Name: _____

TA: _____

# Chemistry 238
# Grading Rubrics

## An Aggie does not lie, cheat or steal or tolerate those who do!

## Experiment 6: Iodination of Salicylamide

**Section I: Pre-lab Write-up** **Possible Points for Section I: 7 pt** Pts Awarded

| | | | |
|---|---|---|---|
| Objectives | Clear Statement of Objectives | **1** | |
| Methods to Objectives | A Statement of overall approach to achieving objectives. NOT a detailed procedure or just a list of techniques | **1** | |
| Reagents and Solvents | A list of chemicals with physical & chemical properties important to this experiment. | **1** | |
| Safety | List of most important safety issues | **2** | |
| Procedure | Steps to follow in procedure are outlined | **2** | |

**Section II: In-lab Observations** **Possible Points for Section II: 10 pt**

| | | | |
|---|---|---|---|
| Details recorded in duplicate lab notebook pages during lab time | Steps followed in procedure are given in notebook pages. | **3** | |
| | All relevant data is included and clearly identified (weights, melting points, boiling points, Rf, etc.) All graphs, printouts, and TLC plates are clearly labeled. | **5** | |
| | All units of measurement are identified. | **2** | |

**Section III: Results and Discussion** **Possible Points for Section III: 28 pt**

| | | | |
|---|---|---|---|
| A summary of results and discussion of how results met objectives. | Two sentence summary of experiment. | **2** | |
| | Summary of results is thorough and presented clearly. | **6** | |
| | Data interpretation conveys understanding of applicable scientific concepts. | **6** | |
| | Unexpected events or problems are identified and explained in a scientifically sound manner. | **6** | |
| | Conclusions derived from results are presented and are explained in terms of scientific concepts related to the lab. Success of meeting objectives is discussed. | **8** | |
| | **Total:** | **45 pt** | Score: |

**Note:**
Points (up to 5 pts) can be deducted for Pre-lab Write-up not being complete at start of lab period. Points (up to 5 pts) may also be deducted for ignoring Safety issues or being unprepared to do the Experiment. Points (up to 5 pts) can be deducted for missing in-lab Notebook pages or instrument printouts.

| | | |
|---|---|---|
| **Order of Report Pages:** (ALL pages must be included for maximum points) | **1.** Printed copy of Rubric<br>**2.** TurnItIn Similarity Report<br>**3.** Initialed Pre-lab Page | **4.** Original copy of in-lab Notebook page<br>**5.** Printed Results and Discussion<br>**6.** Copies of any printouts from instruments or computers during the experiment |

# Guan - Iodination of Salicylamide.pdf

*by* Kathryn Hua Guan

**Submission date:** 19-Mar-2022 06:37PM (UTC-0500)
**Submission ID:** 1787939957
**File name:** Guan_-_Iodination_of_Salicylamide.pdf (74.34K)
**Word count:** 1563
**Character count:** 7843

Kathryn Guan
March 21, 2021
CHEM 238-501

# Iodination of Salicylamide Lab Results and Discussion

## Introduction

In this laboratory experiment, the primary objective was to conduct an electrophilic aromatic substitution reaction, then through purification, obtain an IR spectrum, in which the identity of the product can be analyzed. An electrophilic aromatic substitution reaction occurs when an electrophile reacts with an aromatic compound; the electrophile (E+) then replaces (substitutes) a proton on the aromatic ring. With the electrophilic aromatic substitution reactions, a carbocation intermediate is formed. This intermediate is stabilized by resonance because of the delocalization of the charge around the ring. In this step, the carbocation loses a proton, and the electrophile attacks at that site, and the final product is the substituted aromatic ring. The reaction is drawn on the attached IR sheet. This reaction proves true for the alkylation and also the acylation of aromatic rings, as well as the addition of sulfate, nitrogen, and halogens to the aromatic rings. However, if the aromatic ring already has a substituent group, the character of that substituent group will impact where the electrophilic substitution takes place, as well as determining if the ring is more or less reactive. The more reactive groups are known as activating groups. And the less reactive groups are deactivating groups. Activating groups include -OH, -OR, -NH2, and -R groups. For activating group substituents, they cause the electrophilic substitutions to occur at the ortho or/and para positions on the ring, relative to the location of the substituent group. Activating groups, through the donation of electron density through lone pairs or double bonds, make the ring more reactive, causing a more stable carbocation intermediate that is very prone to electrophilic substitution. Additionally, there are also deactivating groups. These include -CO2R, -COR, -SO3H, halogens, and -CHO groups. These deactivating substituent groups are meta-directors, meaning that the electrophilic substitution occurs at the meta position on the aromatic ring relative to the substituent group. They are also electron-withdrawing groups (except halogens). Halogens still cause ortho/para electrophilic substitutions while being deactivators because of their strong electronegativity.

In this laboratory experiment, salicylamide is used. It contains both activating groups and deactivating groups. The -OH group is activating (ortho/para position substitution) while the

CONH2 amide is deactivating (meta-directing). Therefore, with the iodination of salicylamide, there are two positions where the substitution could occur. The substituent pattern and identity of the product will be determined through the IR spectrum taken of the final product, as well as the melting point of the final product. These tests will allow for the distinguishment between a 1,2,3 tri-substituted product and a 1,2,4 tri-substituted product. The hypothesis for the product is a 1,2,4 tri-substituted product due to the steric hinderance of a 1,2,3 tri-substituted product.

**Procedures and Observations**

This laboratory experiment consisted of a reaction, a purification, an IR, and a melting point. First, an ice bath was prepared. Then, two test tubes were obtained. 20 mL ethanol was added to the first, larger test tube. 10.0 mL of 8.25% NaOCl (bleach) was added to the second test tube. Next, 1.020 g of salicylamide was obtained into a 125 mL Erlenmeyer flask. The salicylamide is a brownish color, crystalline solid. Then, the ethanol was transferred into the Erlenmeyer flask as well, and the salicylamide was dissolved in the ethanol. This took a while to dissolve, but once dissolved, the solution was clear. Then, 1.205 g of NaI (where the iodine substituent will come from) was added to the flask and stirred until homogenous. Some of the NaI was lost in transfer into the flask. The NaI was a solid, fine, white powder. The flask was then cooled in the ice bath to 0°C. The flask was then transferred back onto a heater-stirrer, and the 10.0 mL NaOCl was added slowly. After adding abut ½ mL NaOCl, the solution turned a dark red/orange, wine color. After adding another 2 mL, the solution kept darkening. At around 7mL of NaOCl added, the solution started to lighten, and it began to turn yellow at about 8 mL NaOCl added. Once all 10 mL NaOCl was added, the solution was a dark green, but when it was stirred for a while, it begam a pale yellow. When the solution turned pale yellow, the stirrer was turned off and the solution was left mostly undisturbed for about 10 minutes. The solution was disturbed a bit when taking the flask off the heater-stirrer. During this time of waiting, 10 mL sodium thiosulfate and 6 mL HCl were collected in separate test tubes. After 10 minutes, the sodium thiosulfate was added to the flask via Pasteur pipette and stirred until mixed. This solution was yellow. Then, the HCl was added and using pH strips, the acidity was measured. This solution initially was a pale yellow, but then solution then turned a pale white, and a white precipitate was formed. All 6 mL were used and the pH strips read that the solution was still basic, so more acid was added. The solution had to be acidic because the product would only be insoluble in acidic conditions.

Then, the product (the solid) was isolated via vacuum filtration and washed with cold water and ethanol. This crude product had a mass of 2.031 g. The product was white, thick, and clay-like. This product was then transferred to an Erlenmeyer flask, although some product was lost in the transfer. About 10 mL ethanol was added to the flask and heated until boiling. 25 additional mL of ethanol were added to dissolve the product. The solution was supposed to be clear (homogenous), but it was still cloudy for the sample, indicating impurity. The solution was then removed from the heater-stirrer and left undisturbed (disturbed when removing it from the heater-stirrer). After cooling to room temperature, the flask was placed in the ice bath to help the product recrystallize. This cold solution was then run through vacuum filtration where the crystal was collected and left to dry. In the transfer of crystal from the funnel to the filter, some product was lost. The mass of the solid was found to be 0.011 grams (some of the filter paper was torn off when the tape was removed, possibly decreasing the mass of the final product). A melting point range was then taken of the product, and it was found that the melting point range was 242.1 – 244.0°C. After heating it, the product turned a dark, solid orange.

**Results**

Table 1: In-lab masses

| | |
|---|---|
| Mass of salicylamide | 1.020 g |
| Mass of NaI | 1.205 g |
| Mass watch glass | 28.515 g |
| Mass watch glass + crude product | 30.546 g |
| Mass filter paper | 1.020 g |
| Mass filter paper and product | 1.031 g |
| Melting point range | 242.1-244.0 °C |

Table 2: IR peaks and functional groups

| | |
|---|---|
| N-H stretch (amine) | 3440.54 cm^-1 |
| O-H stretch (alcohol) | 3193.98 cm^-1 |
| C=O (carbonyl) | 1738.39 cm^-1 |
| C=C (aromatic ring) | 1618.55 and 1572.65 cm^-1 |
| 1,2,4 tri-substitution | 840.79 and 816.16 cm^-1 |

Percent yield:

Theoretical yield (1.020 g salicylamide = 0.007438 mol salicylamide = 0.007438 mol product = 1.964 g product

Actual yield: 0.011 grams

Percent yield: 0.011/1.964 = 0.56%

**Discussion**

For this laboratory experiment, the initial hypothesis was that the product would be 1,2,4 tri-substituted. This hypothesis was proven correct on the IR spectra with the peaks at 840.79 and 816.16 cm^-1. In addition to position, the IR spectrum also shows that the product is salicylamide because of the aromatic, carbonyl, alcohol, and amine peaks. Similarly, the melting point also indicates that the compound is 1,2,4 trisubstituted. The melting point range was noted to be 242.1 – 244.0°C in lab. According to literature, the melting point for 4-Iodo Salicylamide is between 208-225°C. Contrastingly, the melting point for 3-Iodo Salicylamide is less than 200°C, so the melting point achieved in lab is closer to the melting point for 4-Iodo salicylamide.

**Error**

While the results supported the initial hypothesis, the low percent yield indicated that there was much error that occurred in lab. This low percent yield may have been caused by product loss in transfer between glassware and filter paper. In addition, while the flasks were supposed to rest undisturbed, the flask was moved by mistake, causing any crystals that may have formed to then redissolve into solution, also lowering the percent yield. Another error was indicated by the high melting point. This indicates that there may have been impurities in the product, which may have resulted from heating or cooling too quickly or slowly. On the IR spectrum, there were also many peaks that would not be present on the IR of pure Salicylamide. This is likely due to cooling the crystals too quickly in ice bath – if the solution was forced to recrystallize too quickly, impurities could be trapped within the crystals.

**Conclusion**

While there were some errors, the overall objective to determine the structure of Iodo salicylamide was successful. The iodination of salicylamide through an electrophilic aromatic substitution did occur, and from lab data, it was concluded that a 1,2,4 tri-substitution product was formed.

# Guan - Iodination of Salicylamide.pdf

ORIGINALITY REPORT

| 19% | 0% | 1% | 19% |
|---|---|---|---|
| SIMILARITY INDEX | INTERNET SOURCES | PUBLICATIONS | STUDENT PAPERS |

PRIMARY SOURCES

| | | |
|---|---|---|
| 1 | Submitted to Texas A&M University, College Station<br>Student Paper | 16% |
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| Exclude quotes | Off | Exclude matches | Off |
| Exclude bibliography | Off | | |

# ELECTROPHILIC AROMATIC SUBSTITUTION—THE IODINATION OF SALICYLAMIDE

Name _____ Date _____

T.A. _____ Room Color _____

## LAB REPORT—PRE-LAB WRITE-UP

This is to be completed prior to *the beginning* of the laboratory session. TA must initial near beginning of lab.

**Objectives:**

The objective for this lab is to add iodine to salicylamide under electrophilic substitution and to determine the identity of the major product using IR

**Methods to Achieve Objectives:**

To achieve the objectives above we will use heating and cooling, vacuum filtration, crystallization, and IR will be utilized

| Reagents and Solvents | Properties | | | | |
|---|---|---|---|---|---|
| | MW g/mol | mp °C | bp °C | Density | Other |
| Ethanol | 46.07 | -114.0 | 78.37 | 789 kg/m³ | EtOH |
| NaOCl | 74.44 | 18°C | 101°C | 1.11 g/mL | |
| Salicylamide | 137.136 | 140°C | 181.5 | 1.33 g/mL | solid |
| NaI | 149.89 | 661°C | 1304 | 3.67 g/mL | solid |
| Sodium thiosulfate | 158.11 | 48.3 | ~~100~~ | 1.67 g/cm³ | |
| HCl | 36.458 | -174 | -85.1 | 1.2 g/mL | |
| | | | | | |
| | | | | | |
| | | | | | |

**Safety Issues:**

- wear nitrile gloves and goggles at
- handle chemicals with caution as many aromatic compounds are toxic
- salicylamide and NaT are irritants and sodium hypochlorite and HCl are corrosive and irritants. Handle with care
- the bleach solution can emit chlorine gas which is a respiratory and eye irritant. Avoid skin contact with chemicals, non M TM OTSpills

**List of Steps to Perform Experiment:**

1. prepare an ice water bath to cool reaction flask
2. label 2 test tubes EtOH and bleach soln. obtain 20mL of EtOH and 7.6 mL NaOCl
3. measure out ~ 1.0 g solid salicylamide and record mass
4. add solid to 125mL flask w/ stir bar and add EtOH
5. measure out 1.2 g solide NaI. Record mass and add to flask. stir
6. cool homogeneous mixture by placing it in ice bath. when it is 0°C, move flask from bath and place it on heat/stir
7. Quickly add 7mL NaOCl w/ pasteur pipet and stir
8. Record color changes that occur. When solution reaches a faint, yellow color will appear to indicate rxn is complete. Turn off stirr and let it sit undisturbed for 10 min
9. obtain 10mL sodium thiosulfate and label test tube. add 6 mL HCl. Add sodium thiosulfate to rxn after 10 min
10. Quench the rxn by slowly adding HCl and monitor the acidity
11. collect solid product w/ vacuum filtration. wash w/ water and ethanol
12. weigh crude product and place in flask w/ 10mL EtOH and heat/stir. Add EtOH to dissolve. Remove from hot to allow cool. Cool in ice bath
13. collect crystals w/ vacuum filtration
14. obtain mass. mp. IR

Teaching Assistant Initials MB

Electroph

POST-LAB

Attach your lab

The Results
(.doc or .rtf
attached to

| Exp. No. 6 | Experiment/Subject Iodination of salicylamide | | Date 2/28/22 |
|---|---|---|---|
| Name Kathryn Gnan | Lab Partner | Locker/ Desk No. | Course & Section No. |

mass: 1.020 g salicylamide light
↳ brownish color, crystalline
- some product lost in transfer to flask

1.205 g NaI → solid fine white powder
① add salicy. to EtOH → took a while to dissolve → clear solution
② add NaI → some product lost in transfer, quick dissolve
③ add NaOCl → dark red/brown/orange, darker @ 1mL, darker @ 2mL, light tan @ ~7mL
faint yellow color
½ mL
turn green → add NaOCl → very dark brown ~~stir~~ stir + turn yellow @ 8 mL
start to turn yellow @ 9 mL

④ add sodium thio → yellow
⑤ add HCl → white → stir → cloudy yellow ← add acid b/c only insoluble in acidic conditions
→ soln turned pale white
→ used 6mL + still basic, add more acid
· white solid in flask

mass watch glass: 28.515 g
⑥ transfer to watch glass → some loss of product
- product was white, thick, clay-like
mass 30.546 g - 28.515 = 2.031 g
product

some prod lost in transfer to 50 mL Erlenmeyer flask
~~add ~5-6mL EtOH/CHCl3~~
~~· added ~20mL EtOH to dissolve~~
· added ~35mL ethanol
· still kind of cloudy → impure
· mass filter paper: 1.020 g
· crystal stuck in bottom of flask → add EtOH to wash
- some crystal couldn't get out of flask
- some crystal lost in transfer to bag filter paper

| Signature | Date | Witness/TA | Date |
|---|---|---|---|

| Exp. No. | Experiment/Subject | Date | |
|---|---|---|---|
| Name | Lab Partner | Locker/ Desk No. | Course & Section No. |

·unknown: solid, bright orange color, clear solution

Distillation

·round bottom flask very dirty - washed w/ acetone

·start to boil at 27.3°C - but that is lower than acetone bump bc bump probe

1st fraction: went up to 62.2°C and fell to ~~51.6~~ 48.2°C

·not back up → keep rising ~62°C and drops 4 to ~50°C

~~top~~.

·smells sweet → distill 102.8 - 143.2°C

-fluctuating many cycles

-fell back down to 135.1°C

·distillate misty

·solution distill clear, dark orange left in flask

·remainer dried out crystallized

Tollens Test

·dark grey precipitate w/ silver nitrate + NaOH

-dissolve precipw/ ammonium hydroxide

·heat added - black precipitate form → aldehyde

solid

reference: butyraldehyde - black/grey precip form

Solubility

·not soluble, misty layer on top of clear water

Schiffs

unknown

·insoluble white puddles

·light pink (very faint)

negative

butyraldehyde

top layer dark purple (2 layers)

Iodoform

·w/ unknown: ~~no precipitate~~ murky white → turn yellow (pos)

·2-butanone: more yellow color

~~Δ~~ redid this

·clear this time, no color change

---

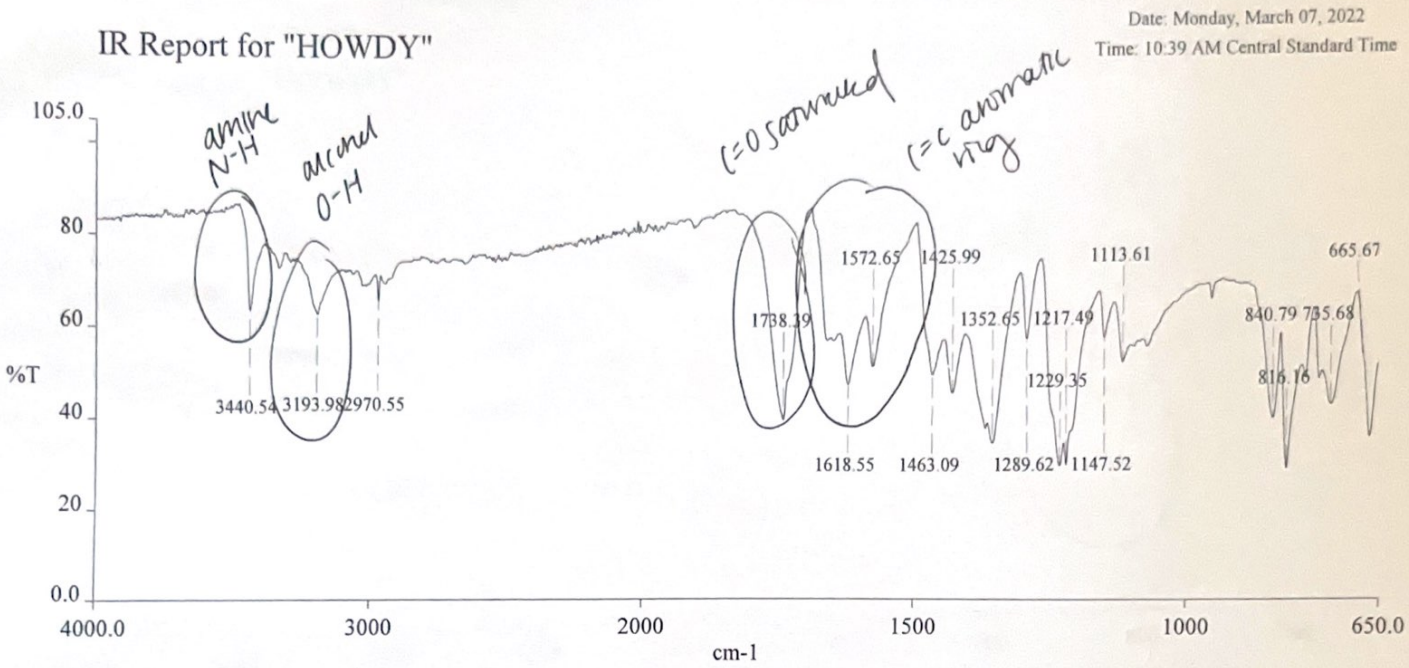
Mass product from last lab

·1.031g (some filter paper came off when the tape was taken off the filter paper so lower mass than acetone)

melting range: 242.1 - 244.8°C

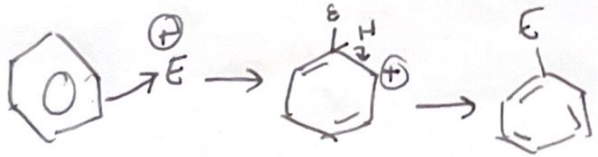
- may have heated too fast b/c it ~~was~~ turned dark orange

| Signature | Date | Witness/TA | Date |
|---|---|---|---|

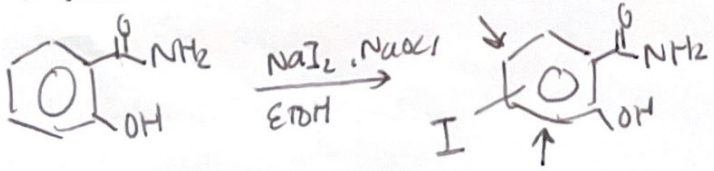



5

Electrophilic Aromatic Sub

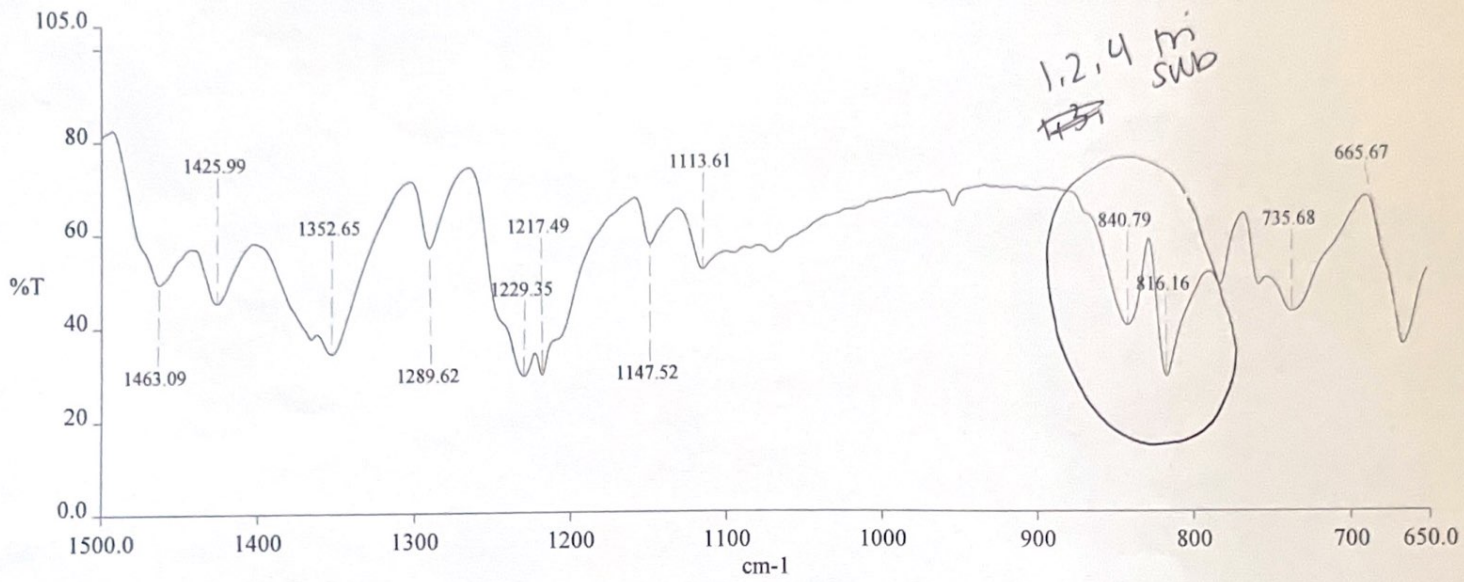


Iodination



IR Report for "HOWDY"

Date: Monday, March 07, 2022
Time: 10:40 AM Central Standard Time




1,2,4 tri sub

~~1451~~

1/ABS

~~.007416~~

262.944984

Kathryn Guan

March 21, 2021

CHEM 238-501

# Iodination of Salicylamide Lab Results and Discussion

## Introduction

In this laboratory experiment, the primary objective was to conduct an electrophilic aromatic substitution reaction, then through purification, obtain an IR spectrum, in which the identity of the product can be analyzed. An electrophilic aromatic substitution reaction occurs when an electrophile reacts with an aromatic compound; the electrophile (E+) then replaces (substitutes) a proton on the aromatic ring. With the electrophilic aromatic substitution reactions, a carbocation intermediate is formed. This intermediate is stabilized by resonance because of the delocalization of the charge around the ring. In this step, the carbocation loses a proton, and the electrophile attacks at that site, and the final product is the substituted aromatic ring. The reaction is drawn on the attached IR sheet. This reaction proves true for the alkylation and also the acylation of aromatic rings, as well as the addition of sulfate, nitrogen, and halogens to the aromatic rings. However, if the aromatic ring already has a substituent group, the character of that substituent group will impact where the electrophilic substitution takes place, as well as determining if the ring is more or less reactive. The more reactive groups are known as activating groups. And the less reactive groups are deactivating groups. Activating groups include -OH, -OR, -NH2, and -R groups. For activating group substituents, they cause the electrophilic substitutions to occur at the ortho or/and para positions on the ring, relative to the location of the substituent group. Activating groups, through the donation of electron density through lone pairs or double bonds, make the ring more reactive, causing a more stable carbocation intermediate that is very prone to electrophilic substitution. Additionally, there are also deactivating groups. These include -CO2R, -COR, -SO3H, halogens, and -CHO groups. These deactivating substituent groups are meta-directors, meaning that the electrophilic substitution occurs at the meta position on the aromatic ring relative to the substituent group. They are also electron-withdrawing groups (except halogens). Halogens still cause ortho/para electrophilic substitutions while being deactivators because of their strong electronegativity.

In this laboratory experiment, salicylamide is used. It contains both activating groups and deactivating groups. The -OH group is activating (ortho/para position substitution) while the

CONH2 amide is deactivating (meta-directing). Therefore, with the iodination of salicylamide, there are two positions where the substitution could occur. The substituent pattern and identity of the product will be determined through the IR spectrum taken of the final product, as well as the melting point of the final product. These tests will allow for the distinguishment between a 1,2,3 tri-substituted product and a 1,2,4 tri-substituted product. The hypothesis for the product is a 1,2,4 tri-substituted product due to the steric hinderance of a 1,2,3 tri-substituted product.

**Procedures and Observations**

This laboratory experiment consisted of a reaction, a purification, an IR, and a melting point. First, an ice bath was prepared. Then, two test tubes were obtained. 20 mL ethanol was added to the first, larger test tube. 10.0 mL of 8.25% NaOCl (bleach) was added to the second test tube. Next, 1.020 g of salicylamide was obtained into a 125 mL Erlenmeyer flask. The salicylamide is a brownish color, crystalline solid. Then, the ethanol was transferred into the Erlenmeyer flask as well, and the salicylamide was dissolved in the ethanol. This took a while to dissolve, but once dissolved, the solution was clear. Then, 1.205 g of NaI (where the iodine substituent will come from) was added to the flask and stirred until homogenous. Some of the NaI was lost in transfer into the flask. The NaI was a solid, fine, white powder. The flask was then cooled in the ice bath to 0°C. The flask was then transferred back onto a heater-stirrer, and the 10.0 mL NaOCl was added slowly. After adding abut ½ mL NaOCl, the solution turned a dark red/orange, wine color. After adding another 2 mL, the solution kept darkening. At around 7mL of NaOCl added, the solution started to lighten, and it began to turn yellow at about 8 mL NaOCl added. Once all 10 mL NaOCl was added, the solution was a dark green, but when it was stirred for a while, it begam a pale yellow. When the solution turned pale yellow, the stirrer was turned off and the solution was left mostly undisturbed for about 10 minutes. The solution was disturbed a bit when taking the flask off the heater-stirrer. During this time of waiting, 10 mL sodium thiosulfate and 6 mL HCl were collected in separate test tubes. After 10 minutes, the sodium thiosulfate was added to the flask via Pasteur pipette and stirred until mixed. This solution was yellow. Then, the HCl was added and using pH strips, the acidity was measured. This solution initially was a pale yellow, but then solution then turned a pale white, and a white precipitate was formed. All 6 mL were used and the pH strips read that the solution was still basic, so more acid was added. The solution had to be acidic because the product would only be insoluble in acidic conditions.

Then, the product (the solid) was isolated via vacuum filtration and washed with cold water and ethanol. This crude product had a mass of 2.031 g. The product was white, thick, and clay-like. This product was then transferred to an Erlenmeyer flask, although some product was lost in the transfer. About 10 mL ethanol was added to the flask and heated until boiling. 25 additional mL of ethanol were added to dissolve the product. The solution was supposed to be clear (homogenous), but it was still cloudy for the sample, indicating impurity. The solution was then removed from the heater-stirrer and left undisturbed (disturbed when removing it from the heater-stirrer). After cooling to room temperature, the flask was placed in the ice bath to help the product recrystallize. This cold solution was then run through vacuum filtration where the crystal was collected and left to dry. In the transfer of crystal from the funnel to the filter, some product was lost. The mass of the solid was found to be 0.011 grams (some of the filter paper was torn off when the tape was removed, possibly decreasing the mass of the final product). A melting point range was then taken of the product, and it was found that the melting point range was 242.1 – 244.0°C. After heating it, the product turned a dark, solid orange.

**Results**

Table 1: In-lab masses

| | |
|---|---|
| Mass of salicylamide | 1.020 g |
| Mass of NaI | 1.205 g |
| Mass watch glass | 28.515 g |
| Mass watch glass + crude product | 30.546 g |
| Mass filter paper | 1.020 g |
| Mass filter paper and product | 1.031 g |
| Melting point range | 242.1-244.0 °C |

Table 2: IR peaks and functional groups

| | |
|---|---|
| N-H stretch (amine) | 3440.54 cm^-1 |
| O-H stretch (alcohol) | 3193.98 cm^-1 |
| C=O (carbonyl) | 1738.39 cm^-1 |
| C=C (aromatic ring) | 1618.55 and 1572.65 cm^-1 |
| 1,2,4 tri-substitution | 840.79 and 816.16 cm^-1 |

Percent yield:

Theoretical yield (1.020 g salicylamide = 0.007438 mol salicylamide = 0.007438 mol product = 1.964 g product

Actual yield: 0.011 grams

Percent yield: 0.011/1.964 = 0.56%

**Discussion**

For this laboratory experiment, the initial hypothesis was that the product would be 1,2,4 tri-substituted. This hypothesis was proven correct on the IR spectra with the peaks at 840.79 and 816.16 cm^-1. In addition to position, the IR spectrum also shows that the product is salicylamide because of the aromatic, carbonyl, alcohol, and amine peaks. Similarly, the melting point also indicates that the compound is 1,2,4 trisubstituted. The melting point range was noted to be 242.1 – 244.0°C in lab. According to literature, the melting point for 4-Iodo Salicylamide is between 208-225°C. Contrastingly, the melting point for 3-Iodo Salicylamide is less than 200°C, so the melting point achieved in lab is closer to the melting point for 4-Iodo salicylamide.

**Error**

While the results supported the initial hypothesis, the low percent yield indicated that there was much error that occurred in lab. This low percent yield may have been caused by product loss in transfer between glassware and filter paper. In addition, while the flasks were supposed to rest undisturbed, the flask was moved by mistake, causing any crystals that may have formed to then redissolve into solution, also lowering the percent yield. Another error was indicated by the high melting point. This indicates that there may have been impurities in the product, which may have resulted from heating or cooling too quickly or slowly. On the IR spectrum, there were also many peaks that would not be present on the IR of pure Salicylamide. This is likely due to cooling the crystals too quickly in ice bath – if the solution was forced to recrystallize too quickly, impurities could be trapped within the crystals.

**Conclusion**

While there were some errors, the overall objective to determine the structure of Iodo salicylamide was successful. The iodination of salicylamide through an electrophilic aromatic substitution did occur, and from lab data, it was concluded that a 1,2,4 tri-substitution product was formed.