mole. The number of moles yielded is:

$$mol\ C_{13}H_9I = 0.195\ g\ C_{13}H_9I\ (collected) * \frac{1\ mol\ C_{13}H_9I}{292.11503\ g\ C_{13}H_9I} = 0.000668\ mol\ C_{13}H_9I$$

The percent yield is:

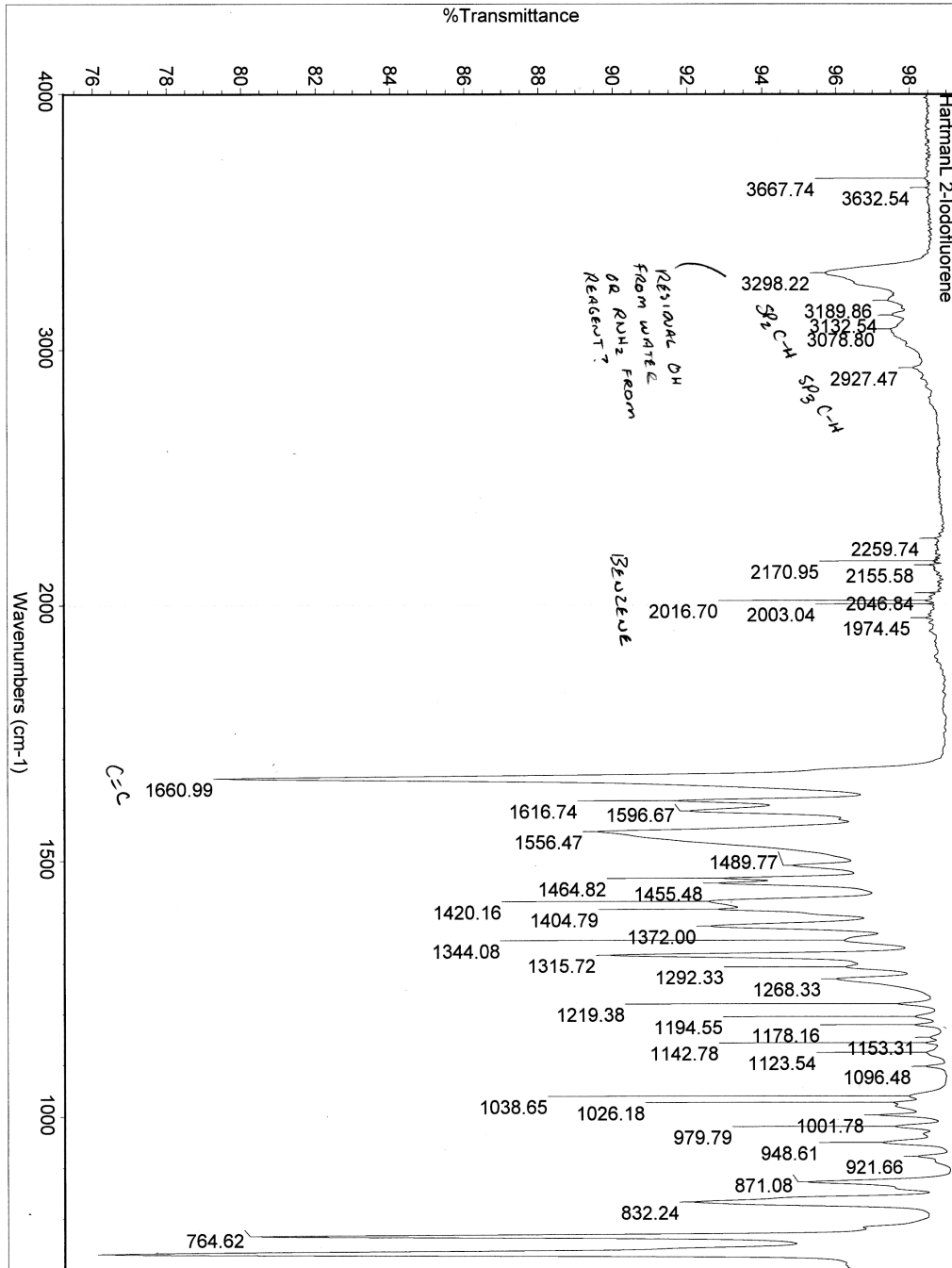
$$\begin{aligned} \% - yield\ C_{13}H_9I &= 0.000668\ mol\ C_{13}H_9I * \frac{1\ mol\ C_{13}H_{11}N}{1\ mol\ C_{13}H_9I} * \frac{1}{0.00149\ mol\ C_{13}H_{11}N} * 100\% \\ &= 44.8\% \end{aligned}$$

The yield is realistic for laboratory conditions.

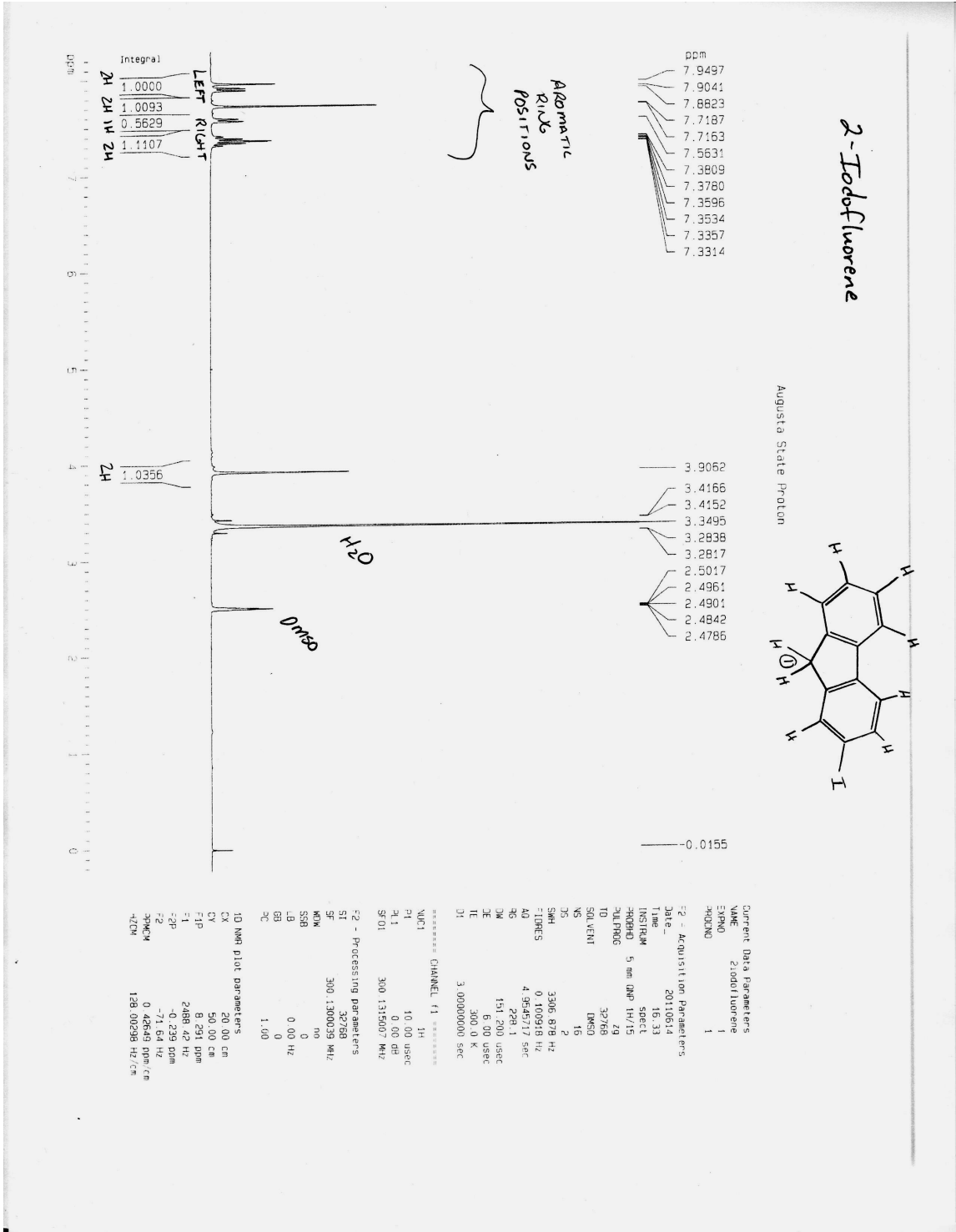
## Conclusion

The product formed was 2-iodofluorene, with a reasonable yield. Purity of excellent quality was confirmed by an obtained melting point range that encompassed the experimental value reported in literature[5]. With the exception of the troublesome OH peak, IR analysis confirms product identity.

Appendix A: IR Spectra  
 Source: Student Notes



Appendix B: Proton NMR Spectra  
Source: Laboratory Instructor





**Appendix D: Experiment Laboratory Notes**  
Source: Student Notes

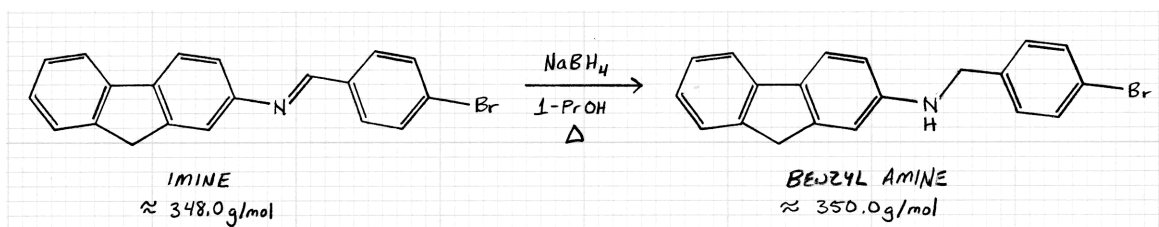




# Synthesis of Benzyl Amine from Imine

## Summary

N-[(4-bromophenyl)methyl]-9H-fluorene-2-imine (fluorene-derived imine) was reduced by sodium borohydride to produce N-[(4-bromophenyl)methyl]-9H-fluorene-2-amine. The solvent 1-PrOH was chosen to allow the reaction to reach a higher boiling point and therefore quicken the pace of the reaction.



**Fig 1:** Imine reduction reaction to yield N-[(4-bromophenyl)methyl]-9H-fluorene-2-amine

## Experimental Method and Observations

This reaction was scaled to 83% of lab manual specifications due to inadequate quantity of the imine. The following compounds were transferred into a clean, dry round bottom flask: 0.216 g (0.62 mmol) of N-[(4-bromophenyl)methyl]-9H-fluorene-2-imine, 0.115 g (3.03 mmol) of sodium borohydride and 5 mL propanol. The mixture was stirred and heated with reflux for a total of 30 minutes. An initial TLC was performed with 10% ethyl acetate in hexanes to verify targeted product was formed.

A few ml of water was added and the mixture was placed in an ice bath for a few minutes to form precipitate. Afterwards the crude was vacuum filtered and washed with water. It was then recrystallized from methanol. A small amount of water was used to induce recrystallization.

After the crude was dried in open atmosphere for one full week, a final TLC was performed with 10% ethyl acetate in hexanes. The final product was pale yellow in color ([Appendix A: Final Product Photograph](#)).

## Discussion and Results

Thin layer chromatography results depict a product that differs from the original. The  $R_f$  for imine was 0.65 while the  $R_f$  for N-[(4-bromophenyl)methyl]-9H-fluorene-2-amine was 0.79.

The melting point obtained 91.6 – 92.6° C. No experimental values stemming from literature searches were discovered to corroborate obtained value. The obtained value differs from the melting point of the reagent, imine, which was reported as 184 – 185.2° C above.

The IR spectrum of the product ([Appendix B: IR Spectra](#)) shows standard SP3 C–H peaks, standard SP2 C–H peaks and standard benzene peaks. Two peaks of interest are displayed on the plot that can substantiate product identification: an  $R_2NH$  peak at wavenumber 3404.03 and a C=C peak at wavenumber 1612.49.

The proton NMR spectrum of the product (*Appendix C: Proton NMR Spectra*) shows a number of unremarkable peaks indicative of benzene rings. There are also two peaks that accord with the two central hydrogens between the two rings and the SP<sup>3</sup> C–H. The peak belonging to the hydrogen attached to the nitrogen is very likely hidden among those of the benzene rings.

The proposed mechanism for N-[(4-bromophenyl)methyl]-9H-fluoren-2-amine is presented in *Appendix D: Mechanism*. There is some preliminary interaction between the sodium borohydride and alcohol to release the nucleophilic hydrogen. The primary reaction proceeds with the nucleophilic hydrogen attacking the imine. The byproduct is the ionic molecule NaEtOBH<sub>3</sub>.

The molar quantity of the primary reagent N-[(4-bromophenyl)methyl]-9H-fluor-2-imine (C<sub>20</sub>H<sub>14</sub>BrN) was:

$$\text{mol C}_{20}\text{H}_{14}\text{BrN} = 0.216 \text{ g C}_{20}\text{H}_{14}\text{BrN} * \frac{1 \text{ mol C}_{20}\text{H}_{14}\text{BrN}}{348.0 \text{ g C}_{20}\text{H}_{14}\text{BrN}} = 0.000621 \text{ mol C}_{20}\text{H}_{14}\text{BrN}$$

The molar mass of the yielded product (N-[(4-bromophenyl)methyl]-9H-fluoren-2-amine) is 350.25 g per mole. The number of moles yielded is:

$$\text{mol C}_{20}\text{H}_{16}\text{BrN} = 0.041 \text{ g C}_{20}\text{H}_{16}\text{BrN (collected)} * \frac{1 \text{ mol C}_{20}\text{H}_{16}\text{BrN}}{350.25 \text{ g C}_{20}\text{H}_{16}\text{BrN}} = 0.000117 \text{ mol C}_{20}\text{H}_{16}\text{BrN}$$

The percent yield is:

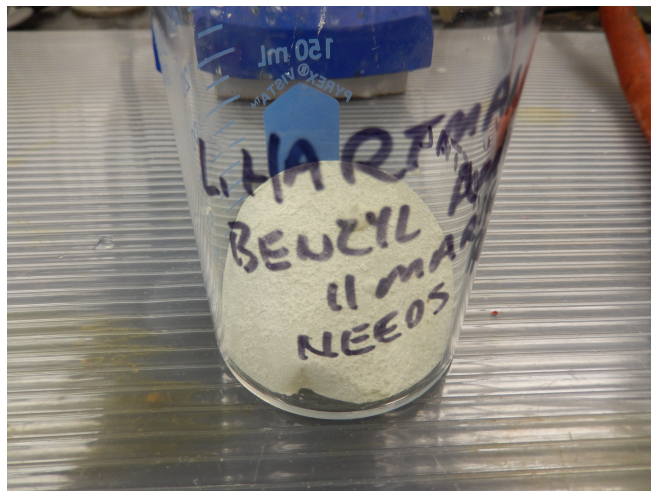
$$\begin{aligned} \% \text{ - yield C}_{20}\text{H}_{16}\text{BrN} &= 0.000117 \text{ mol C}_{20}\text{H}_{16}\text{BrN} * \frac{1 \text{ mol C}_{20}\text{H}_{14}\text{BrN}}{1 \text{ mol C}_{20}\text{H}_{16}\text{BrN}} * \frac{1}{0.000621 \text{ mol C}_{20}\text{H}_{14}\text{BrN}} * 100\% \\ &= 18.9\% \end{aligned}$$

The yield is low. No difficulties were noted during the experiment.

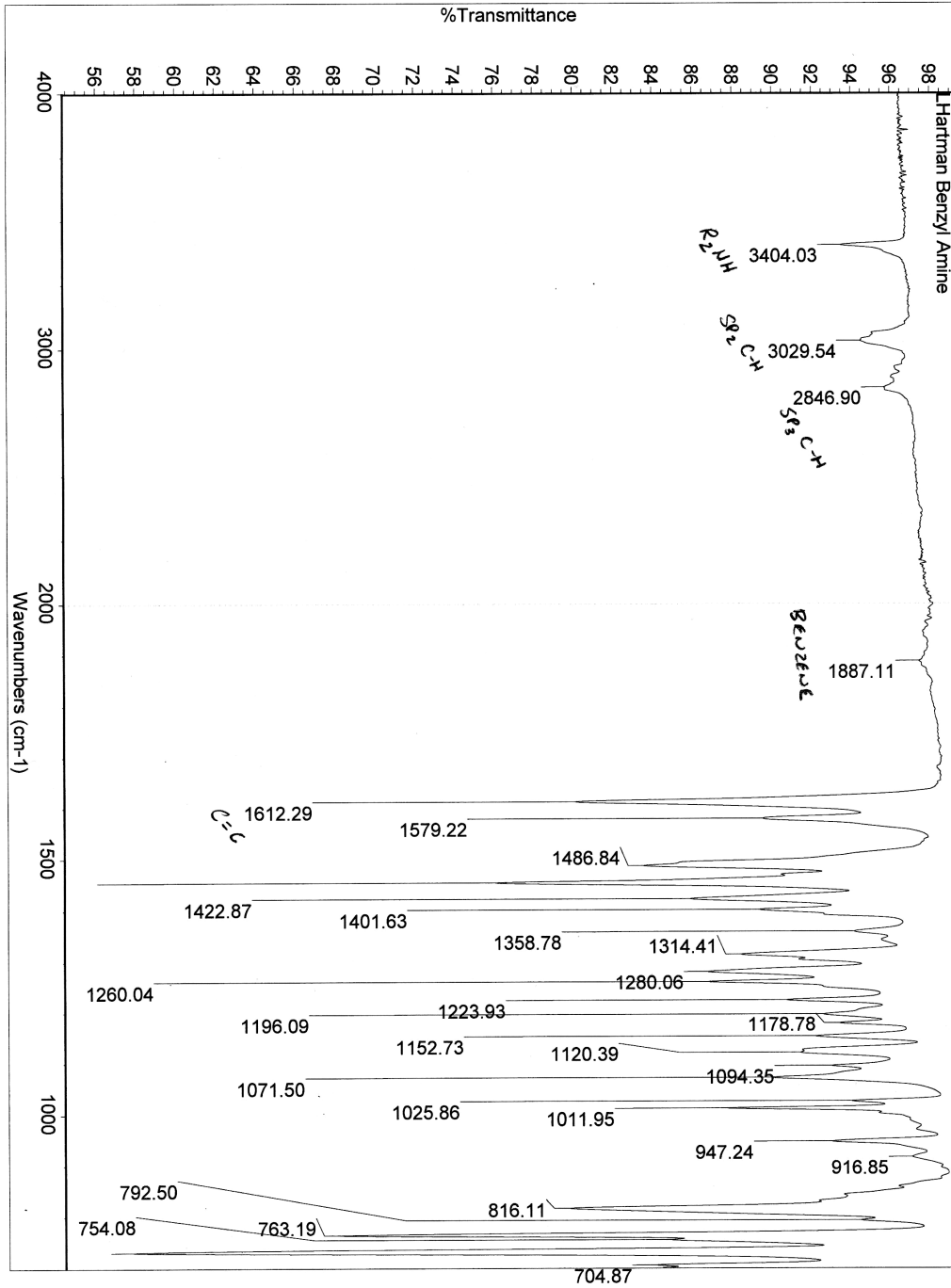
## Conclusion

The product formed was likely N-[(4-bromophenyl)methyl]-9H-fluoren-2-amine, with a low yield. IR spectrum analysis does not refute this conclusion. Without further data—an experimental melting point from literature would be useful—certainty can not be ascertained.

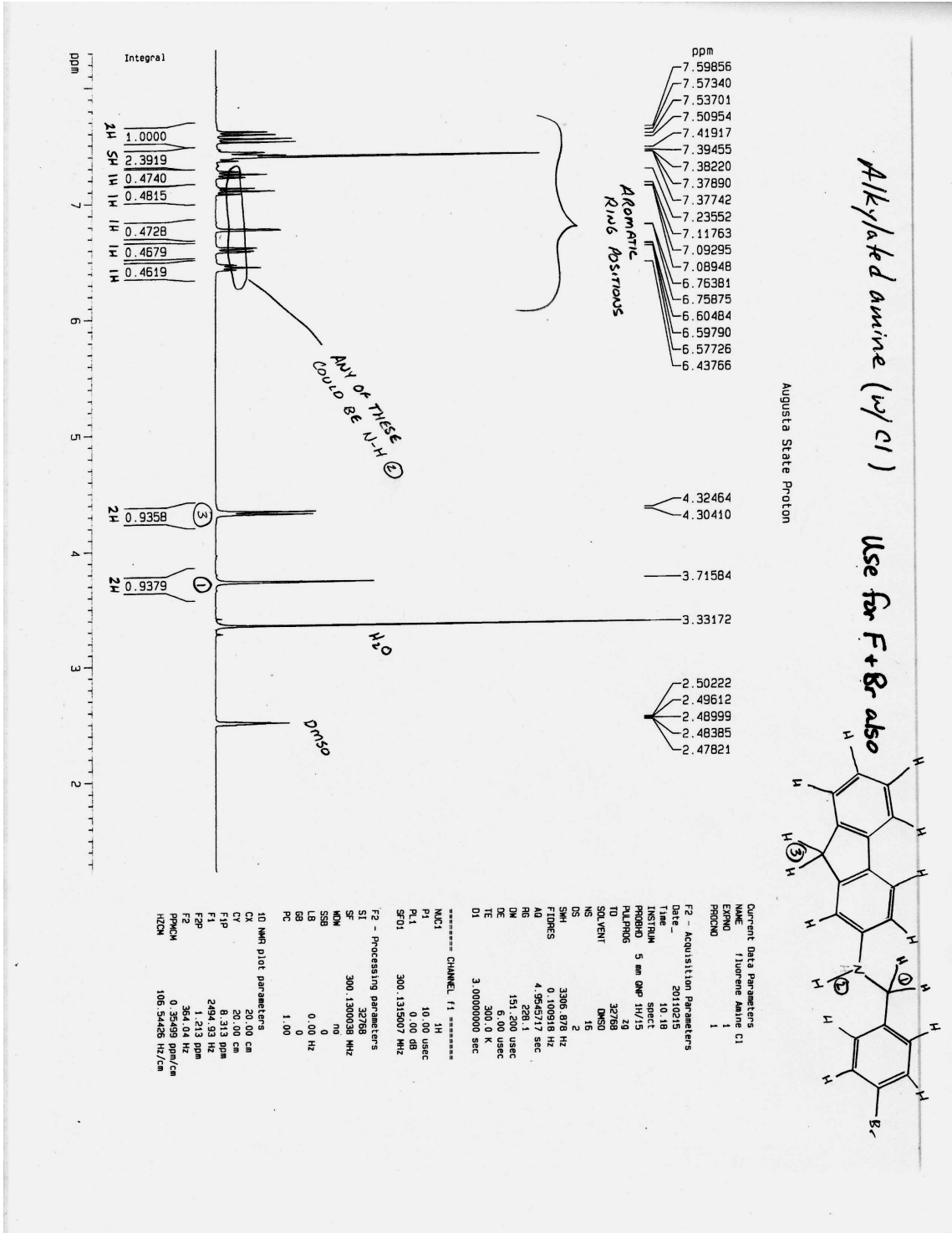
**Appendix A: Final Product Photograph**  
Source: Student Notes



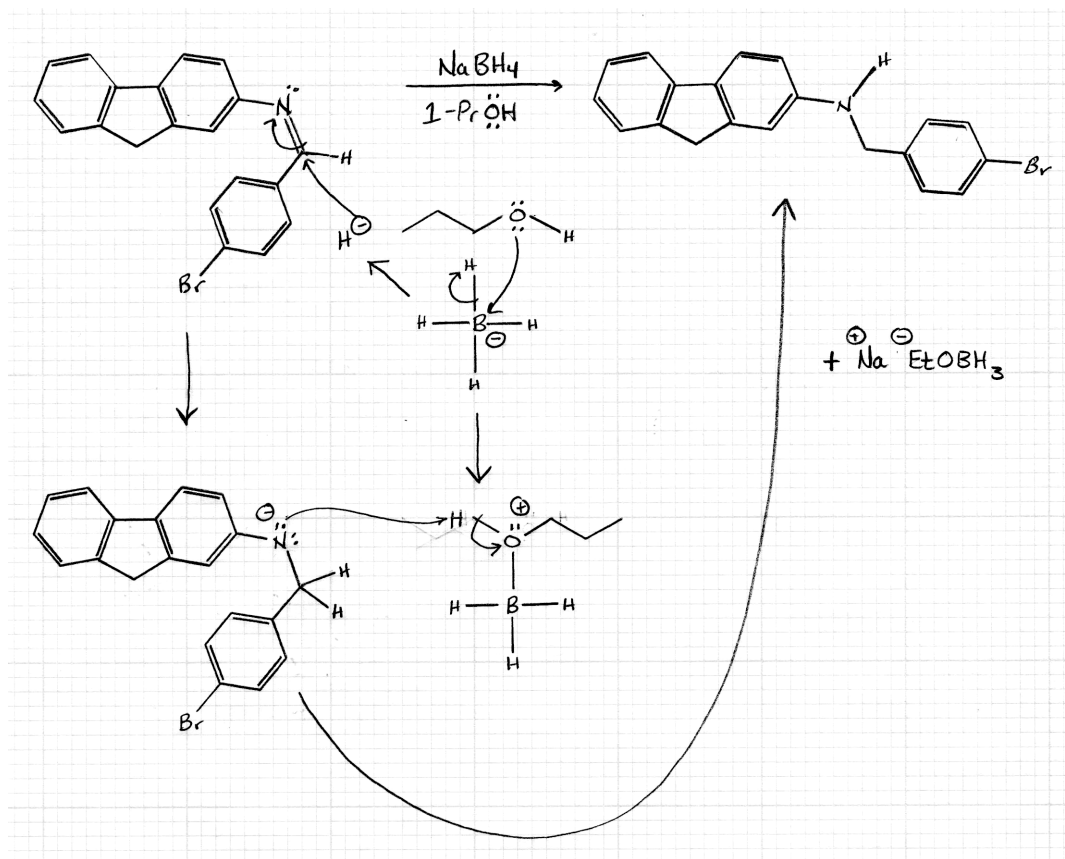
Appendix B: IR Spectra  
Source: Student Notes



Appendix C: Proton NMR Spectra  
Source: Laboratory Instructor



Appendix D: Mechanism  
Source: Student Notes



**Appendix E: Experiment Laboratory Notes**  
Source: Student Notes





# Synthesis of Suzuki Product from 2-Iodofluorene

## Summary

Palladium(II) was reduced in situ in the presence of 4-cyanobenzeneboronic acid to Pd(0). 2-Iodofluorene was then catalyzed by Pd(0) to produce an aryl-aryl reaction where the cyanobenzene ring replaced the iodine on the fluorene structure to produce 4-(9H-fluoren-2-yl)-benzonitrile.

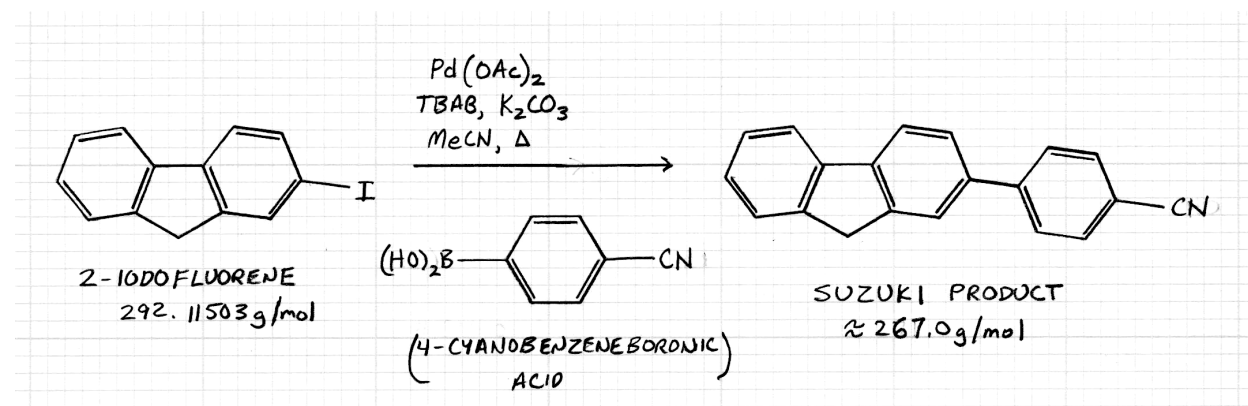


Fig 1: 2-Iodofluorene Suzuki reaction to yield 4-(9H-fluoren-2-yl)-benzonitrile

## Experimental Method and Observations

The following compounds were transferred into a clean, dry round bottom flask: 0.148 g (0.506 mmol) of 2-iodofluorene, 0.089 g (0.606 mmol) of 4-cyanophenylboronic acid, 0.138 g (0.999 mmol) of potassium carbonate, 0.800 g (2.48 mmol) tetrabutylammonium bromide, 5 mL acetonitrile, 1 mL water. The mixture was stirred and heated with reflux for a total of 15 minutes. At this point a double check of the lab notebook showed a reactant was missed. To the solution 0.005 g (0.022 mmol) palladium (II) acetate was added, heat was intensified, and the solution refluxed for an additional 25 minutes.

After reflux, water was added and the mixture was chilled in ice bath to precipitate the product. The crude was vacuum filtered then recrystallized from ethanol two times to enhance purity.

After the crude was dried in open atmosphere for one full week, a TLC was performed with pure hexanes.

## Discussion and Results

Thin layer chromatography results depict a product that differs from the original. The  $R_f$  for 2-iodofluorene was 0.32 while the  $R_f$  for 4-(9H-fluoren-2-yl)-benzonitrile was 0.09.

The melting point obtained 224 – 226° C. No experimental values stemming from literature searches were discovered to corroborate obtained value. The obtained value differs from the melting point of the reagent, 2-iodofluorene, which was reported as 125.6 – 127° C above.

The IR spectrum of the product (*Appendix B*) shows standard SP3 C–H peaks, standard SP2 C–H peaks and standard benzene peaks. Two peaks of interest are displayed on the plot that can substantiate product identification: a C≡N peak at wavenumber 2221.48 and a C=C peak at wavenumber 1648.76.

The molar quantity of the primary reagent 2-iodofluorene (C<sub>13</sub>H<sub>9</sub>I) was:

$$\text{mol C}_{13}\text{H}_9\text{I} = 0.148 \text{ g C}_{13}\text{H}_9\text{I} * \frac{1 \text{ mol C}_{13}\text{H}_9\text{I}}{292.11503 \text{ g C}_{13}\text{H}_9\text{I}} = 0.000507 \text{ mol C}_{13}\text{H}_9\text{I}$$

The molar mass of the yielded product (4-(9H-fluoren-2-yl)-benzotrile) is 267.32 g per mole. The number of moles yielded is:

$$\text{mol C}_{20}\text{H}_{13}\text{N} = 0.046 \text{ g C}_{20}\text{H}_{13}\text{N (collected)} * \frac{1 \text{ mol C}_{20}\text{H}_{13}\text{N}}{267.32 \text{ g C}_{20}\text{H}_{13}\text{N}} = 0.000172 \text{ mol C}_{20}\text{H}_{13}\text{N}$$

The percent yield is:

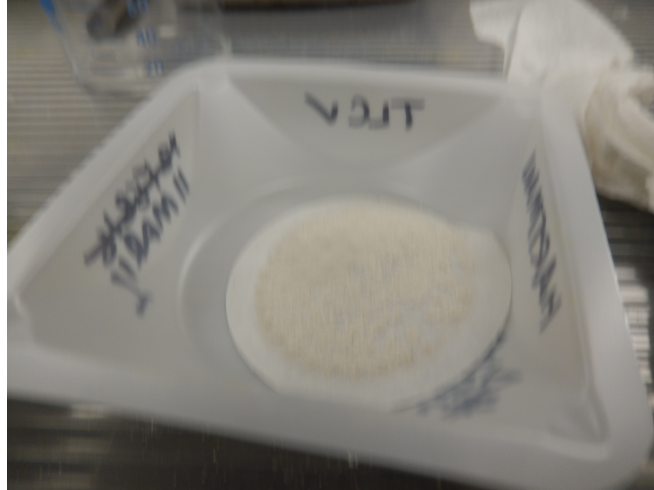
$$\begin{aligned} \% \text{ - yield C}_{20}\text{H}_{13}\text{N} &= 0.000172 \text{ mol C}_{20}\text{H}_{13}\text{N} * \frac{1 \text{ mol C}_{13}\text{H}_9\text{I}}{1 \text{ mol C}_{20}\text{H}_{13}\text{N}} * \frac{1}{0.000507 \text{ mol C}_{13}\text{H}_9\text{I}} * 100\% \\ &= 34.0\% \end{aligned}$$

The yield is slightly low. The low yield was possibly a result of the delayed step noted above.

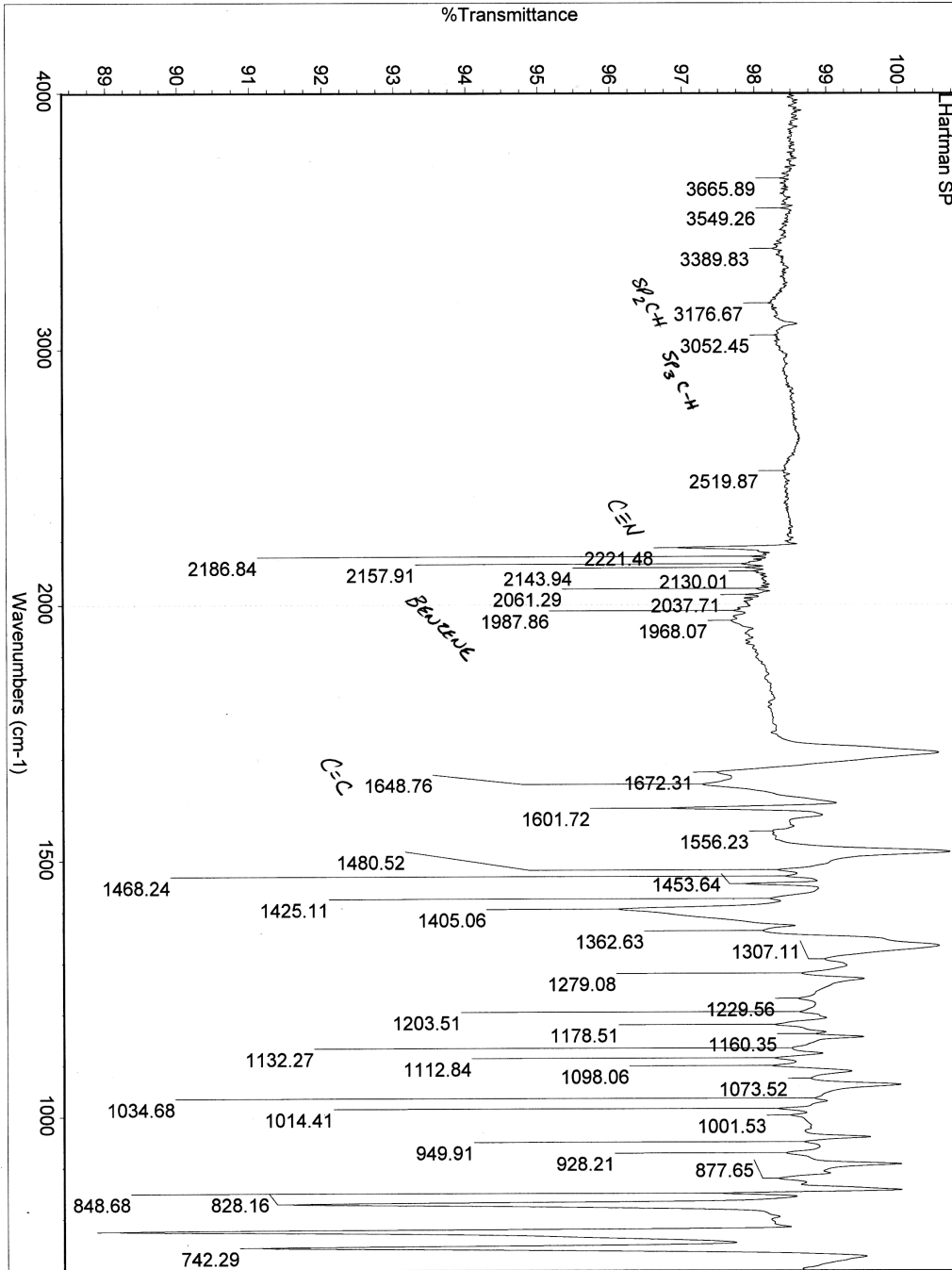
## Conclusion

The product formed was likely 4-(9H-fluoren-2-yl)-benzotrile, with a slightly low yield. IR spectrum analysis does not refute this conclusion. Without further data—an experimental melting point from literature would be useful—certainty can not be ascertained.

**Appendix A: Final Product Photograph**  
Source: Student Notes



Appendix B: IR Spectra  
 Source: Student Notes



**Appendix C: Experiment Laboratory Notes**  
Source: Student Notes



## Author Information

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## References

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## Annex 1: General Chemical Properties and Safety Data

(presented in alphabetical order)

### 1-propanol

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/1031>

- \* CAS: 71-23-8
- \*  $C_3H_8O$
- \* 60.09502 g per mole
- \* Boiling Point: 97.2° C
- \* Density: 0.8053 g/cm<sup>3</sup>
- \* Solubility: Miscible in water
- \* pKa: 16.10
- \* Clear colorless liquid
- \* Alcohol-like odor
- \* Highly flammable liquid and vapor
- \* Causes serious eye damage
- \* May cause drowsiness and dizziness
- \* May cause nausea

### 2-acetamidofluorene

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/5897>

- \* CAS: 53-96-3
- \*  $C_{15}H_{13}NO$
- \* 223.26982 g per mole
- \* Melting Point: 194° C
- \* Solubility: Soluble in ether, AcOH
- \* Tan crystalline powder
- \* Absorbed via inhalation, ingestion, and skin contact
- \* Reduced function of liver, kidneys, bladder, pancreas
- \* Carcinogen, mutagen

### 2-aminofluorene

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/1539>

- \* CAS: 153-78-6
- \*  $C_{13}H_{11}N$
- \* 181.23314 g per mole
- \* Melting Point: 131 – 132° C
- \* Solubility: Soluble in alcohol
- \* Brown crystal powder
- \* Toxic fumes when heated
- \* Possible carcinogen

### 2-iodofluorene

Sources:

<https://pubchem.ncbi.nlm.nih.gov/compound/224094>,  
<http://www.sigmaaldrich.com/catalog/product/aldrich/653918>

- \* CAS: 2523-42-4
- \*  $C_{13}H_9I$
- \* 292.11503 g per mole
- \* Melting Point: 126 – 129° C
- \* Causes eye irritation

### 2-nitrofluorene

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/11831>

- \* CAS: 607-57-8
- \*  $C_{13}H_9NO_2$
- \* 211.21606 g per mole
- \* Melting Point: 158° C
- \* Solubility: Soluble in Acetone
- \* Cream colored solid
- \* Toxic
- \* Carcinogen

### 2-nitrofluorenone

Sources:

<https://pubchem.ncbi.nlm.nih.gov/compound/18356>,

<http://www.sigmaaldrich.com/catalog/product/aldrich/299758>

- \* CAS: 3096-52-4
- \*  $C_{13}H_7NO_3$
- \* 225.19958 g per mole
- \* Melting Point: 222 – 223° C
- \* Density: 1.0446 g/cm<sup>3</sup>
- \* Causes skin irritation
- \* Causes eye irritation
- \* Causes respiratory irritation

### 2-propanol (isopropanol)

Sources:

<https://pubchem.ncbi.nlm.nih.gov/compound/3776>

- \* CAS: 67-63-0
- \*  $C_3H_8O$
- \* 60.09502 g per mole
- \* Boiling Point: 82.3° C
- \* Density: 0.78509 g/cm<sup>3</sup>
- \* Solubility: Miscible in alcohol, ether, chloroform; soluble in benzene
- \* Colorless liquid
- \* Alcohol odor
- \* Highly flammable liquid and vapor
- \* Causes serious eye irritation
- \* Causes drowsiness and dizziness
- \* Causes damage to organs with prolonged exposure
- \* Causes irritation to respiratory tract
- \* Fire produces corrosive, toxic gases

### 4-(9H-fluoren-2-yl)-benzonitrile

Source:

<https://scifinder.cas.org>

- \* CAS: 404574-92-1
- \*  $C_{20}H_{13}N$
- \* 267.32 g per mole
- \* No further physical data found
- \* No safety data found

### 4-bromobenzaldehyde

Sources:

<https://pubchem.ncbi.nlm.nih.gov/compound/70741>

<http://www.sigmaaldrich.com/catalog/product/aldrich/b57400>

- \* CAS: 1122-91-4
- \*  $C_7H_5BrO$
- \* 185.018 g per mole
- \* Melting Point: 55 – 58° C
- \* Harmful if swallowed
- \* Causes skin irritation
- \* May cause allergic skin reaction
- \* Causes severe eye irritation
- \* May cause respiratory irritation

### 4-cyanobenzeneboronic acid

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/2734326>

- \* CAS: 1266747-17-6
- \*  $C_7H_6BNO_2$
- \* 146.93904 g per mole
- \* No further physical data found
- \* No safety data found

### 4-fluorobenzoyl chloride

Source:

<http://www.sigmaaldrich.com/catalog/product/aldrich/119946>

- \* CAS: 403-43-0
- \*  $C_7H_4ClFO$
- \* 158.557463 g per mole
- \* Melting Point: 10 – 12° C
- \* Boiling Point: 82° C
- \* Density: 1.342 g/cm<sup>3</sup>
- \* No safety data found

### N-[(4-bromophenyl)methyl]-9H-fluoren-2-amine

Source:

<https://scifinder.cas.org>

- \* CAS: 38179-98-5
- \*  $C_{20}H_{16}BrN$
- \* 350.25 g per mole
- \* No further physical data found
- \* No safety data found

### N-9H-fluoren-3-yl-4-fluoro-benzamide

Source:

<https://scifinder.cas.org>

- \* CAS: 1007752-95-5
- \*  $C_{20}H_{14}FNO$
- \* 303.33 g per mole
- \* No further physical data found
- \* No safety data found

### acetic acid

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/176>

- \* CAS: 64-19-7
- \*  $C_2H_4O_2$
- \* 60.05196 g per mole
- \* Boiling Point: 117.9° C
- \* Density: 1.0446 g/cm<sup>3</sup>
- \* Colorless liquid, pungent odor
- \* Flammable liquid/vapor
- \* Harmful if inhaled
- \* Harmful if icontact with skin
- \* May be harmful if swallowed
- \* Causes severe skin/eye damage
- \* May cause respiratory irritation

### acetic anhydride

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/7918>

- \* CAS: 108-24-7
- \*  $C_4H_6O_3$
- \* 102.08864 g per mole
- \* Boiling Point: 139.0 – 139.5° C
- \* Density: 1.082 g/cm<sup>3</sup>
- \* Solubility: Soluble in benzene; miscible in ethanol, ether, AcOH, and ethyl acetate
- \* Colorless liquid
- \* Pungent odor
- \* Flammable liquid and vapor
- \* Harmful if swallowed
- \* Causes severe skin and eye burns
- \* Causes severe burns on skin with wet clothing
- \* Causes bronchitis

### acetonitrile

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/6342>

- \* CAS: 75-05-8
- \*  $C_2H_3N$
- \* 41.05192 g per mole
- \* Boiling Point: 81.6° C
- \* Density: 0.78745 g/cm<sup>3</sup> @ 15° C
- \* pKa: -4.30
- \* Solubility: Soluble in alcohol, water; miscible in acetone
- \* Colorless liquid
- \* Highly flammable liquid and vapors
- \* Toxic in skin contact
- \* Toxic if inhaled
- \* Harmful if swallowed
- \* Severe eye irritation
- \* Damage to blood with prolonged, repeated exposure

### ammonium chloride

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/25517>

- \* CAS: 12125-02-9
- \*  $NH_4Cl$
- \* 53.49146 g per mole
- \* Melting Point: 350° C
- \* Density: 1.5 g/cm<sup>3</sup>
- \* Solubility: Soluble in methanol
- \* Colorless to white powder
- \* odorless
- \* Harmful if swallowed
- \* Causes serious eye irritation
- \* Causes respiratory system irritation
- \* Causes nausea

### diethyl ether

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/3283>

- \* CAS: 60-29-7
- \*  $C_4H_{10}O$
- \* 74.1216 g per mole
- \* Boiling Point: 34.6° C
- \* Density: 0.7134 g/cm<sup>3</sup> @ 15° C
- \* Solubility: Miscible in most organic solvents
- \* Colorless liquid, sweet odor
- \* Extremely flammable liquid and vapor
- \* Harmful if swallowed
- \* May be fatal if swallowed and enters airways
- \* Causes mild skin irritation
- \* Causes eye irritation
- \* May cause respiratory irritation
- \* Suspected of damaging fertility and unborn child

### *dimethyl sulfoxide*

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/679>

- \* CAS: 67-68-5
- \*  $C_2H_6OS$
- \* 78.13344 g per mole
- \* Melting Point: 18.45° C
- \* Boiling Point: 189° C
- \* Density: 1.101 g/cm<sup>3</sup> @ 15° C
- \* Solubility: Soluble in methanol and acetone; miscible in water
- \* Colorless liquid, odorless
- \* Flammable
- \* Produces toxic fumes
- \* Explosive vapor at 87° C
- \* Causes skin irritation
- \* Causes headaches, nausea
- \* Causes blurred vision
- \* Causes drowsiness

### *ethanol*

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/702>

- \* CAS: 64-17-5
- \*  $C_2H_6O$
- \* 46.06844 g per mole
- \* Boiling Point: 78.29° C
- \* Density: 0.7893 g/cm<sup>3</sup> @ 20° C
- \* Solubility: Miscible in water
- \* Colorless liquid with fragrant odor
- \* Highly flammable liquid and vapor
- \* Irritation to skin, eyes, and nose
- \* May cause headaches and drowsiness
- \* May induce coughing

### *fluorene*

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/6853>

- \* CAS: 86-73-7
- \*  $C_{13}H_{10}$
- \* 166.2185 g per mole
- \* Melting Point: 116 – 117° C
- \* Density: 1.203 g/cm<sup>3</sup>
- \* Solubility: Soluble in AcOH
- \* White leaflets or flakes
- \* Combustible

### *hydrochloric acid*

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/313>

- \* CAS: 7647-01-1
- \* HCl
- \* 36.46094 g per mole
- \* Density: 1.639 g/cm<sup>3</sup>
- \* Causes severe skin and eye burns
- \* Toxic if inhaled
- \* Corrosive

### *lutidine*

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/7937>

- \* CAS: 108-48-5
- \*  $C_7H_9N$
- \* 107.15306 g per mole
- \* Boiling Point: 144° C
- \* Density: 0.9252 g/cm<sup>3</sup>
- \* Oily liquid
- \* Flammable liquid and vapor
- \* Harmful if swallowed
- \* Respiratory irritation, headaches, skin irritation

### *methanol*

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/887>

- \* CAS: 67-56-1
- \*  $\text{CH}_4\text{O}$
- \* 32.04186 g per mole
- \* Boiling Point:  $64.7^\circ\text{C}$
- \* Density:  $0.8100\text{ g/cm}^3$  @  $20^\circ\text{C}$
- \* Solubility: Miscible in ethanol, ether, benzene
- \* Colorless liquid with fragrant odor
- \* Highly flammable liquid and vapor
- \* Harmful if swallowed
- \* Serious irritation to skin, eyes, and nose
- \* May damage fertility or unborn child
- \* Causes damage to organs: central nervous system, gastrointestinal tract
- \* Causes headaches, dizziness, drowsiness, and nausea
- \* Causes optic nerve damage

### *nitric acid*

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/944>

- \* CAS: 7697-37-2
- \*  $\text{HNO}_3$
- \* 63.01284 g per mole
- \* Boiling Point:  $83^\circ\text{C}$
- \* Density:  $1.5129\text{ g/cm}^3$  @  $25^\circ\text{C}$
- \* pKa: -1.38
- \* Solubility: Miscible in water
- \* Colorless to yellow liquid
- \* Pungent odor
- \* Corrosive to metals
- \* Fatal if swallowed
- \* Causes severe skin and eye burns
- \* Causes damage to respiratory tract if inhaled
- \* Causes damage to digestive tract if ingested
- \* Oxidizer, may intensify fire
- \* Causes dental erosion

### *p-toluenesulfonic acid*

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/6101>

- \* CAS: 104-15-4
- \*  $\text{C}_7\text{H}_8\text{O}_3\text{S}$
- \* 172.20162 g per mole
- \* Melting Point:  $106 - 107^\circ\text{C}$
- \* Density:  $1.24\text{ g/cm}^3$
- \* Solubility: Very soluble in water
- \* Colorless to black dry powder
- \* Odorless
- \* Harmful if swallowed
- \* Causes severe skin and eye burns
- \* Causes respiratory irritation
- \* Corrosive
- \* Combustible

### *palladium (II) acetate*

Sources:

<https://pubchem.ncbi.nlm.nih.gov/compound/167845>

<http://www.sigmaaldrich.com/catalog/product/aldrich/379875>

- \* CAS: 3375-31-3
- \*  $\text{C}_4\text{H}_6\text{O}_4\text{Pd}$
- \* 224.50804 g per mole
- \* Melting Point:  $216.3 - 223.7^\circ\text{C}$
- \* Causes serious eye damage

### *potassium carbonate*

Sources:

<https://pubchem.ncbi.nlm.nih.gov/compound/11430>

- \* CAS: 584-08-7
- \*  $K_2CO_3$
- \* 138.2055 g per mole
- \* Melting Point: 899° C
- \* Density: 2.29 g/cm<sup>3</sup>
- \* Dry powder
- \* Odorless
- \* Causes throat and chest irritation
- \* May cause respiratory tract irritation
- \* May cause skin and eye irritation
- \* Reacts violently in acids and chlorine trifluoride
- \* Reacts with powdered metals

### *potassium iodide*

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/4875>

- \* CAS: 7681-11-0
- \* KI
- \* 166.00277 g per mole
- \* Melting Point: 681° C
- \* Density: 3.12 g/cm<sup>3</sup>
- \* Colorless to white powder
- \* Causes eye irritation
- \* Irritates open wounds

### *potassium phosphate*

Sources:

<https://pubchem.ncbi.nlm.nih.gov/compound/62657>

<http://www.sigmaaldrich.com/catalog/product/sial/p5629>

- \* CAS: 7778-53-2
- \*  $K_3PO_4$
- \* 212.266262 g per mole
- \* Density: 2.564 g/cm<sup>3</sup>
- \* Dry powder
- \* Causes serious eye damage
- \* Causes respiratory irritation

### *sodium borohydride*

Source:

<http://www.sigmaaldrich.com/catalog/product/aldrich/480886>

- \* CAS: 12125-02-9
- \*  $NH_4Cl$
- \* 53.49146 g per mole
- \* Melting Point: 350° C
- \* Density: 1.5 g/cm<sup>3</sup>
- \* Solubility: Soluble in methanol
- \* Colorless to white powder
- \* Odorless
- \* Harmful if swallowed
- \* Causes serious eye irritation
- \* Causes respiratory system irritation
- \* Causes nausea

### *sodium hydroxide*

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/14798>

- \* CAS: 1310-73-2
- \* NaOH
- \* 39.997109 g per mole
- \* Melting Point: 323° C
- \* Density: 2.13 g/cm<sup>3</sup> @ 25° C
- \* Solubility: Soluble in water
- \* White solid pellets
- \* Harmful if swallowed
- \* Severe skin/eye burns
- \* May cause respiratory irritation
- \* Causes pneumonitis

### *sodium nitrite*

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/23668193>

- \* CAS: 7632-00-0
- \*  $\text{NaNO}_2$
- \* 68.995269 g per mole
- \* Melting Point: 271° C
- \* Density: 2.17 g/cm<sup>3</sup>
- \* Solubility: Soluble in water; moderately soluble in methanol
- \* White to yellow crystalline powder
- \* May intensify fire
- \* Oxidizer
- \* Toxic if swallowed
- \* Causes headaches, vertigo, and nausea
- \* Causes palpitations
- \* Causes diarrhea
- \* Possible carcinogen—stomach cancer

### *sodium tert-butoxide*

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/23676156>

<http://www.chemnet.com/cas/en/865-48-5/Sodium-t-butoxide.html>

<http://www.sigmaaldrich.com/catalog/product/aldrich/703788>

- \* CAS: 865-48-5
- \*  $\text{C}_4\text{H}_9\text{NaO}$
- \* 96.103429 g per mole
- \* Melting Point: 84.6° C
- \* Melting and boiling point data on various sites confused
- \* Density: 1.104 g/cm<sup>3</sup>
- \* Flammable, self heating
- \* Causes severe skin and eye burns
- \* Protect from sunlight

### *tetrabutylammonium bromide*

Sources:

<https://pubchem.ncbi.nlm.nih.gov/compound/74236>

<http://www.sigmaaldrich.com/catalog/product/sial/193119>

- \* CAS: 1643-19-2
- \*  $\text{C}_{16}\text{H}_{36}\text{BrN}$
- \* 322.36774 g per mole
- \* Melting Point: 102 – 106° C
- \* Clear colorless to yellow dry powder
- \* Causes skin and eye irritation
- \* Causes respiratory irritation

### *tin*

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/5352426>

- \* CAS: 7440-31-5
- \* Sn
- \* 118.71 g per mole
- \* Melting Point: 231.9° C
- \* Density: 7.265 g/cm<sup>3</sup> @ 15° C
- \* Solubility: Soluble in HCl and H<sub>2</sub>SO<sub>4</sub>; slightly soluble in HNO<sub>3</sub>
- \* Silver, white crystalline powder
- \* Causes irritation to eyes
- \* Causes irritation to respiratory system
- \* Combustible
- \* Spontaneous combustion with Br<sub>2</sub>



### *toluene*

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/1140>

- \* CAS: 108-88-3
- \*  $C_7H_8$
- \* 92.13842 g per mole
- \* Boiling Point:  $110.6^\circ C$
- \* Density:  $0.8636 \text{ g/cm}^3 @ 20^\circ C$
- \* Solubility: Miscible in alcohol, ether, acetone, acetic acid
- \* Colorless liquid, sweet odor
- \* Highly flammable liquid and vapor
- \* May be fatal if swallowed and enters airways
- \* Causes skin and eye irritation
- \* Harmful if inhaled
- \* May damage fertility or the unborn child
- \* Causes damage to organs

### *zinc*

Source:

<https://pubchem.ncbi.nlm.nih.gov/compound/23994>

- \* CAS: 7440-66-6
- \* Zn
- \* 65.38 g per mole
- \* Melting Point:  $419.5^\circ C$
- \* Density:  $7.14 \text{ g/cm}^3 @ 15^\circ C$
- \* Solubility: Soluble in acids and alkenes
- \* Grayish colored powder
- \* Pyrophoric liquid and solid
- \* Releases flammable gases in contact with water
- \* Causes dry skin
- \* Causes nausea



## Annex 2: Experimental Results Summary

Product	Primary Reagent	Mass Reagent (g)	Moles Reagent	Mass Product (g)	Moles Product	% Yield
2-nitrofluorene	fluorene	3.0	0.018	2.338	0.011	61.5
2-nitrofluorenone	2-nitrofluorene	0.158	0.000748	0.082	0.000364	48.7
2-aminofluorene	2-nitrofluorene	1.265	0.00599	0.685	0.000378	63.5
bromo-imine	2-aminofluorene	0.273	0.00151	0.218	0.000626	41.6
acetamide	2-aminofluorene	0.136	0.00750	0.175	0.000784	104
fluoro-benzylamide	2-aminofluorene	0.075	0.000414	0.015	0.000049	11.9
2-iodofluorene	2-aminofluorene	0.270	0.00149	0.195	0.000668	44.8
bromo-benzyl amine	bromo-imine	0.041	0.000621	0.041	0.000117	18.9
suzuki product	2-iodofluorene	0.046	0.000507	0.046	0.000172	34.0



# Annex 3: General Laboratory Notes