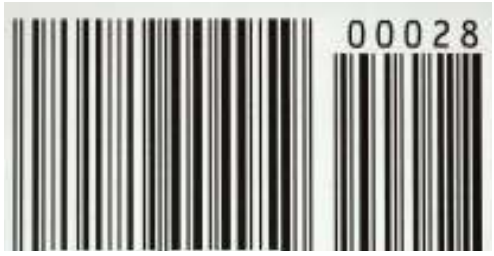
Composition Notebook

Student Name: Kip Nibly

Class: CHM 1025/1045/2210/2211

Instructor: Dr. Barnett



(Back of Front Cover)

Table of Contents

Table of Contents (Continued)

Table of Contents (Continued)

Experimental Title	Page Numbers

Calculation/Graph Page for Experiment #1:

Student Name: Kip Nibly

Experimental Title: Synthesis of Acetanilide

Date Started: 08/29/2011

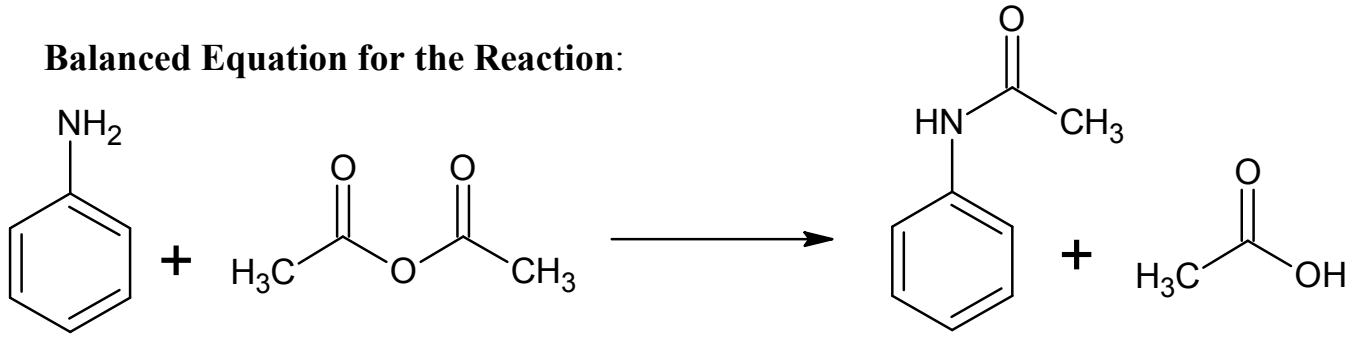
Unknown Letter/Number: Not Applicable (N/A)

Purpose of the Experiment: The purpose of this experiment is to perform a nucleophilic acyl substitution reaction on acetic anhydride with aniline to synthesize acetanilide. A solution of sodium acetate will be added as a base to deprotonate the water soluble intermediate and to liberate the product. Following crystallization in an ice-water bath, the product will undergo vacuum filtration to isolate. The product with be dried and the purity of the product will be assessed by running a melting point and by comparing the experimental value to the true value.

Reference to the Source of Procedure/Tables:

1) Barnett, "Experiment #7: The Complete Synthesis of Sulfanilamide from Benzene," *Experimental Laboratory Manual for Organic Chemistry-II*, 2012, pg. 60 – 72.

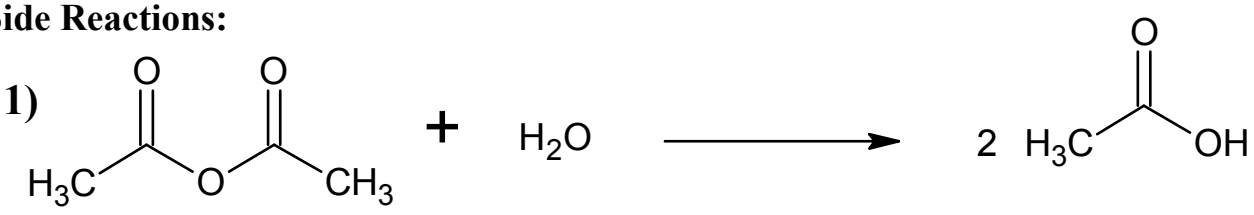
Balanced Equation for the Reaction:



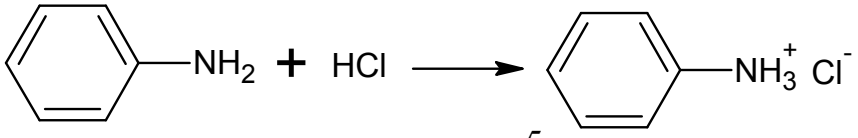
Aniline **Acetic Anhydride** **Acetanilide** **Acetic Acid**

Side Reactions:

1)



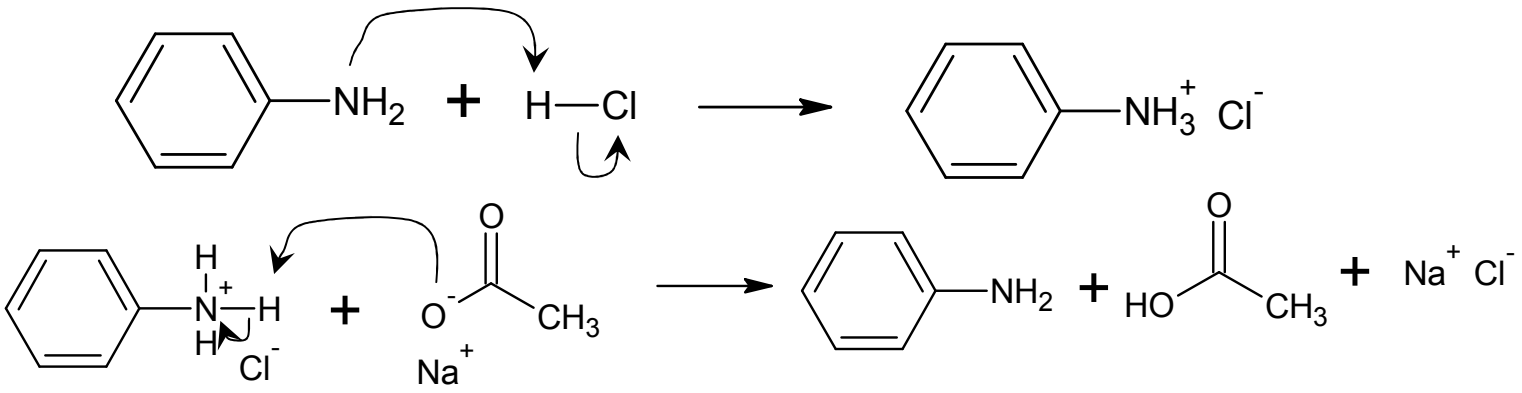
2)



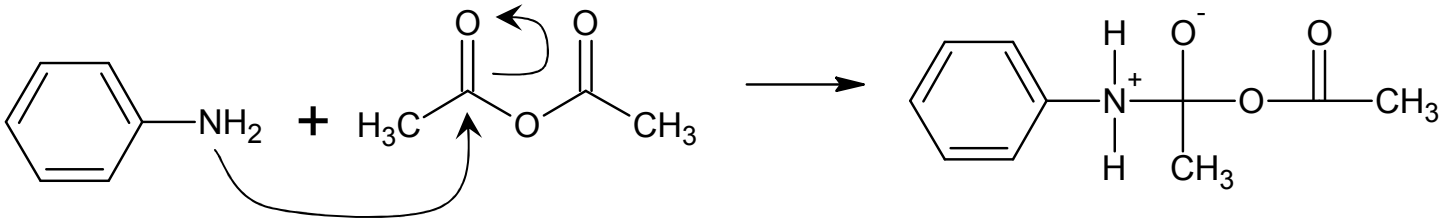
Calculation/Graph Page for Experiment #1:

Complete Reaction Mechanism:

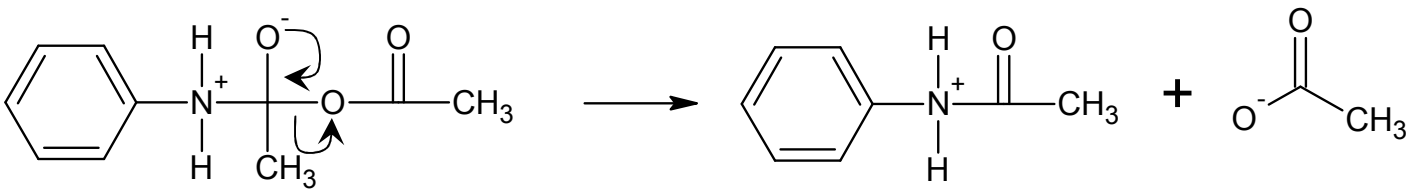
Protonation and Deprotonation of Aniline:



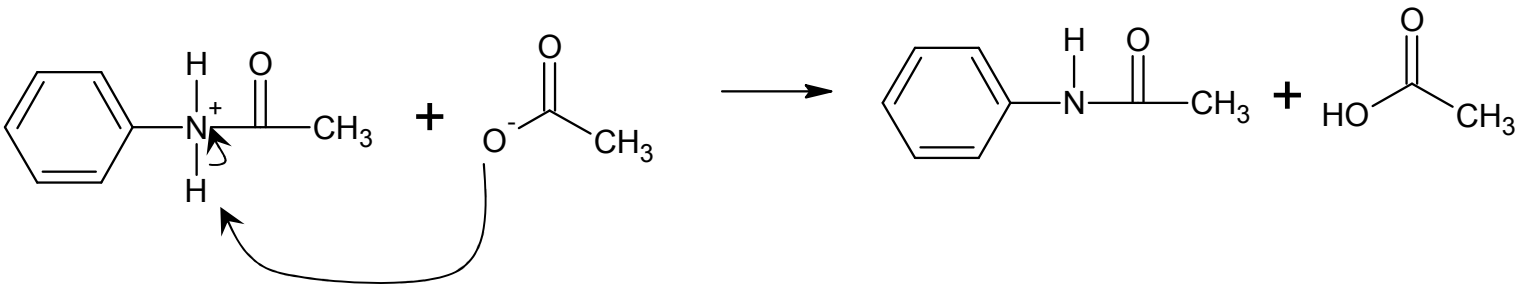
Nucleophilic Attack by Aniline on Acetic Anhydride:



Collapse of the Tetrahedral Intermediate and Loss of the Acetate Group:

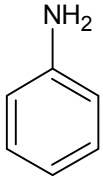
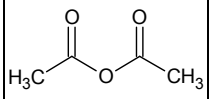
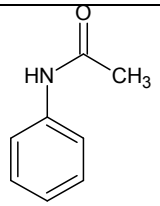
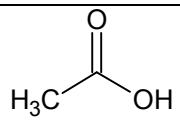
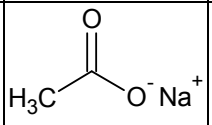


Proton Removal:



Calculation/Graph Page for Experiment #1:

Table of Physical and Chemical Properties for Reactants and Products:

Chemical Name	Atomic, Ionic, Molecular Formula and/or Structure	Atomic, Ionic, Molecular Mass or Weight: (in g/mol)	Melting Point (MP): (in ^{o}C)	Boiling Point (BP): (in ^{o}C)	Density: (g/mL)	Other Info: (such as pK_a, solubility, etc.)	Hazard and Safety Info:
aniline	 (C_6H_7N)	93.12 g/mol	–6^{o}C (solidifies)	184–186^{o}C	1.022 g/mL	pK_b = 9.30 Soly: 1g in 28.6 mL H_2O	Poison! Eye, skin, and lung irritant. Do not get on skin. Wear goggles, gloves, protective clothing
acetic anhydride	 ($C_4H_6O_3$)	102.09 g/mol	–73^{o}C	139^{o}C	1.080 g/mL	Hydrolyzes in water producing acetic acid	Irritant to eye, skin, and lungs. Flammable. Wear goggles & gloves
acetanilide	 (C_8H_9NO)	135.16 g/mol	113–115^{o}C	304–305^{o}C	1.219 g/mL	pK_a = 13.0 Soly: 1g/185 mL cold H_2O, 1g/20mL hot H_2O	Irritant to eye, skin, and lungs. Watch for allergic reaction. Wear goggles & gloves
acetic acid	 ($C_2H_4O_2$)	60.05 g/mol	16.7^{o}C	118^{o}C	1.049 g/mL	Miscible in water, alcohol	Acid! Burns skin, eyes. Wear goggles & gloves
sodium acetate	 ($C_2H_3O_2Na$)	82.03 g/mol	Anhydrous: 324^{o}C, hydrate: 58^{o}C	N/A	Hydate: 1.45 g/mL	N/A	May irritate skin, eyes, lungs; wear goggles & gloves
water	H_2O	18.02 g/mol	0.0^{o}C	100^{o}C	1.00 g/mL	N/A	Do not inhale

References: www.msdsxchange.com, www.msdshazcom.com, www.hazard.com, www.chemexper.com

Calculation/Graph Page for Experiment #1:

Procedure and Observations:

Procedure (In your own words)	**Observations**
1) Place 135 mL of deionized/distilled H_2O into 250 mL Erlenmeyer flask & then add 4.5 mL conc. HCl 2) Add 5.0 g aniline to flask 3) In 125 mL flask, place 5.3 g of sodium acetate + 30 mL H_2O 4) Add 6.2 mL of acetic anhydride to aniline soln then stir 5) Add sodium acetate solution to aniline solution + stir 6) Clamp into an ice-water bath for 20 min 7) Preweigh filter paper & watch glass 8) Vacuum filter w/ Buchner funnel & wash with ice cold water 9) Place onto watch glass & dry 10) Obtain mass of dried product and then run a melting point	1) Added 135.5 mL H_2O, 4.4 mL HCl, flask heated up 2) Volume of aniline measured = 5.2 mL, black and oily looking, dissolved slowly 3) Mass of sodium acetate measured = 5.42 g 4) Volume of acetic anhydride measured = 6.3 mL. Signs of precipitate forming after addition 5) After adding, precipitation increases 6) Start time: 12:30pm End time: 12:50pm 7) Mass of filter paper + watch glass: 67.55 g 8) Dark brown/tan crystals observed 9) Left in lab drawer for 1 week 10) Mass of filter paper + watch glass + product: 72.70 g. Melting point range: 111.5 – 115.0^oC

Waste Disposal and Clean Up:

Waste Disposal/Clean Up	**Action**
1) Aqueous filtrate should be neutralized (if acidic) with sodium bicarbonate & flushed down sink 2) Clean all glassware and return	1) Solution was slightly acidic, added 1 scoop of $NaHCO_3$, then flushed down sink 2) Done

Calculation/Graph Page for Experiment #1:

Volume of aniline to measure: 5.0 g aniline x (1 mL/1.022 g) = 4.9 mL

Actual volume of aniline obtained: 5.2 mL x (1.022 g/mL) = 5.3 g

Theoretical yield calculation:

1) Determination of limiting reactant and theoretical yield:

    a. From aniline: 5.3 g aniline x (1 mol aniline/93.12 g aniline) x (1 mole acetanilide/1 mol aniline) x (135.16 g acetanilide/1 mol acetanilide) = 7.7 g acetanilide **(Aniline is the limiting reagent)**

    b. From acetic anhydride: 6.3 mL acetic anhydride (AA) x (1.080 g AA/1 mL AA) x (1 mol AA/102.09 g AA) x (135.16 g acetanilide/1 mol acetanilide) = 9.0 g acetanilide **(Acetic anhydride is the limiting reagent)**

2) Percent yield calculation

    a. Actual yield: 72.70 g – 67.55 g = 5.15 g acetanilide

    b. Percent yield = (Actual yield/theoretical yield) x 100% = (5.15 g/7.7 g) x 100% = 67%

Data Table(s):

Data Collection Table for Lab

Volume of Water Added:	135.5 mL
Volume of HCl Added:	4.4 mL
Volume of Aniline Added:	5.2 mL
Mass of Sodium Acetate Measured:	5.43 g
Volume of Acetic Anhydride Used:	6.3 mL
Mass of Filter Paper + Watch Glass:	67.55 g
Mass of Filter Paper + Watch Glass + Product:	72.70 g

Data Table for Yield Information for Acetanilide

Limiting Reactant:	aniline
Theoretical Yield (for Acetanilide):	7.7 g
Actual Yield (for Acetanilide):	5.15 g
Percent Yield (for Acetanilide):	67%

Data Table for Melting Points (MP) for Acetanilide

Actual/Experimental Melting Point (for Acetanilide):	111.5 – 115.0^{o}C
Theoretical/True Melting Point (for Acetanilide):	113^{o}C – 115^{o}C

Calculation/Graph Page for Experiment #1:

Conclusion:

The purpose of this experiment was to perform a nucleophilic acyl substitution reaction on acetic anhydride with aniline to synthesize acetanilide. A solution of sodium acetate was added as a base to deprotonate the water soluble intermediate and to liberate the product. Following crystallization in an ice-water bath, the product underwent vacuum filtration to isolate. The product was dried and the purity of the product was assessed by running a melting point and by comparing the experimental value to the true value.

To an acidic aqueous solution, 5.2 mL of aniline was added producing the water soluble aniline hydrochloride. This was evident as the black oily liquid disappeared upon swirling. This was followed by the addition of 6.3 mL of acetic anhydride which produced some precipitation. Following the addition of an aqueous solution of sodium acetate, the acetanilide product formed much more readily by the observance of plentiful brown crystals. The product was isolated using a Buchner funnel via vacuum filtration using cold water as a rinse. Following the reaction and allowing to air dry, the mass of the acetanilide was determined to be 5.15 g. The limiting reactant was determined to be aniline and 7.7 g of product was theoretically possible. This allowed for a low 67% yield.

The low yield is likely due to a combination of factors. Some of the product was lost to the filter paper and Erlenmeyer flask upon filtration. This loss can decrease the percent yield. Since acetic anhydride undergoes hydrolysis in the presence of water, the high humidity conditions and wet glassware could account for the loss of a reactant. Furthermore, if the acetic anhydride provided was old, then this hydrolysis could have severely limited the amount of reactant available and thus would decrease the yield. Lastly, since the graduated cylinders and balances were not calibrated ahead of time, the masses and volumes used throughout the experiment could be unreliable.

The purity of the product was assessed by comparing the observed melting point to the theoretical range. The observed melting point of the product was 111.5–115^{o}C which was close to the theoretical value of 113–115^{o}C. Although reasonably pure, the wider range and depressed values indicate the presence of impurities. Improper drying and removal of water could account for this result. In addition, incomplete washing the product during the filtration process could have allowed for some acidic impurities to remain in the sample. This could have been remedied by recrystallizing the product.

The purity of the acetanilide product could have been assessed by running an infrared (IR) spectrum in KBr. The presence of an O–H stretch and/or a second carbonyl (C=O) stretch around 1750 cm^{-1} could indicate that water and/or acetic acid was not properly removed from the sample.

Kip Nibly 08/30/2011