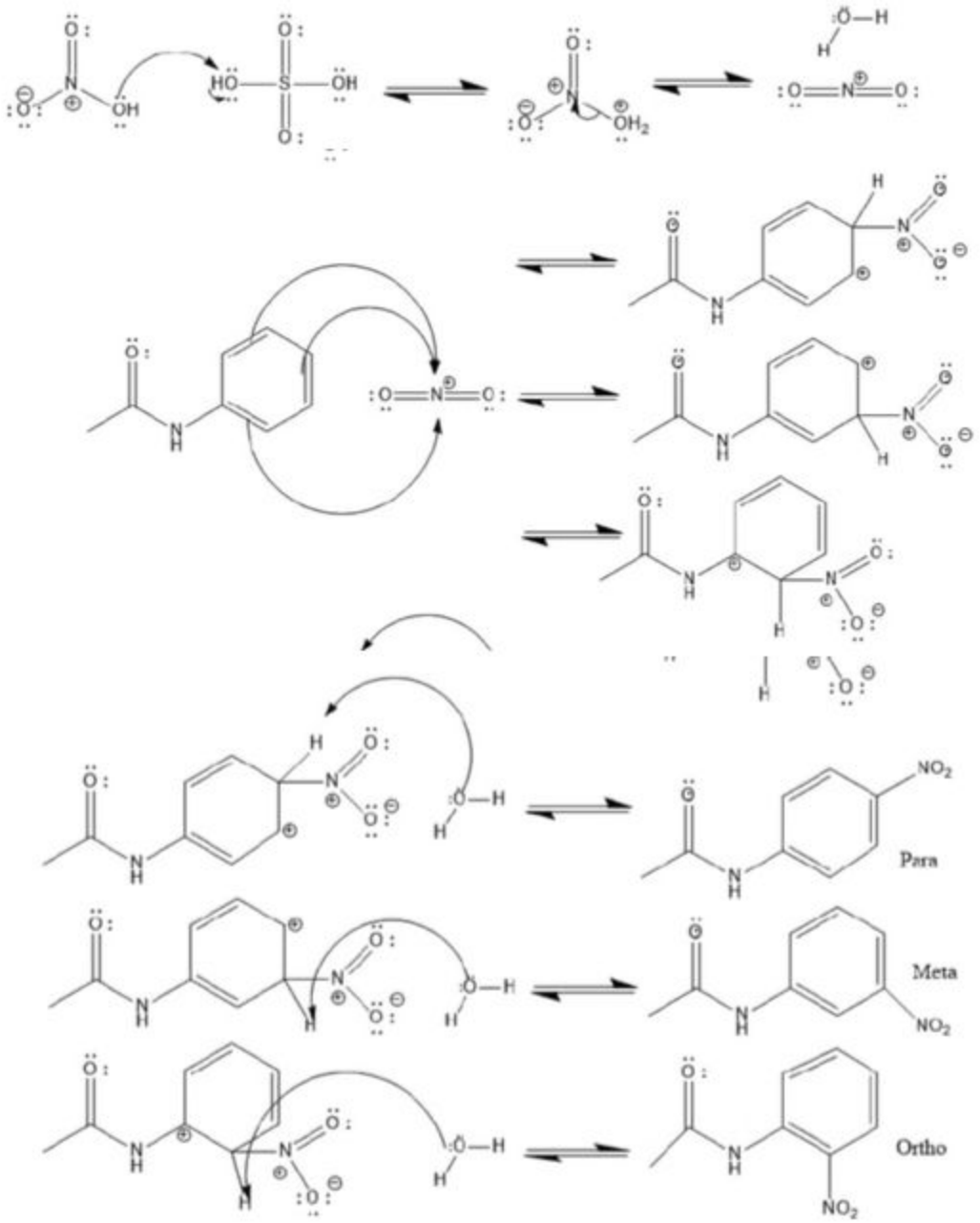
Introduction

The mechanism of isomers;



Procedure

Refer to “CHM 1321 Organic Chemistry Laboratory Manual” page 7.

Table 1. Reagents

Compound	**Amount (g or mL)**	**Molecular Weight (g/mol)**	**Density (g/mL)**	**Moles**
Acetanilide	1	137.17	1.22	0.0073
Nitric Acid	0.9	63.01	1.51	0.014
Sulfuric Acid	1.2	98.08	1.84	0.012
Ethanol	5	46.07	0.7892	0.085

Observations

- Sulphuric acid was a clear liquid
- Acetanilide had light beige flakes
- Adding the sulphuric acid and acetanilide turned it into a deep brownish liquid, while acetanilide still remained on the bottom due to the fact that the flakes never fully dissolved
- Once solution was cooled with ice and cold water it turned a foamy yellow urine like colour
- After filtration, the solid obtained was a yellow mustard powder

TLC Plates

Figure 1

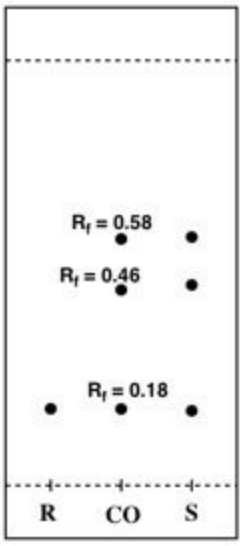


Figure 3

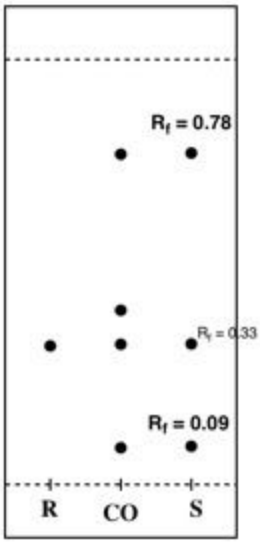


Figure 2

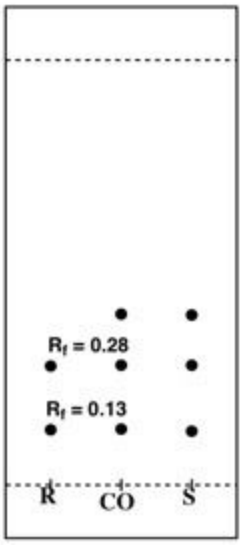


Figure 4

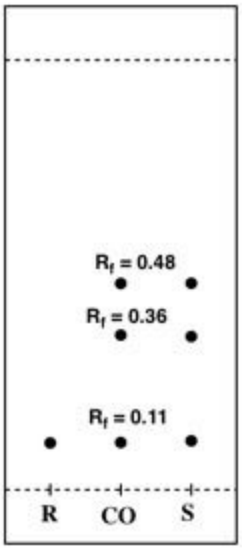


Figure 1; Meta and crude product in EtOAc:Hexanes 5:5

Figure 2: 2,4 dinitro and crude product in EtOAc:Hexanes 5:5

Figure 3: Ortho and crude product in EtOAc:Hexanes 5:5

Figure 4: Para and crude product in EtOAc:Hexanes 5:5

*For figures 1-5 it was crude as the sample spot and then whatever was listed in the caption (ex. reference spot for figure 4 is para)

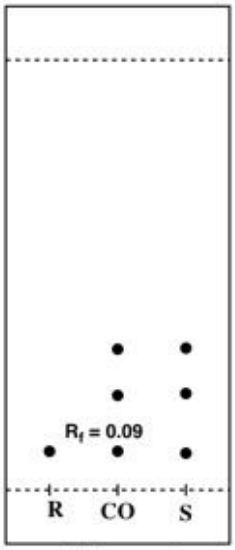


Figure 5

Figure 6

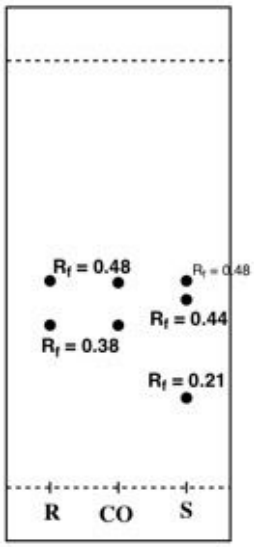


Figure 7

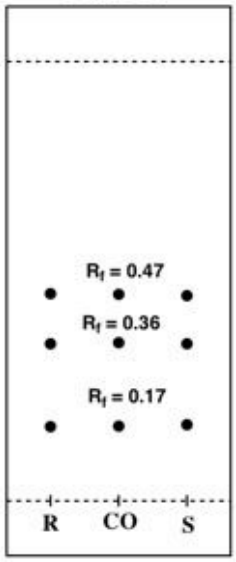


Figure 5: Acetanilide (in acetone) and solution in EtOAc:Hexanes 5:5
R: acetanilide in acetone (suction liquid ethanol)
CO: acetanilide and solution
S: Solution purified with dichloro

Figure 6: Crude product and purified product in EtOAc:Hexanes 5:5
R: Crude
Co: purified and crude
S: Purified product

Figure 7: Crystal product and mother liquid in EtOAc:Hexanes 5:5.
R: purified product
CO: purified and Mother liquid
S: Mother liquid: purified product with dichloromethane

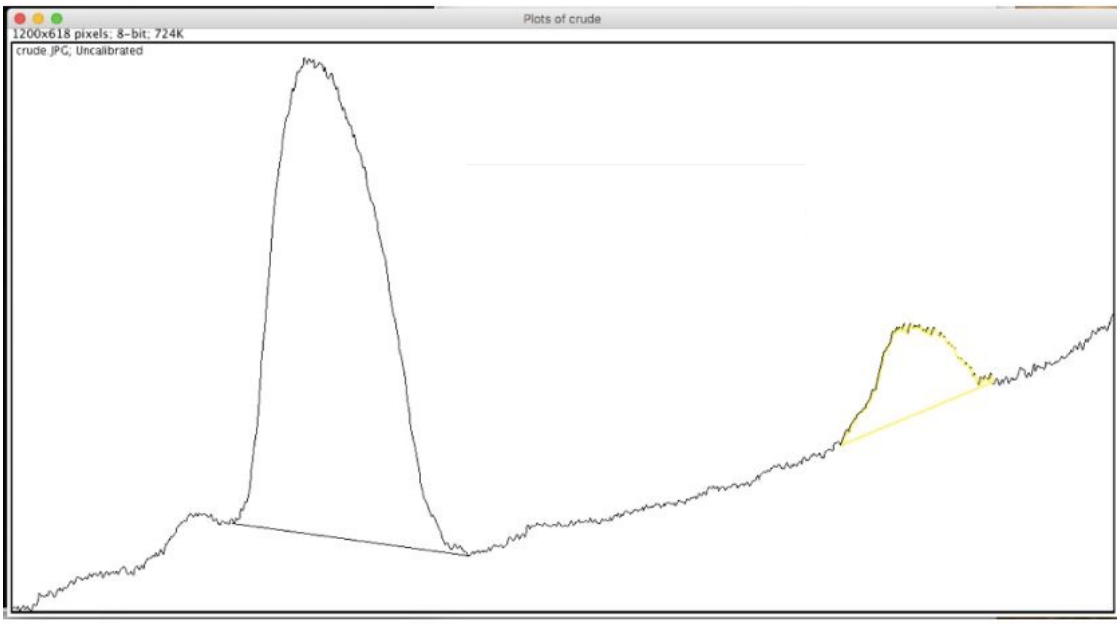
- There were two faint spots above the first 2 dots in the R and CO lane. This final TLC plate demonstrates that we successfully removed some of the impurities.

Table 2. Results

Compound	**Amount (g)**	**Molecular Weight (g/mol)**	**Density (g/mol)**	**Moles**
Nitroacetanilide	0.96	180.16	1.34	0.005

Calculations
% yield = (mass collected/mass started with) x 100
= (0.96g/1g) x 100% = 96%

Image J



Absorbance of para = 6653.047
Absorbance of dinitro = 8996.243
% Absorbance of para = (6653.047/6653.047 + 8996.243)x100% =42.51%
% Absorbance of 2,4 dinitro = (8996.243/8996.243 + 6653.047)x100% = 57.49%
Y= %absorbance 2,4-dinitro = 57.49%
%mole dinitro = _____%
%mole para = _____%
We were unable to find the mole values since in the end we ended up getting meta not dinitro, this gave us the wrong curve which made it not possible to get the mole percentage.

Discussion

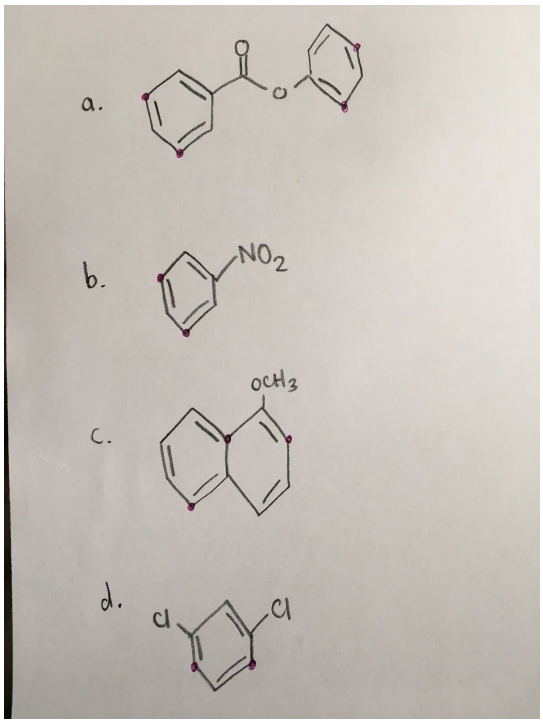
The overall purpose of this lab was to show a nitration of acetanilide using two different acids; sulfuric and nitric acid. Nitric acid acts as a base in this experiment as it is the weaker acid compared to sulfuric acid. Since the addition of the nitronium ion occured on the benzene ring, we could then determine which isomer (para, ortho or meta) was most present. Following this, TLC plates were used to distinguish between these isomers. After analyzing TLC results, we can conclude the para isomer was the most favoured due to the fact that the sample and reference lanes show the most cohesion between the three different isomers. Para is the most preferred of the three isomers due to the least amount of steric hindrance as the two functional groups are on opposite sides. After testing the three isomers, the crude product was placed in boiling ethanol to obtain the pure product of the para isomer. When ethanol is heated, both other isomers will dissolve and this happens because ethanol is a polar substance. After recrystallization, the para isomer will crystallize and the other two (meta & ortho) will be left in the mother liquid.

Sources of Error

- The most common error to of occured was most likely due to human error when measuring the exact amount of chemicals used. With the TLC contamination could have occurred and caused some TLC plates to have dots that shouldn't be present or in places they shouldn’t have been.
- Another source of error could have been done by the suction filtration, and this entails not giving enough time for the recrystallization to dry. Also, it was possible that when we added the ice that the reaction was not cold enough which never caused the reaction to fully complete it.

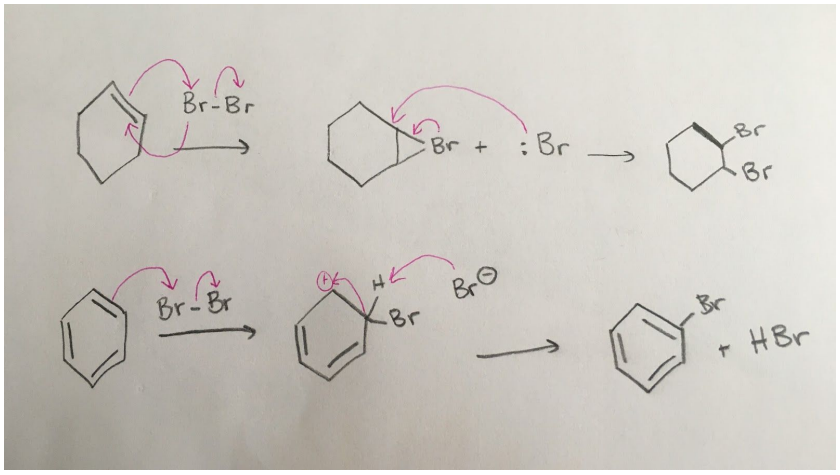
Questions

1. The addition of the second nitro group is slower than the first due to the difference in energy. This is caused by the first addition of the nitro group. The molecule is moved to a lower energy level due to the addition, and this makes it less reactive. Finally this lengthens it because it needs to add onto a less reactive molecule.
2.



3. a) By observing this reaction, we can see that it is an anti-electrophilic bromine addition.

b) By observing this reaction, we can see that this is an electrophilic aromatic substitution of bromine.



4. **a)** Moles of nitric acid = 0.009 L x 16 mol/L = 0.14 mol

Moles of benzene = 10 g / 78 g/mol = 0.13

Moles of sulfuric acid = 0.009 L x 18 mol/L = 0.16 mol

Therefore benzene is the limiting reagent.

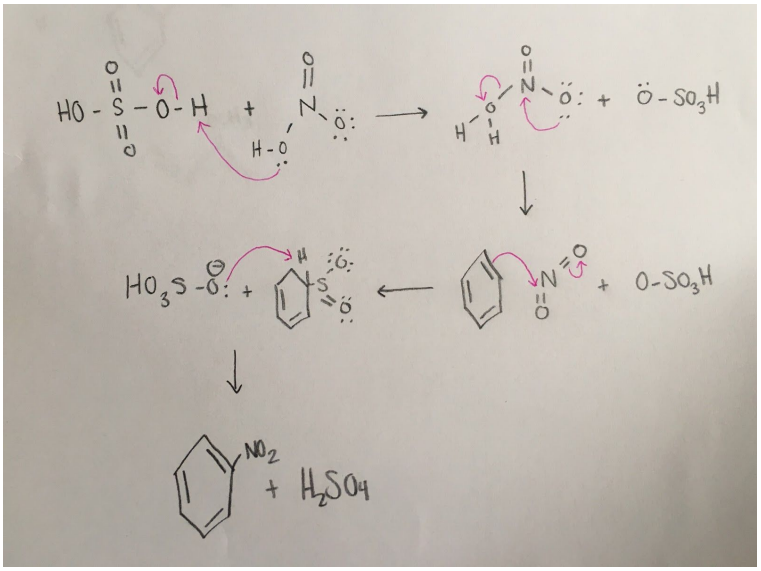
b) Theoretical yield = 0.13 mol x 123 g/mol = 16g

% yield = (actual yield) / (theoretical yield) x 100%

= (8.1g) / (16 g) x 100% = 50.63%

c) Since benzene is the limiting reagent, nitronium ions that are leftover will react with nitrobenzene and will then attack the meta position. This will result in m-dinitrobenzene.

d) The role of H2SO4 is both a catalyst and solvent which then reacts with nitric acid. The result of this reaction entails the creation of a strong electrophile known as nitrogen dioxide from the nitronium ion.



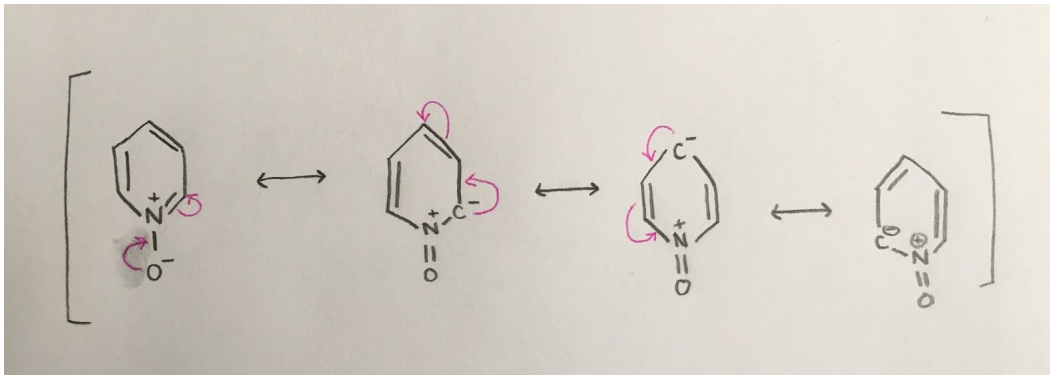
e) Molar Mass = 168.108 g/mol

Theoretical Mass = 10 mol x 168.108 = 1681.08 g

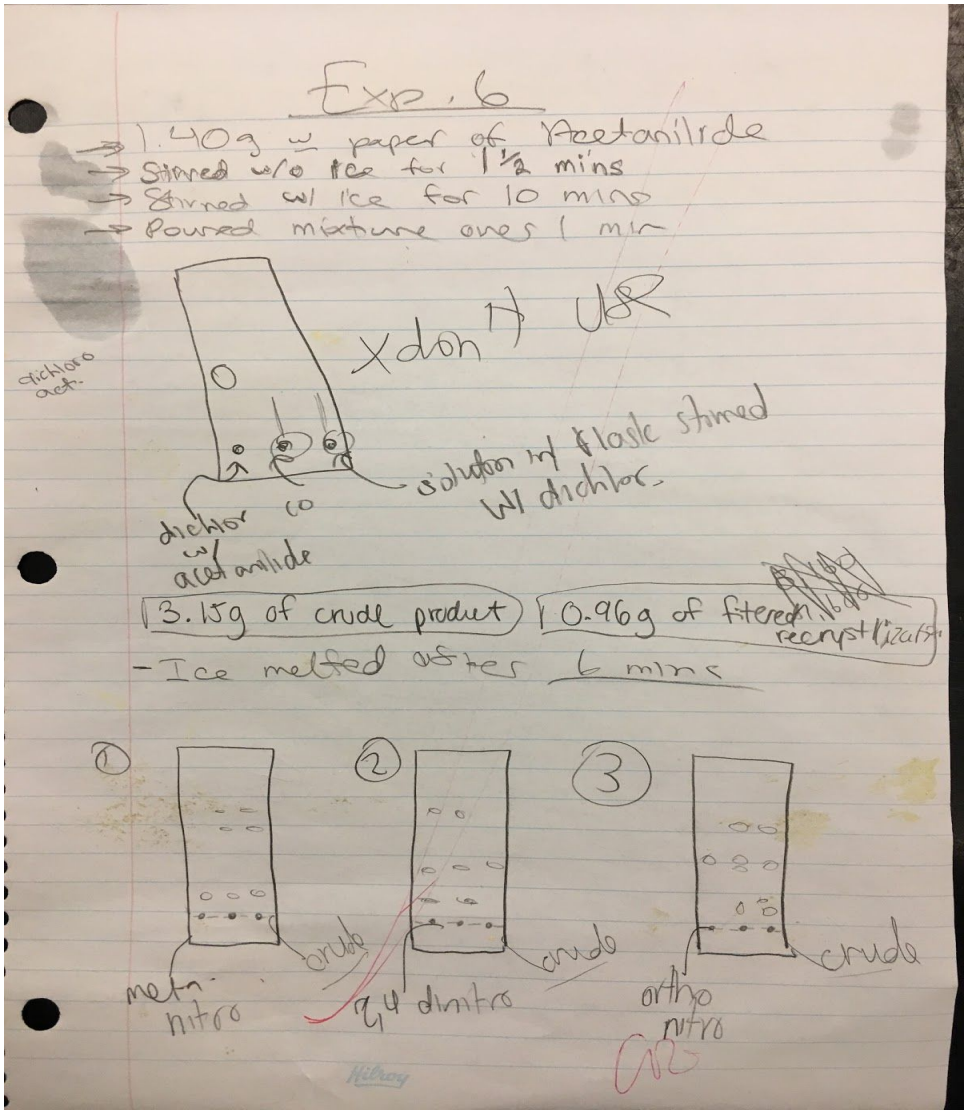
Percent yield = (1.2 g) / (1681.08 g) x 100% = 0.07%

5. This is an electrophilic addition reaction. The oxygen can use it's lone pairs as it is ortho directing to increase the electron cloud on the ring, emphasizing its ortho directing. This then leads to 1-nitrofuran being produced rather than 2-nitrofuran isomer.

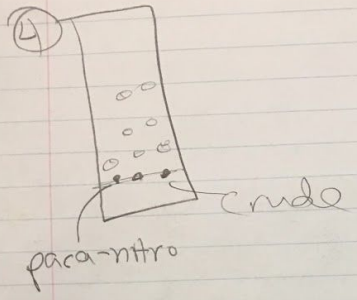
6. The meta site is the least nucleophilic between all three. Since the nitrogen on the pyridine N-oxide has a positive charge, the inductive effect becomes prevalent on the ortho site on the ring. This effect caused the nucleophilicity of the ortho site to lower.

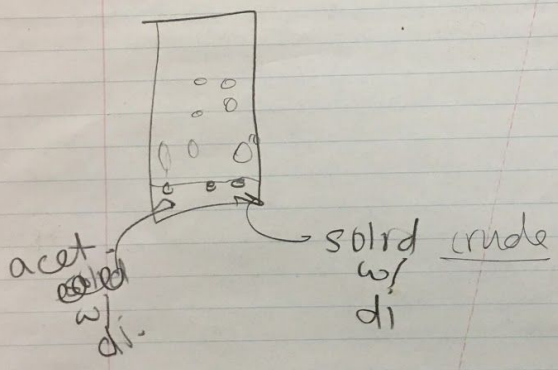


Raw Data


Exp. 6
1.40g ~ paper of Acetanilide
Stirred w/o ice for 1½ mins
Stirred w/ ice for 10 mins
Poured mixture over 1 min
dichloro acet.
xdon't use
solution in flask stirred w/ dichlor.
dichlor w/ acetanilide
3.15g of crude product
10.96g of filtered
recrystallized
-Ice melted after 6 mins
1
2
3
meta-nitro
crude
2,4 dinitro
crude
ortho nitro
crude

use plate 1 or 4 for Image J.





Last TLC