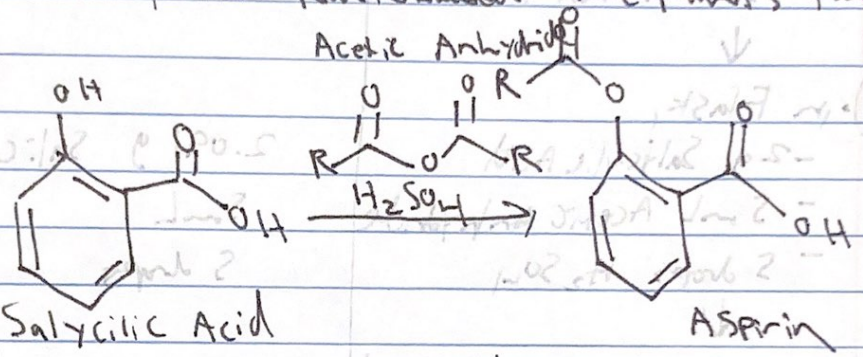
Synthesis of Aspirin and Indigo

Purpose: Creating Synthetic organic compounds like Aspirin and Indigo is beneficial so we don't have to rely on ~~[illegible]~~ the plants themselves.

Aspirin:



Salycilic Acid → Aspirin

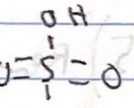
Materials:

- Large beaker
- Hot Plate
- 50mL Erlenmeyer Flask
- Graduated cylinder
- Ring Stand
- Ice
- Suction Filtration apparatus
- TLC Plates, aspirin Standard Sol'n, Ethanol

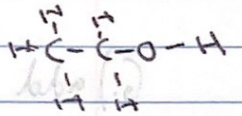
Chemicals

- Salicylic Acid 2g
- Acetic Anhydride 5 mL
- Sulfuric Acid 5 drops
- Ethanol 10 mL

Sulfuric Acid:



Ethanol:



Hazards:

Flammable
- Salicylic Acid
- Acetic Anhydride

Corrosive
- Ethanol

- Salicylic Acid – Causes burns –
- Acetic Anhydride – Causes severe burns – rinse 15 min (Cold water)
- Sulfuric Acid – Causes severe burns – rinse 15 min

TS

Aspirin

Procedure:

1) Fill beaker 3/4 water
   - boil on hot plate
   ↓
2) in Flask,
   - 2 g Salicylic Acid — 2.091 g Salic
   - 5 mL Acetic Anhydride — 5 mL
   - 5 drops H_2SO_4 — 5 drops
   ↓
3) Clamp Flask to ring Stand
   - lower into boiling H_2O
   ↓
4) Reflux 10 min
   ↓
5) Remove From heat
   - cool to room temp
   ↓
6) add 1 mL, sit 1 min
   ↓
7) Fill beaker with ice water
   - lower flask into ice
   ↓
8) Add 40 mL di water to flask - 10 min
   ↓
9) Suction Filter

Yellow tint to liquid

Aspirin

Procedure:

10) Place solid in flask
- add min. Ethanol until dissolves
- add dI water until Precipitate

drop wise

↓

11) Place in ice bath 20 min — needed more time

↓

12) weigh filter & funnel — Funnel weight: ~~16.56 g~~ 16.52 g

↓

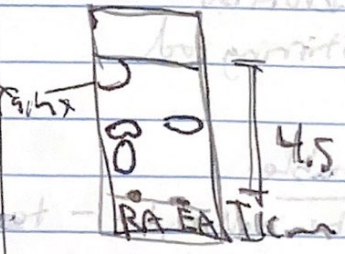
13) Suction filter — weight #2: 17.260

↓

14) Press capillary into product and find melting point — T_1: 110°C, T_2: 120°C

↓

15) TLC

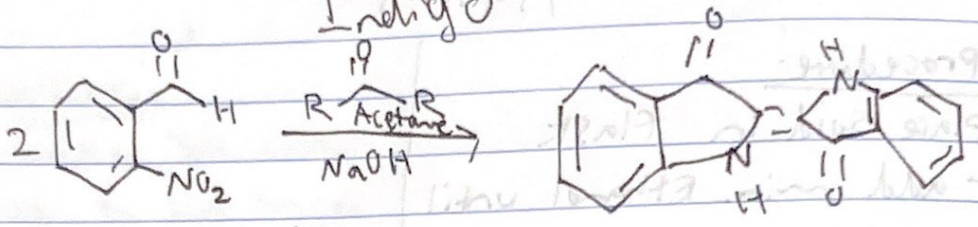


EA: 2.8 cm
RA: 2.8
2.3
4.3

16) IR Spectra — Salicylic Acid:

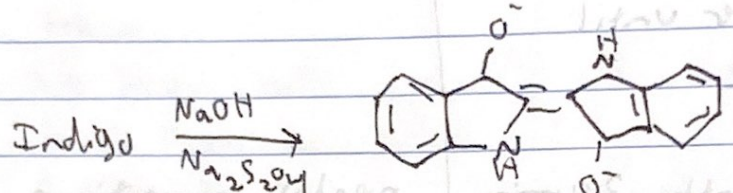
Aspirin:

Indigo

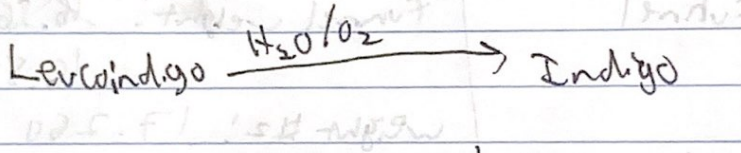


2 Nitrobenzaldehyde

Indigo



Leucoindigo



Materials:	Chemicals:	
• Stir bar	• 2-Nitrobenzaldehyde	0.5 g
• 50 mL Beaker	• Acetone	5 mL
• Suction Filtration apparatus	• 1M NaOH	2.5 mL
• #10 microfiber	• Sodium Dithionite	0.15 g
• Glass Stirring rod	• 1M NaOH	10 mL

Flammable:

- 2 Nitrobenzaldehyde – toxic, irritant
- Acetone – irritant – wash soap/water 5 min
- Sodium Dithionite
- NaOH – Corrosive – wash cold water
- Sodium Hydrosulfite – toxic – irritant – can spontaneously ignite – DO NOT LEAVE OPEN TO AIR

Indigo

Procedure

1) Combine:
- Stir bar
- 0.5 g 2 Nitrobenz — 0.511 g 2N
- 5 mL Acetone
- 2.5 mL 1M NaOH

dropwise

- Stir 10 min
↓

2) weigh Buchner Funnel w/ filter — weigh 1: 16.56 g
↓

3) Suction filter
- pre wet filter
- while aspirator is running:
  - pour indigo soln onto center of filter
- weigh — weigh 2: 17.18 g
↓

4) Combine:
- 0.1 g Product — 0.125 g
- 0.15 g Sodium Dithionite — 0.19 g
- grind with glass rod — turned blue (7:38)
- 10 mL 1M NaOH
↓

5) Heat until soln runs yellow — did not turn yellow
↓

6) Add multifiber fabric 10 to beaker — added paper towel

Indigo

7) **Procedure:**

Rinse fabric with water
- dry

Fabric:

- Rayon
- Cotton
- Nylon
- Dacron
- Orlon
- Wool

Paper towel did not dye well

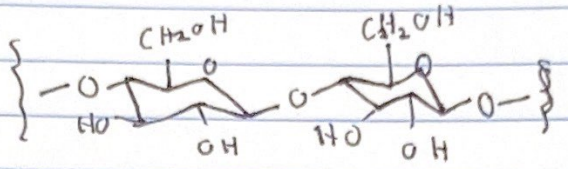
- M_w Salic: 138.12 g/mol
- M_w Aspirin: 180.158 g/mol

theoretical: $\frac{m_{sal}}{M_{w\,sal}} = \frac{2.091\ g}{138.12\ g/mol} = 0.0151\ mol$

Actual: $\frac{m_A}{M_{w\,A}} = \frac{17.26 - 16.52}{180.158} = 0.004108\ mol$

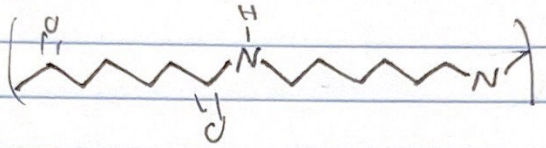
% yield = $\frac{\text{Actual}}{\text{theory}} \cdot 100 =$ 27% yield

Rayon:

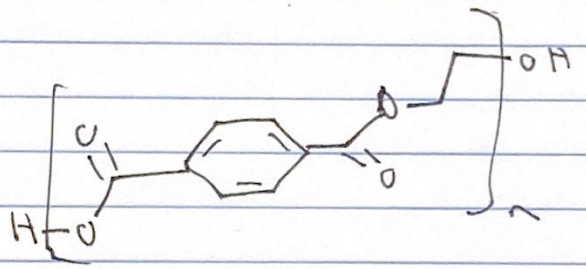


Cotton:

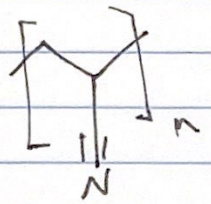
Nylon:



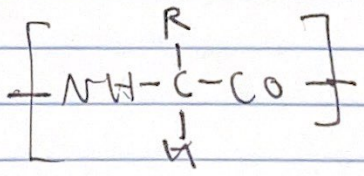
Dacron:



Orlon:



Wool:



Josh Whitehead
U1069343
Chem 2315-001

Lab 3 "Synthesis of Aspirin and Indigo" Conclusion

Aspirin:

The percent yield is given by the equation $\frac{Experimental\ yield}{Theoretical\ yield} * 100\%$. We started with 2.091g salicylic acid and produced 0.740g aspirin. Using $180.158\frac{g}{mol}$ as the molar mass for aspirin and $138.12\frac{g}{mol}$ as the molar mass for salicylic acid, I converted from grams to moles and found the percent yield to be about 27.13%. One reason for obtaining less than 100% yield is possibly due to length of time the solution was in the ice bath. The procedure instructs to leave it for 20 min but we did not find precipitate until after about 30 min. Based on this observation it is possible that the molecule needed more time to precipitate. A reason for obtaining over 100% yield is possibly due to calculation error, or if there are many impurities in the solid when it is weighed.

We observed the melting point of the synthesized aspirin to be (110-120)°C. This is a very close range so it is possible that the aspirin was very pure. However, when my lab partner was using the "Mel-temp", she looked away after the sample started melting and when she looked back it had already melted completely. This error suggests it is also possible that we were increasing the temperature too fast to take accurate readings. A potential impurity would be salicylic acid that did not react completely.

According to our observations of the TLC plate, our aspirin was very pure and had an Rf value of 0.62. We did see multiple spots with the reference aspirin with Rf values of 0.51, 0.62, and 0.96. The spot closest to the top of the plate was very faint so it is possible that someone touched that plate on accident. The reference aspirin was also in a bottle that was shared between many students and is likely contaminated.

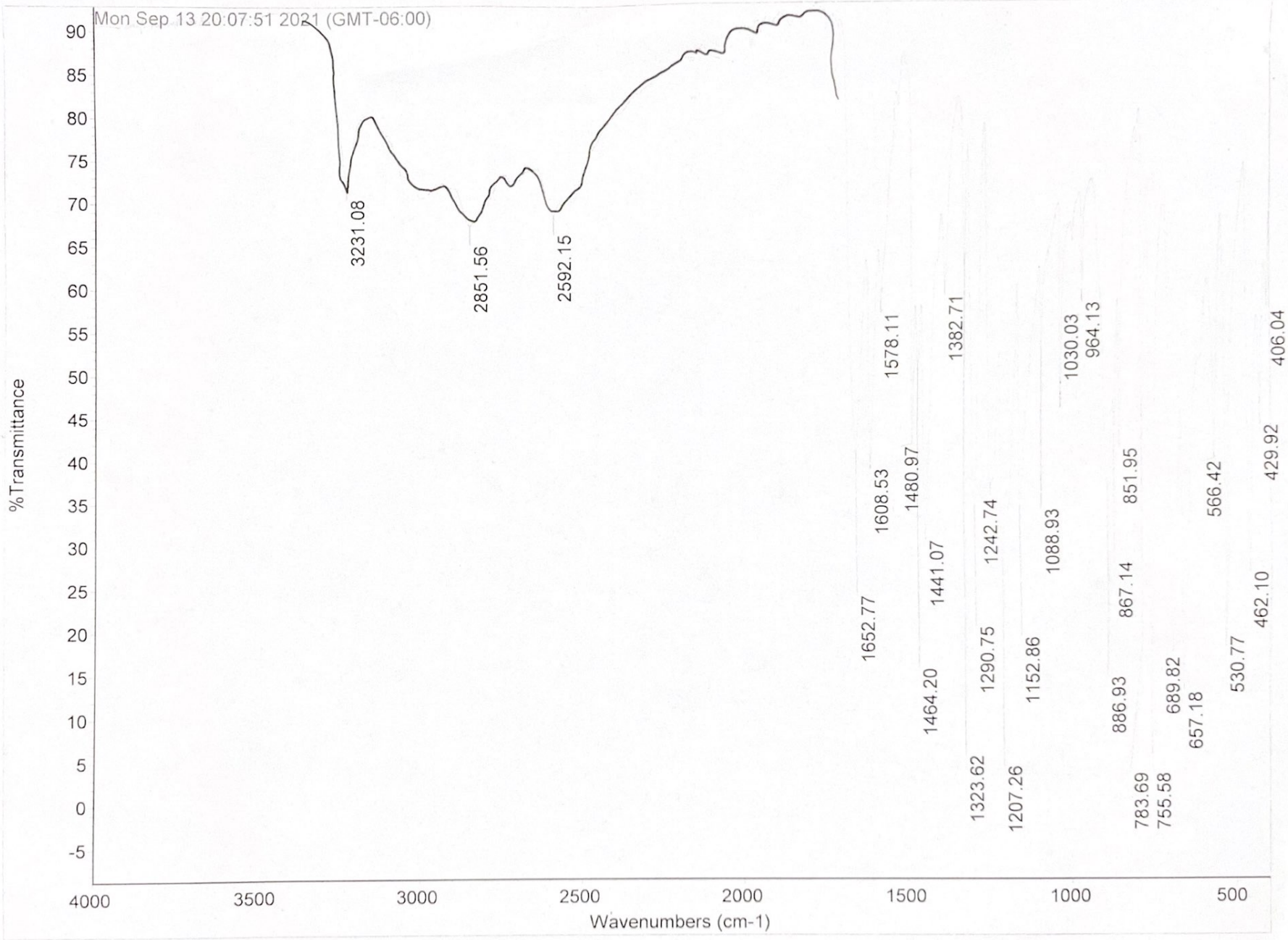
The IR spectra for salicylic acid has three distinct peaks with wavenumbers of 3231.08, 2851.56, and 2592.15cm^{-1}. The spectra for aspirin had two peaks around 2592 and 2851cm^{-1}. These similarities could be due to the similar chemical structure of salicylic acid and aspirin. Both molecules have a phenol group attached to a carboxylic acid. The main difference is that salicylic acid has a hydroxyl group where aspirin has a ketone. The hydroxyl group could be the cause of the third peak of 3231.08cm^{-1}. Based on the IR spectra results, the synthesized aspirin appears to be relatively pure because there are no substantial peaks with unexpected wavenumbers.

Indigo:

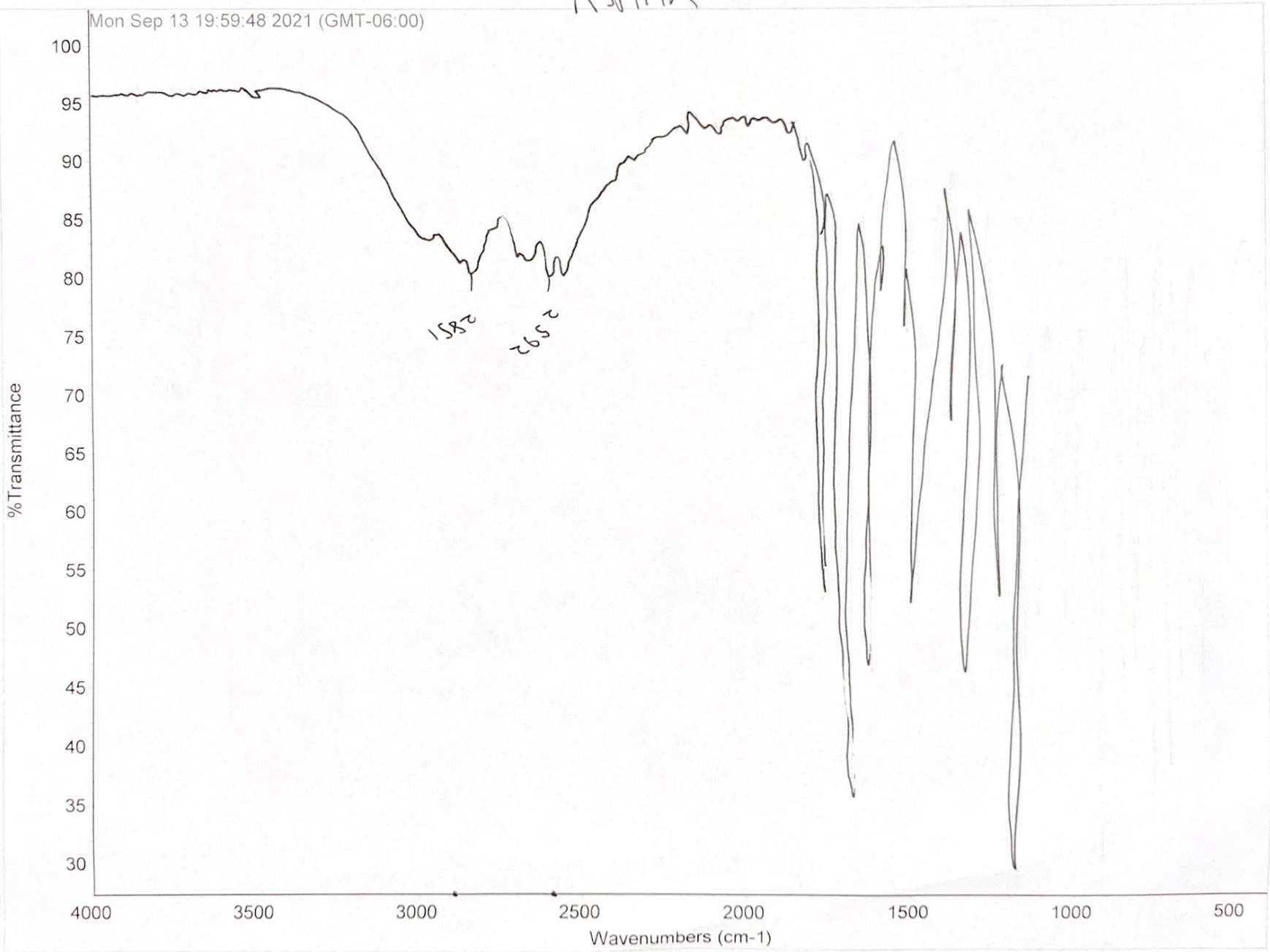
Percent yield of indigo was calculated using the same method as with aspirin. We started with 0.511g 2-nitrobenzaldehyde and obtained 0.558g indigo. Using $151.12\frac{g}{mol}$ as the molar mass of 2-nitrobenzaldehyde and $262.27\frac{g}{mol}$ for indigo, our percent yield was 62.9%. Possible sources for obtaining less than 100% yield are adding too little acetone so it is the limiting reagent, or not allowing the 2-nitrobenzaldehyde to completely react with the acetone. A reason for obtaining over 100% yield could be due to some of the starting material not filtering out of the product.

We did not have enough of "multifiber fabric 10" so we tried to dye a paper towel instead. The towel did not hold the dye well because it is made of cellulose fibers which are polar and is capable of many hydrogen bonds, dipole-dipole interactions and London dispersion forces but indigo is only partially polar with much less opportunity for hydrogen bonding.

Salicylic Acid



Aspirin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Wavenumbers (cm-1)