**Chemistry 350**

Organic Chemistry I

Report Book

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Course team

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## Writing Laboratory Reports

In *Chemistry 350*, you are expected to prepare a report on each experiment as soon as possible after you have completed your lab sessions and to submit the report (PDF) to your Academic Expert for grading (Lab 3 and 4 have a joint report). It is recommended that you send in one or two reports initially to get feedback. Avoid sending them all at once.

**Short Report Forms**

Most experiments will only require filling in a laboratory form essentially highlighting key results and answering post-lab questions. The following are important points for short reports:

1. The short forms can be found in this Report Book.
2. The forms are in WORD so you can type directly into it and add graphics as necessary. If you wish to write your report by hand, make sure you provide yourself enough blank space to write legibly on any printed copy. Marks will be lost for reports that are hard to read.
3. Under any request for *Procedure*, you may simply refer to the relevant pages in the lab manual (referenced properly). Whatever you do, do not regurgitate the laboratory manual. If the procedure has been modified, or changed in any way, note the changes here.

**Formal Laboratory Reports**

Effective written communication is a vital skill for any scientist in disseminating their work. To this end, you will build up your own writing skills through the creation and submission of two (2) formal laboratory reports. One formal report is required for Labs 3 and 4 combined and one for Lab 9. These reports will be done in a format of an article that might be submitted to a peer-reviewed journal by a professional chemist. We provide a sample student report from an introductory organic chemistry laboratory at the University of Toronto in this Report Book to illustrate how this might look.

**These reports must be typed in WORD and not handwritten.** Once complete, **save the report as a PDF** and send to your Academic Expert. Reaction schemes and mechanisms included must be drawn using a chemical drawing program such as ChemDraw or ChemSketch.[[1]](#footnote-1)

Use the ‘journal-style’ formal laboratory WORD template provided in this Report Book.

The template contains the following components:

*Title of Experiment & Author(s)*

Must be different from the title of the experiment found in the laboratory manual.

Needs to be clear and concise (i.e., not long or vague, and not “Experiment #”).

Include your name, student ID number, the date, and lab instructor name (listed as a co-author).

*Abstract (30 – 100 words)*

It is best to write this last. The abstract should be “stand on its own” (be independent of the rest of the report and have a clear beginning, middle, and end). Include the purpose of the work and a brief summary of techniques and findings. (Do not provide data or specific details here.)

*Introduction (100-300 words)*

Give a brief introduction to the purpose of the experiment and the approach to be used.

Provide background information for the reader and demonstrate that you understand the objective and the key concepts of the experiment. (Do not copy directly from the laboratory manual.) Reference any previous or similar work in the literature. You may include relevant balanced and fully labelled chemical equations at this point.

Use only the third person, present tense, passive voice when writing the introduction. For example,

Correct: In this experiment, cyclohexanol is converted to cyclohexene using......

Incorrect: In this experiment, I will be performing an acid catalyzed dehydration…

*Experimental Procedure (50-200 words)*

Describe the practical details of the experiment without reporting or interpreting experimental results. (Whatever you do, do not regurgitate the laboratory manual.)

If the procedure has been modified, or changed in any way, note the changes here.

Remember that the procedure section should be sufficiently detailed for another student to be able to repeat the whole experiment based on your report.

Finally, keep the following points in mind:

i. use the third person, the passive voice, and the past tense.

**Correct:** The solution was heated on a hot plate for 30 minutes.

**Incorrect:** I heated the solution on a hot plate for 30 minutes.

**Incorrect:** The solution is heated on a hot plate for 30 minutes.

ii. avoid the “recipe format”.

**Incorrect:** Heat the solution on a hot plate for 30 minutes.

iii. avoid incorporating your observations or result explanations into the procedure.

**Incorrect:** The solution was heated on a hot plate for 30 minutes, during which time the colour of the solution changed from red to green as the hydroxyl group was oxidized to the ketone.

iv. avoid unnecessary detail (even though the lab manual often has this for you).

**Acceptable:** 20 mL of hydrochloric acid (3 mol L−1) was added to the solution with constant stirring.

**Unnecessary detail:** 20 mL of 22.5° C hydrochloric acid (3 mol L−1) was poured from a graduated cylinder into a 100-mL beaker containing the solution. During this process the solution in the beaker was stirred with a 15-cm long glass rod having a diameter of 5 mm.

v. bracket amounts when indicating how much compound was used.

**Correct:** Compound Y (152.3 mg, 1.27 mmol) was added slowly.

**Incorrect:** 152.3 mg or 1.27 mmol of Compound Y was added slowly.

*Results & Discussion (300-800 words)*

This is most important section of your report.

Summarize the results of the experiment (e.g., compounds synthesized, yields, characterization). Wherever possible, tabulate your data. Use the sample student report as a guide for reporting NMR data. For IR spectral assignments note sample preparation (Nujol mull, chloroform thin film, KBr disk, neat, etc.), major peaks, and corresponding functional group.

Example: IR (Nujol): 3060 and 3030 (aromatic C-H stretch), 2950 and 2835 (aliphatic C‑H stretch), 1600 and 1498 (benzene ring C=C stretch), 1247 (asymmetric C-O-C stretch), 1040 (symmetric C-O-C stretch), 795 (out-of-plane C-H bend) cm−1.

Show your calculations for the % yield in the Supporting Information section. Include all spectra (usually IR) in the Supporting Information. Never make written assignments directly on the spectra. The discussion portion gives you an opportunity to discuss the significance of your results, to assess the validity of the method, to indicate possible reasons for a poor yield, and so on. Remember to provide literature references where appropriate. Do not over-comment on NMR and IR spectra, just pick out and comment on the spectral peaks of importance. Add the answers to the post lab questions, each one beginning in a separate paragraph.

*Conclusions (40-150 words)*

You would usually include a short paragraph that summarizes your results and puts them into some kind of context. A good conclusion is sometimes very hard to write. You have to address the purpose you've mentioned at the start of the experiment (do not repeat the purpose word for word!!), mention your key result and say something about the success/failure of the experiment and its relevance beyond the work presented.

*Acknowledgements*

A chance to acknowledge people and other resources used.

*Supporting Information Available*

List additional information you will be providing at the end of the formal report (in the same order as it will appear). Also, remember to note for the reader within the text of the report itself any relevant information that might be available within Supporting Information.

*References*

Ensure that the report is properly referenced. Please adopt the format used by the American Chemical Society (ACS). See for example ACS Style Quick Guide at: https://pubs.acs.org/doi/full/10.1021/acsguide.40303

*Mandatory “Academic Honesty” Pledge*

This must be typed at the end of the report and signed/dated: use the exact wording given in the template.

*Supporting Information*

A table of reagent properties: the name of each reagent used, molecular weight, density, amount used, mmol used, melting point (mp) and boiling point (bp) (where appropriate). For solvents, include amount used, mp, bp, and density. For all chemicals include the hazards of each one. Calculations for the reaction yield(s). All original spectra (NMR, infra-red etc.) obtained from your results sheet but NO SPECTRAL ASSIGNMENTS.

## Formal Report Template Organic Transactions CHEM 350

CHEM 350

ORGANIC TRANSACTIONS

**Laboratory Report Title**

Student Name (ID Number)\* and Lab Instructor(s) Name(s)

*Faculty of Science & Technology, Athabasca University, 1 University Drive, Athabasca, Alberta, Canada T9S 3A3*

Received Month Day Year; E-mail xxxx@xxxxx

**Abstract –** *\*write this last\**

**Introduction**

Write the introduction here, including the purpose of the experiment, a description of the reaction performed as well as the reaction scheme and mechanism.

A black line drawing of a molecule

Description automatically generated

***Figure 1.*** *4-Hydroxy-butanoic acid.*

A black background with a cat in the middle

Description automatically generated

***Scheme 1.*** *Hydrogenation of cyclohexene.*

**Experimental Procedure**

Write this in your own words, passive voice, past tense – do not directly copy from laboratory manual.

*\*\*Please note:* Include a table of reagent properties in the Supporting Information.

**Results & Discussion**

Include product yield and characterization information. If a purification was performed, you must include a crude and purified yield for your product.

*\*\*Please note:* Show all yield calculations and include them in the Supporting Information.

Include all product spectral data, assignments and analysis, including IR and NMR peak assignments and comparisons with literature values.

Make sure to include the answers to the post-lab questions.

**Conclusions**

Include a summary of the experiment.

**Acknowledgements.** Financial support from Athabasca University Government of Alberta is gratefully acknowledged. Consultation with [Lab Tech Name/Lab Coordinator] was very much appreciated.

**Supporting Information Available**

Here, state all of the contents included in the supporting information, e.g. “Yield calculations and a sketch of the TLC plate from the experiment can be found in the Supporting Information”. Then, provide the information listed in the Supporting Information after the report.

**References**

Please use American Chemistry Society (ACS) style.

(1)

(2)

I certify that this submitted laboratory report represents entirely my own efforts. I have read and understand the Athabasca University policies regarding, and sanctions for, plagiarism.

Signature: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

Date: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

***\*\*Please note:*** Be sure to review the instructions for writing a formal lab report provided in the “CHEM 350 Lab Manual”

**Supporting Information to:**

**Laboratory Report Title**

Student Name\* and Lab Instructor(s) Name(s)

*Faculty of Science & Technology, Athabasca University, 1 University Drive, Athabasca, Alberta, Canada T9S 3A3*

Received Month Day Year; E-mail xxxx@xxxxx

Include a table of reagent properties, yield calculations, spectra, etc.

\*\*Please note: Remember to label information for identification and have the items in the same order as stated in your “Supporting Information Available” list of the report.

## A blue and white logo Description automatically generated Sample Report: CHM 249 Communications

Using Lemon Juice as Greener Solvent Alternative and Catalyst in Imine Synthesis for Reductive Amination

Student Name

*Department of Chemistry, University of Toronto, 80 St. George St., Toronto, Ontario, Canada M5S 3H6*

Received 22 February, 2024

**Abstract**

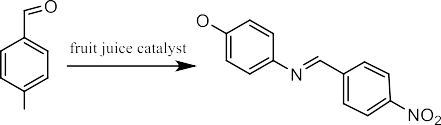
Amine compounds are important functionalities found in a variety of biological molecules and synthetic drugs. This report demonstrated

a more environmentally-friendly method of synthesising the imine compound N-(4-methoxyphenyl)-1-(4-nitrophenyl)methanimine from condensation of 4-nitrobenzaldehyde and 4-anisidine under reflux conditions using lemon juice as both catalyst and solvent. The imine was purified by recrystallisation and underwent reductive amination with NaBH4 to form the final amine product 4-methoxy-N-(4-nitrobenzyl)aniline. Both imine and amine samples were collected and dried by vacuum filtration. The reaction generated moderate yield and purity based on analysis through melting point determination and 1H NMR. The protocol adhered to several Principles of Green Chemistry, including the use of safer solvents, catalysis and energy efficient design. Overall, this experiment aimed to explore greener and more efficient alternative reaction conditions to traditional catalysts and organic solvents that still provide the desired amine product with high yield.

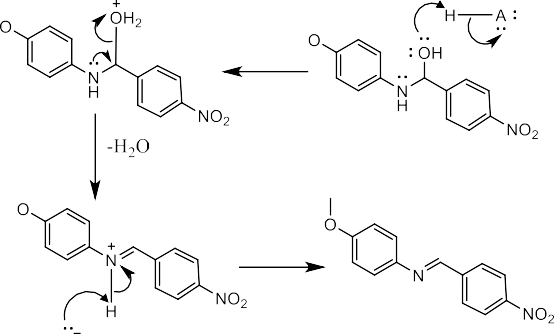
**Introduction**

Amine compounds are important functionalities found in biological molecules and synthetic drugs. Amine groups are engaged in biologically-significant processes within the body, such as the formation of peptide bonds with carboxylic acid groups to produce various proteins. Pharmaceutically active ingredients including fentanyl and verapamil also contain amine groups to increase drug solubility and activate its key physiological therapeutic effects.1-3 Owing to the necessity of amine compounds, reductive amination is one of the most frequently used synthetic methods in medicinal chemistry to generate amine C-N bonds.4-6 However, like many other chemical processes, reductive amination requires large quantities of organic solvents which are often toxic, corrosive, and environmentally harmful. The use of separate chemical species as solvents and catalysts further increases the risk of handling hazardous material while more quickly depleting non-renewable resources. Hence, there exists an opportunity to explore more environmentally-friendly and efficient reaction conditions that still provide the desired amine product with high yield.

This experiment aimed to synthesise the amine compound 4-methoxy-N-(4-nitrobenzyl)aniline through a two-step process with greater adherence to the Twelve Principles of Green Chemistry.7 In the first step, the intermediary imine product N-(4-methoxyphenyl)- 1-(4-nitrophenyl)methanimine was formed from condensation reaction between 4-anisidine and 4-nitrobenzaldehyde, with lemon juice acting as the greener alternative to traditionally-used catalysts and organic solvents (*Scheme 1*). This encourages the use of natural, renewable, and non-toxic resources through repurposing “ugly food”, specifically fruits or vegetables that are aesthetically unappealing and go to waste as a result. The shorter reaction time of 15 minutes additionally lends itself to energy efficiency, which is another advantage to the proposed method involving lemon juice. In the second step, the recrystallised imine was reduced to the amine final product using NaBH4 under reflux conditions with methanol solvent (*Scheme 2*). This experiment also aimed to analyse the purity of synthesised imine and amine compounds using 1H nuclear magnetic resonance (NMR) spectrometry and melting point determination.



***Scheme 1.*** *Condensation reaction between 4-anisidine and 4- nitrobenzaldehyde to form imine with fruit juice acting as both catalyst and solvent, carried out at room temperature.*

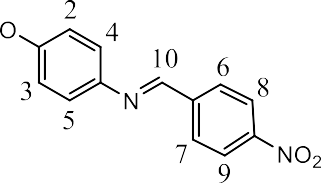
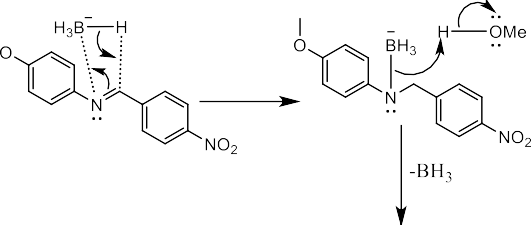


***Figure 1.*** *Reaction mechanism for condensation between 4-anisidine and 4-nitrobenzaldehyde to form imine.*



***Scheme 2.*** *Reduction of imine to amine using NaBH4 under reflux conditions with methanol solvent.*

point temperature range, its identity could still be confirmed through 1H NMR. The absence of amine proton broad peak between 1.0 ppm and 5.0 ppm and aldehyde proton peak between 9.0 ppm and 10.0 ppm in its 1H NMR spectrum indicated that the reaction proceeded to completion with no starting materials remaining in the imine sample. The experimental yield was moderately higher than the literature value of 41% for the same method, possibly due to incomplete removal of isopropanol solvent after only a short drying period, and remaining isopropanol would contribute to the measured mass of imine sample.9 However, the experimental yield was lower than the literature value of 98% when MgSO4 was used as catalyst instead, suggesting that lemon juice catalysis was not the most effective method.



***Figure 2.*** *Reaction mechanism for reduction of imine to amine using NaBH4.*

**Experimental Procedure**

To synthesise the imine, 4-nitrobenzaldehyde (0.4581 g, 3.031 mmol) and 4-anisidine (0.4060 g, 3.297 mmol) were vigorously stirred with juice (10 mL) extracted from a lemon for 15 minutes at room temperature. After the reaction was complete, the solid imine product was collected by vacuum filtration, washed with distilled water (15 mL) and dried under vacuum for 10 minutes. The crude imine was then purified through recrystallisation by first dissolving in a minimal volume of hot isopropanol (approximately 20 mL). The solution was allowed to cool to room temperature, then placed in an ice bath for 10 minutes to complete crystallisation. The crystals were collected by vacuum filtration, washed with a small portion of ice-cold isopropanol, and dried under vacuum for 10 minutes. The recrystallised imine was weighed, and its melting point temperature range measured using the Fischer-Johns melting point apparatus.

To convert the imine to amine, the recrystallised imine was dissolved in methanol (10 mL) and heated in a hot water bath to reflux. Once refluxing, NaBH4 (0.1906 g, 5.038 mmol) was added slowly and the reaction mixture was stirred vigorously under reflux for 30 minutes. After the reaction was complete, the solution was neutralised to pH 7 using aqueous HCl (1 M, approximately 3mL), then cooled in an ice water bath for 10 minutes. The solid amine product was collected by vacuum filtration, washed with a small portion of cold methanol and dried under vacuum for 10 minutes before being weighed. Its melting point temperature range was also measured using the Mel-Temp melting point apparatus.

**Results & Discussion**

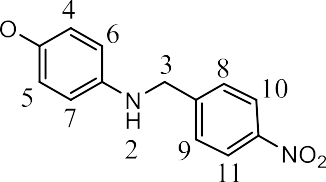
The recrystallised intermediary imine product (*Figure 3*) was a bright yellow crystalline powder with a melting point temperature range of 137.2 °C to 142.9 °C. The theoretical yield was 0.7767 g while the actual yield was 0.5094 g, giving a percentage yield of approximately 65.59%. The synthesised imine was slightly impure. The measured melting point temperature range was above the literature values of 131.1 °C to 133.5 °C, and wide with a maximum difference of 5.7 °C.8 This could be caused by aqueous impurities insoluble in isopropanol that were collected with imine crystals since gravity filtration was not performed before recrystallisation. Another factor may be the large size of imine crystals hindering heat transfer into their interior, resulting in non- uniform heating to give the appearance of a higher melting point and account for the wide melting temperature range. While the synthesised imine was not entirely pure due to its deviated melting

***Figure 4.*** *Skeletal structure of N-(4-methoxyphenyl)-1-(4- nitrophenyl)methanimine with labelled protons for 1H NMR characterisation.*

*The 1H NMR characterisation is as follows: 1H NMR (300 MHz, CDCl3) δ 8.58 (s, 1H, H-10), 8.30-8.33 (d, 2H, H-8 or H-9), 8.04-*

*8.07 (d, 2H, H-6 or H-7), 7.26-7.32 (t, 2H, H-4 or H-5), 6.95-6.98 (d, 2H, H-2 or H-3), 3.85 (s, 3H, H-1).*

The final amine product (*Figure 4*) was a dry light orange powder with a melting point temperature range of 91.7 °C to 94.3 °C. The theoretical yield was 0.7828 g while the actual yield was 0.1065 g, giving a percentage yield of approximately 13.61%. The synthesised amine was comparatively purer than the imine. The measured melting point temperature range was slightly below the literature melting point temperature of 95 °C to 96 °C, and relatively narrow with a maximum difference of 2.6 °C.10 This could be caused by incomplete removal of methanol solvent from the insufficiently extended drying process. Since the amine was more soluble in methanol at higher temperatures, a portion would dissolve in the remaining methanol, giving the appearance of melting at a lower temperature than the actual melting point. Trace amounts of aqueous impurities insoluble in methanol may have also been retained and collected with the amine precipitate. However, since the difference between experimental and literature melting point temperatures was minimal, the final amine product could be considered mostly pure. The identity and purity of the sample was further supported by its 1H NMR spectrum showing a characteristic broad peak at 3.98 ppm for the amine proton. The experimental yield was significantly lower than the literature value of 48%, owing to incomplete product formation as reaction mixture temperature under reflux was not consistently maintained at boiling point of methanol or product loss during washing of amine precipitate.9



***Figure 4.*** *Skeletal structure of 4-methoxy-N-(4-nitrobenzyl)aniline with labelled protons for 1H NMR characterisation.*

*The 1H NMR characterisation is as follows: 1H NMR (300 MHz, CDCl3) δ 8.17-8.20 (q, 2H, H-10 or H-11), 7.52-7.55 (d, 2H, H-8 or*

*H-9), 6.75-6.78 (q, 2H, H-6 or H-7), 6.53-6.56 (q, 2H, H-4 or H-5),*

*4.43 (s, 2H, H-3), 3.98 (br s, 1H, H-2), 3.73 (s, 3H, H-1).*

The citrus juices were aqueous in nature and able to form favourable hydrogen bonding with polar groups on 4- nitrobenzaldehyde and 4-anisidine, allowing the starting materials to dissolve in solution for condensation reaction to occur. Additionally, the citrus juices were within acidic pH range, and likely contained natural acids that also act as catalyst by donating a proton to the carbinolamine group of the intermediate (*Figure 5*) to become ionised. Protonation of the carbinolamine oxygen converted the alcohol group into the better leaving group OH +, which was more readily removed as water to promote formation of the imine C=N double bond at a faster rate. At pH = 7, pH of the solution would have exceeded the pKa of natural acids present in the citrus juice, causing the equilibrium to shift in favour of a greater extent of acid dissociation. A larger portion of the acid exist as its deprotonated conjugate base, losing its ability to function as catalyst. The reaction would proceed at a slower rate, requiring more time and harsher conditions to go to completion. At pH = 0.5, pH of the solution was likely lower than pKa of the aminobenzoic group of 4-anisidine, considered a weak base due to delocalisation of the lone electron pair on nitrogen into the benzene ring pi system by resonance. The amine group would become protonated such that the lone electron pair became less accessible and unavailable to act as nucleophile to attack the partially-positive carbon of carbonyl group on 4- nitrobenzaldehyde as the first step of the mechanism, causing the reaction to also proceed at a slower rate.

2



***Figure 5.*** *Intermediate formed from third step of condensation reaction containing carbinolamine group.*

The CHM249 method for imine formation was greener than the literature method. Concerning Principle 5: Safer Solvents and Auxiliaries, the CHM249 method only required lemon juice as both solvent and catalyst, while the literature method utilised separate compounds of MgSO4 as catalyst and dichloromethane (CH2Cl2) as reaction solvent. In contrast to naturally-derived lemon juice that was safe to handle with no associated toxicity, dichloromethane was carcinogenic and acutely toxic, posing a greater danger when working with it. Additionally, the literature method required 30 minutes for reaction to be complete, significantly longer than 15 minutes for the CHM249 method, which could therefore be considered more economical and efficient.

**Conclusions**

Although traditional methods produced a higher yield, the use of lemon juice as greener alternative to traditional catalysts and organic solvents could still be considered a sufficiently effective and efficient protocol for imine synthesis and reductive amination. Despite only moderate purity as indicated by inconsistencies in melting point temperatures, it would be reasonable to conclude that the imine compound N-(4-methoxyphenyl)-1-(4- nitrophenyl)methanimine was successfully synthesised and converted to the amine compound 4-methoxy-N-(4- nitrobenzyl)aniline when further corroborated with physical appearance of the products and 1H NMR spectral data.

**Acknowledgements**

Financial support from the University of Toronto and Government of Ontario is gratefully acknowledged.

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I certify that this submitted laboratory report represents entirely my own efforts. I have read and understand the University of Toronto policies regarding, and sanctions for, plagiarism.

Signature:

Date: 22 February, 2024

***Supporting Information to:***

***Using Citrus Juice as Greener Solvent Alternative and Catalyst for Reductive Amination***

*Department of Chemistry, University of Toronto, 80 St. George St., Toronto, Ontario, Canada M5S 3H6*

Received 22 February, 2024

**Table of Reagent, Solvent and Product Properties**

***Table S1.*** *Properties of reagents, solvents and products involved in reductive amination.*

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| **Compound Name** | **Amount Used** | **Molecular Weight (g/mol)** | **Molar Amount (mmol)** | **Molar Ratio** | **Physical Properties** | **Safety Information** |
| 4-anisidine | 406.0 mg | 123.152 | 3.297 | 1.0 | density: 1.07 g/mL  melting point:  57.2 °C  boiling point: 243 °C | Carcinogenic and acutely toxic, fatal if swallowed or if inhaled. |
| 4-nitrobenzaldehyde | 458.1 mg | 151.12 | 3.31 | 1.0 | density: 1.546 g/mL  melting point: 103 °C – 106 °C  boiling point: approximately 300 °C | Causes serious eye and skin irritation. |
| isopropanol | 20 mL | 60.094 | -- | -- | density: 0.786 g/mL  melting point: - 89 °C  boiling point:  82.3 °C | Highly flammable liquid and vapour, causes serious eye irritation. |
| N-(4-methoxyphenyl)-1- (4-  nitrophenyl)methanimine | -- | 256.256 | -- | 1.0 | -- | -- |
| methanol | 10 mL | 32.042 | -- | -- | density: 0.792 g/mL  melting point: -  97.6 °C  boiling point:  64.7 °C | Highly flammable liquid and vapour, toxic if swallowed or inhaled. |
| sodium borohydride | 190.6 mg | 37.832 | 5.038 | 1.0 | density: 1.074g/mL | Emit flammable |

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  |  |  |  |  | melting point: > 400 °C  boiling point: > 500 °C | gases when in contact with water, acutely toxic if swallowed, corrosive in causing skin and eye irritation. |
| aqueous hydrochloric acid | 3 mL | 36.458 | -- | -- | density: 1.0585 g/mL | Corrosive, causes serious eye and skin irritation. |
| 4-methoxy-N-(4- nitrobenzyl)aniline | -- | 258.272 | -- | 1.0 | -- | Combustible solid, causes severe eye damage, acutely toxic and fatal if swallowed or inhaled. |

**Percentage Yield Calculations**

A close-up of a math problem

Description automatically generated

***Figure S1.*** *Calculations for theoretical yield and experimental percentage yield.*

**Nuclear Magnetic Spectroscopy (NMR) Spectra**

A graph of a chemical structure

Description automatically generated

***Figure S2.*** *NMR spectrum for intermediary imine product.*

A graph of a chemical formula

Description automatically generated

***Figure S3.*** *NMR spectrum for final amine product.*

# Experiment 1: Melting-point Determinations

**Objectives**

1. This experiment is designed to introduce you to the use of a typical “melting-point apparatus”. Which of the numerous types of “melting-point apparatus” you will use may depend on the location at which you carry out the laboratory component of this course. You will use the “melting-point apparatus” repeatedly throughout this course.

2. To demonstrate that pure compounds have “sharp” melting points; that is that pure compounds melt over a small temperature range.

3. To demonstrate how an impurity lowers the melting point of a substance and broadens its melting range.

4. To illustrate the use of the “mixed melting-point” procedure.

**Write-up**

Fill in the following form below and answer the post-lab questions. Use the WORD version of the report form so you can add additional space for your answers. When complete save as a PDF and email as an attachment to your Academic Expert for grading.

[Hint: Do not send all reports in at the same time. Initially send only 1-2 reports to first obtain feedback for later reports.]

**CHEM 350 Experiment 1 Report Form**

**Melting-point Determinations**

**Date:\_\_\_\_\_\_\_\_\_\_\_**

**Student Name:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ ID Number:\_\_\_\_\_\_\_\_\_\_\_**

**Part A**

Melting point of sample # \_\_\_\_\_\_\_\_\_\_\_\_ = \_\_\_\_\_\_\_\_\_\_\_\_\_\_

Suggest identity of unknown compound \_\_\_\_\_\_\_\_\_\_\_\_\_

**Part B**

Possible identity of unknown compound # \_\_\_\_\_\_\_\_\_\_\_\_\_ :

1. \_\_\_\_\_\_\_\_\_\_\_\_\_ ; m.p. (Reference )

2. \_\_\_\_\_\_\_\_\_\_\_\_\_ ; m.p. (Reference )

Melting point of unknown compound # \_\_\_\_\_\_\_\_\_\_\_\_ = \_\_\_\_\_\_\_\_\_\_\_\_\_

Melting point obtained when unknown compound # \_\_\_\_\_\_\_\_\_\_\_\_ is mixed with

1. \_\_\_\_\_\_\_\_\_\_\_\_\_\_ = \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

2. \_\_\_\_\_\_\_\_\_\_\_\_\_\_ = \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

Conclusion: The above results indicate that unknown compound # \_\_\_\_\_\_\_\_\_\_ is   
probably \_\_\_\_\_\_\_\_\_.

**Questions**

1. In the introduction to this experiment, you were warned that heating the sample too quickly in the region of the melting point will result in the experimentally determined melting point being higher than the true value. Explain why this is so.
2. What is a “eutectic mixture”? How would you decide whether a given sample was a pure compound or an eutectic mixture of two compounds?
3. You are working in the lab, and you find an unlabelled vial with a white crystalline solid inside. To determine the identity of the compound, what would you do?

I certify that this submitted laboratory report represents entirely my own efforts. I have read and understand the Athabasca University policies regarding, and sanctions for, plagiarism.

Signature: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Date: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

# Experiment 2: Recrystallization

**Objectives**

The purpose of this experiment is to show how organic compounds can be purified through the process of recrystallization. Techniques used in the experiment include hot gravity filtration and vacuum filtration. You will also learn more about the solubility of organic compounds and the use of activated charcoal. You will use the compound that you purify, acetanilide, in a subsequent experiment.

**Write-up**

Fill in the following form below and answer the post-lab questions. Use the WORD version of the report form so you can add additional space for your answers. When complete save as a PDF and email as an attachment to your Academic Expert for grading.

[Hint: Do not send all reports in at the same time. Initially send only 1-2 reports to first obtain feedback for later reports.]

**CHEM 350 Experiment 2 Report Form**

**Recrystallization**

**Date:\_\_\_\_\_\_\_\_\_\_\_**

**Student Name:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ ID Number:\_\_\_\_\_\_\_\_\_\_\_**

**Procedure:**

(Ref: )

Changes/Modification:

**Results**

**Table 1. Observations**

|  |  |
| --- | --- |
| **Procedural Step** | **Comment or Observation** |
| Recrystallization solvent used. |  |
| Volume of recrystallization solvent used. |  |
| Hot filtration (solids present) |  |
| Appearance of solution after addition of charcoal |  |
| Time allowed for crystals to form. |  |
| Second crop . . . |  |
|  |  |
|  |  |

**Table 2. Product Recrystallization Results**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|  | Mass of Impure Acetanilide (g) | Mass of Pure Acetanilide Recovered (g) | Appearance of Crystals | % Recovery Yield | Melting Point  (°C) |
| Impure acetanilide |  |  |  |  |  |
| 'Pure' acetanilide |  |  |  |  |  |
| 2nd crop 'Pure' acetanilide |  |  |  |  |  |

Show % recovery yield calculation for your first crop.

**Questions**

1. The table below shows the solubility of a certain organic compound in water at five different temperatures.

Temperature (o C) Solubility of compound (in 100 mL of water)

0 1.5 g

20 3.0 g

40 6.5 g

60 11.0 g

80 17.0 g

a. Plot a graph of the solubility of the compound versus temperature. Draw a smooth curve through the data points.

b. If a student attempts to recrystallize a 0.5 g sample of this compound by heating it to 80o C with 5.0 mL of water, would all of the sample dissolve? Briefly justify your answer.

c. Assuming that the answer to part b is “Yes”, at what temperature will the crystals begin to appear when the student’s solution begins to cool?

d. If the student cooled the solution to 0o C and filtered off the crystals, what is the maximum possible percentage recovery? What mass of the sample will remain in the filtrate?

1. Explain why you should slowly cool the hot filtered saturated solution obtained in the recrystallization procedure?
2. During the last step of the recrystallization procedure, you collect the crystals by vacuum filtration. Why do you use ice cold recrystallization solvent to help transfer all the crystals to the Büchner funnel and wash the crystals?
3. Briefly explain the circumstances under which a mixed solvent recrystallization method would be used to recrystallize a given compound.

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Signature: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Date: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

# Experiment 3: Distillation

**Objectives**

This experiment is designed to:

1. demonstrate how a liquid may be purified by simple distillation and its boiling point determined during the process.

2. illustrate how two liquids can be separated by fractional distillation.

**Write-up**

One single formal report of Experiments 3 and 4 together will be required. Use the formal report template (WORD) in the Report Book and follow the instruction outlined in “Writing Laboratory Reports.” When complete save as a PDF and email as an attachment to your Academic Expert for grading.

# Experiment 4: Refractive Index

**Objectives**

This experiment is designed to

1. illustrate the use of refractive index as a criterion of purity.

2. demonstrate the use of refractive index in estimating the composition of a mixture of two liquids.

**Write-up**

One single formal report of Experiments 3 and 4 together will be required. Use the formal report template (WORD) in the Report Book and follow the instruction outlined in “Writing Laboratory Reports.” When complete save as a PDF and email as an attachment to your Academic Expert for grading.

**Questions**

Answers are to be included with your report.

1. Suggest a reason why the boiling point of cyclohexanol is so much higher than those of cyclohexane and toluene.
2. Suggest a reason why the refractive index of cyclohexanol is higher than that of water.
3. To reduce the percentage error in the *n*D reading of your purified cyclohexanol (compared to the literature value), what should you do?
4. Is there a way you could suggest to improve the experiment (should you do it again)?

# Experiment 5: Extraction, separation and the use of drying agents

**Objectives**

This experiment is designed to

1. demonstrate how a solute can be extracted from one solvent to another.

2. show how a mixture of organic compounds can be separated into its components on the basis of differences in acidity and basicity.

3. illustrate the use of a drying agent to remove traces of water from non-aqueous solutions.

1. introduce the concept of using a flow-chart to summarize laboratory procedures.

**Write-up**

Fill in the following form below and answer the post-lab questions. Use the WORD version of the report form so you can add additional space for your answers. When complete save as a PDF and email as an attachment to your Academic Expert for grading.

**CHEM 350 Experiment 5 Report Form**

**Extraction, separation, and the use of drying agent**

**Date:\_\_\_\_\_\_\_\_\_\_\_**

**Student Name:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ ID Number:\_\_\_\_\_\_\_\_\_\_\_**

**Procedure:**

(Ref: )

Note any changes/modifications:

**Part A:** Extraction of the organic acid through salt formation.

|  |  |
| --- | --- |
| **Procedural Step** | **Observations** |
| Record Unknown Code: |  |
|  |  |
|  |  |
|  |  |
|  |  |
|  |  |

**Part B:** Extraction of the organic base through salt formation.

|  |  |
| --- | --- |
| **Procedural Step** | **Observations** |
|  |  |
|  |  |
|  |  |
|  |  |
|  |  |
|  |  |

**Part C:** Recovery of the organic acid from its salt.

|  |  |
| --- | --- |
| **Procedural Step** | **Observations** |
|  |  |
|  |  |
|  |  |
|  |  |
|  |  |
|  |  |

Provide sample calculation of volume of 12 M HCl to add:

**Part D:** Recovery of the organic base from its salt.

|  |  |
| --- | --- |
| **Procedural Step** | **Observations** |
|  |  |
|  |  |
|  |  |
|  |  |
|  |  |
|  |  |

Provide sample calculation of volume of 6 M NaOH to add:

**Yield and Characterization of Unknown #\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_**

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
|  | Yield  (g) | Appearance of Crystals | Melting Point  (° C) | Tentative  Identification  of Unknown | Melting Point of Known\*  (° C) | Mixed Melting Point  (° C) |
| Organic Acid |  |  |  |  |  |  |
| Organic Base |  |  |  |  |  |  |
| Neutral Compound |  |  |  |  |  |  |

\*Literature value. Provide reference.

Provide reaction equations with your identified unknowns.

**Reaction 1: Reaction of Organic acid with dilute sodium hydroxide:**

**Reaction 2: Reaction of Organic base with dilute hydrochloric acid:**

**Reaction 3: Reaction of the salt of the organic acid with strong acid:**

**Reaction 4: Reaction of the salt of the organic base with strong base:**

Structure of Products

**Questions**

1. Why is the procedure used in this experiment called liquid-liquid extraction?
2. When extracting an organic compound from an aqueous solution into an organic solvent (e.g., diethyl ether), a chemist will sometimes add sodium chloride to the aqueous solution. What is the purpose of such an addition? What is the procedure called?
3. When an aqueous solution of an organic compound is shaken with an immiscible organic solvent, such as diethyl ether, the solute distributes itself between the two phases. When the two phases separate into two distinct layers, an equilibrium will have been established such that the ratio of the concentrations of the solute in each solvent defines a constant, K, called the distribution coefficient (or partition coefficient).

The distribution coefficient for compound X in the diethyl ether/water system is 3.0. If you were given a solution containing 8.0 g of X in 500 mL of water and wanted to extract compound X into diethyl ether, show that it would be more effective to extract X using three 50 mL aliquots of diethyl ether rather than a single 150 mL aliquot. (**Hint:** Determine how much of X would remain in the aqueous solution in each case.)

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# Experiment 6: Infrared Spectroscopy Tutorial

**Objectives**

The purpose of this experiment is to improve your skill in

1. identifying the functional group or groups present in a compound, given a list of the most prominent absorptions in the infrared spectrum and a table of characteristic absorption frequencies.
2. identifying the broad regions of the infrared spectrum to determine the presence of functional groups, such as alcohols, amines, and carbonyl groups, in an unknown compound.

**Write-up**

Fill in the following form below and answer the post-lab questions. Use the WORD version of the report form so you can add additional space for your answers. You will also need to download four (4) unknown spectra and include that in your report. When complete save as a PDF and email as an attachment to your Academic Expert for grading.

**CHEM 350 Experiment 6 Report Form**

**Infrared Spectroscopy Tutorial**

**Date:\_\_\_\_\_\_\_\_\_\_\_**

**Student Name:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ ID Number:\_\_\_\_\_\_\_\_\_\_\_**

**Infrared Knowns**

Fill in the following three (3) analyses tables to reflect your characterization of the spectra provided (above).

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **cyclohexanone** | Absorption  Band# | Wavenumber (cm-1) | Peak  Shape  (sharp, broad) | Peak Intensity  (strong, medium or weak) | Functional Group  Indicated |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

Functional Group(s) absent:

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **ethyl benzoate** | Absorption  Band# | Wavenumber (cm-1) | Peak  Shape  (sharp, broad) | Peak Intensity  (strong, medium or weak) | Functional Group  Indicated |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

Functional Group(s) absent:

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **benzonitrile** | Absorption  Band# | Wavenumber (cm-1) | Peak  Shape  (sharp, broad) | Peak Intensity  (strong, medium or weak) | Functional Group  Indicated |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

Functional Group(s) absent:

**Infrared Unknowns**

Select four (4) unknowns from the ‘Exp. 6 Infrared Unknown Downloads’ list provided online at:

<https://www.athabascau.ca/science-and-technology/resources/centre-for-science/labs/chemistry-labs.html#organicchemistry>

Download 4 of the possible 20 spectra (PDFs). Please neatly fill out the table on the unknown spectra and remember to fully label each of the absorption bands identified and identify the compound.

If you find the tables on the PDFs too small use this WORD template to give yourself more space to write/type.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Code:  Name: | Absorption  Band# | Wavenumber (cm-1) | Peak  Shape  (sharp, broad) | Peak Intensity  (strong, medium or weak) | Functional Group  Indicated |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

Functional Group absent:

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Code:  Name: | Absorption  Band# | Wavenumber (cm-1) | Peak  Shape  (sharp, broad) | Peak Intensity  (strong, medium or weak) | Functional Group  Indicated |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

Functional Group absent:

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Code:  Name: | Absorption  Band# | Wavenumber (cm-1) | Peak  Shape  (sharp, broad) | Peak Intensity  (strong, medium or weak) | Functional Group  Indicated |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

Functional Group absent:

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Code:  Name: | Absorption  Band# | Wavenumber (cm-1) | Peak  Shape  (sharp, broad) | Peak Intensity  (strong, medium or weak) | Functional Group  Indicated |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

Functional Group absent:

**Questions**

1. What are the major differences you would see in the infrared spectra of an alkane, alkene, and alkyne?
2. Consider the C=O absorption of three compounds: 2-butanone (1715 cm−1), propanoyl chloride (1772 cm−1), and propyl amide (1650 cm−1). Explain the observed differences.
3. Describe how IR spectroscopy might be used to monitor the progress of each of the following reactions.



`

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Signature: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Date: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

# Experiment 7: Extraction of Usnic Acid from Lichen

**Objectives**

The purpose of this experiment is to

1. isolate an enantiomer of usnic acid, a natural antibacterial organic, optically active compound with a very high specific rotation, found in a native species of lichen called ‘Old Man’s Beard’ (*Usnea* sp.). **Note:** Lichens are fungi/algae symbionts, where the fungus provides a physical support structure and micronutrients for the algal cells while the algal cells provide the fungus with nutrients derived from photosynthesis.
2. learn the technique of liquid solid extraction used in this experiment and the method of two-solvent recrystallization.
3. determine the specific rotation of the optically active product using a polarimeter, thereby exposing the student to the fundamentals of polarimetry.

**Write-up and Calculations**

Fill in the following form below and answer the post-lab questions. Use the WORD version of the report form so you can add additional space for your answers. When complete save as a PDF and email as an attachment to your Academic Expert for grading.

**CHEM 350 Experiment 7 Report Form**

**Extraction of Usnic Acid from Lichen**

**Date:\_\_\_\_\_\_\_\_\_\_\_**

**Student Name:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ ID Number:\_\_\_\_\_\_\_\_\_\_\_**

**Procedure:**

(Ref: )

Changes/Modification:

Fill in the flowchart with procedural details, data, and key observations.**SAMPLE EXPERIMENT 7 FLOW CHART**

REAGENT PROCEDURE / STEP OBSERVATION

**Reagent**

**Equip.**

**Prep.**

**Reaction**

**Product**

**Recovery**

**(Rxn.**

**Workup)**

**Prod.**

**Charact.**

Final appearance:

mp

mmp

etc.

Recrystallization

Evaporate Solvent by

Mix for :

Gravity Filter

Collected Filtrate

Discard

Lichen

\_\_\_ mL

Acetone

\_\_\_ g Lichen

**Part A-C. Usnic Acid Extraction from Lichen**

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
|  | Mass  Lichen  (g) | Product Yield  (g) | Appearance of Crystals | Melting Point  (°C) | Mixed Melting Point.  (°C) | Reference Melting Point  (°C) | % Lichen (w/w) |
| ( ) Usnic acid |  |  |  |  |  |  |  |

Show % Weight of Lichen Calculation:

**Part D-E. Results of Polarimetry Measurements for Unknown and Usnic Acid.**

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
|  | Mass  (g) | [Solution]  (g/mL) | Observed Rotation  ()\* | Corrected Observed Rotation  (blank) | **Specific Rotation\***  D | Reference Rotation  D20 | Optical Purity |
| Unknown  (L-tartaric acid) |  |  |  |  |  |  |  |
| ( ) Usnic acid |  |  |  |  |  |  |  |

\*At the temperature of solution during optical rotation determination:

Show specific rotation (D) and optical purity calculations for usnic acid:

**Questions**

1. What is a real life example of solid-liquid extraction?
2. Define the difference between diastereomers and enantiomers. Choose a specific example (e.g., glucose/fructose) to help explain your answer.
3. Draw the Fischer and line/wedge diagrams for the two enantiomers of usnic acid. Label the drawings with the structure’s absolute configuration.

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Signature: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Date: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

# Experiment 8: Preparation of Cyclohexene from Cyclohexanol

**Objectives**

The purpose of this experiment is to

1. prepare a pure sample of cyclohexene from cyclohexanol using an acid catalyzed dehydration reaction, and
2. acquire more experience with the techniques of simple distillation and liquid-liquid separations, and the use of drying agents.

**Write-up**

Fill in the following form below and answer the post-lab questions. Use the WORD version of the report form so you can add additional space for your answers. When complete save as a PDF and email as an attachment to your Academic Expert for grading.

**CHEM 350 Experiment 8 Report Form**

**Preparation of Cyclohexene from Cyclohexanol**

**Date:\_\_\_\_\_\_\_\_\_\_\_**

**Student Name:\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ ID Number:\_\_\_\_\_\_\_\_\_\_\_**

**Procedure:**

(Ref: )

Changes/Modification:

Fill in the flowchart with procedural details, data, and key observations.

**SAMPLE EXPERIMENT 8 FLOW CHART**

**REAGENT PROCEDURE / STEP OBSERVATION**

**Reagent**

**Equip.**

**Prep.**

**Reaction**

**Product**

**Recovery**

**(Rxn.**

**Workup)**

**Prod.**

**Charact.**

Simple Distillation

Apparatus

Clean rb flask, condensor, vac. adapter, etc.

Add boiling stones.

\_\_\_\_\_ g cyclohexanol

\_\_\_ mL

conc. phosphoric acid

Distil

Discard

Residue

Predrying

\_\_\_ g NaCl

\_\_\_ mL

\_\_\_ % Na2CO3

Neutralization

Dry Solvent

Gravity Filter or

Decant

\_\_\_ g

anhydr. CaCl2

Distillation

bp

density

atmospheric pressure

infrared spec.

**Properties of the Acid-Catalyzed Dehydration Product, Cyclohexene**

Calculations for theoretical yield, percent yield, and boiling point correction should be shown below the table. Note: \_\_\_\_\_\_\_\_\_\_\_ was the limiting reagent, since the only other reagent involved in the reaction, phosphoric acid, served as a catalyst.

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
|  | Mass  (g) | Appearance of Liquid | Boiling Pt.  (°C)  (/Pressure) | Theoretical Yield  (g) | % Yield |
| Cyclohexene |  |  |  |  |  |

Boiling Point Pressure Correction:

Theoretical Yield Calculation:

% Yield Calculation:

**Tabulation of Characteristic Infrared Absorptions for cyclohexanol and cyclohexene.[[2]](#footnote-2)**

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **cyclohexanol** | Peak# | Wavenumber (cm−1) | Peak  Shape  (sharp, broad) | Peak Intensity  (strong, medium or weak) | Functional Group  Indicated |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| **cyclohexene** | Peak# | Wavenumber (cm−1) | Peak  Shape  (sharp, broad) | Peak Intensity  (strong, medium or weak) | Functional Group  Indicated |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

**Questions**

1. What is the purpose of adding 10% sodium carbonate solution to the distillate in step 7 of the procedure?
2. Identify two possible by-products that could be formed from cyclohexanol in this experiment. (You may also want to search through your textbook to find what other reactions can occur between an alcohol and a concentrated mineral acid (e.g. phosphoric acid).
3. What evidence do you have of the purity of your cyclohexene product? Explain.
4. If you did a similar acid catalyzed dehydration of 4-methyl-2-pentanol you would have more than one product. Draw all the possible products of that reaction. [Hint: Remember intermediate hydride and alkyl shifts.]

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Signature: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_ Date: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

# Experiment 9: The Nitration of Acetanilide

**Objectives**

The purpose of this experiment is to provide the student with a practical example of an aromatic electrophilic substitution reaction, and to illustrate how the two isomeric products can be separated through recrystallization using an appropriate solvent. An introduction to the practical aspects of infrared spectroscopy is provided when the student obtains and compares the infrared spectrum of the reactant, acetanilide, and the product,

4-nitroacetanilide.

**Write-up**

A formal report is required for Experiment 9. Use the formal report template (WORD) in the Report Book and follow the instruction outlined in “Writing Laboratory Reports.” When complete save as a PDF and email as an attachment to your Academic Expert for grading.

**Questions**

Answers to be submitted with your lab report.

1. During the nitration of acetanilide care is taken to keep the reaction mixture cool. What do you think might be the consequences of allowing the reaction mixture to become too warm?
2. What organic compound (other than ethanol) would you reasonably expect to isolate from the ethanol/water mixture that was used to recrystallize your 4-nitroacetanilide?
3. Is there a way you could suggest to improve the experiment (should you do it again)?

1. ChemSketch is freely available at: https://www.acdlabs.com/resources/free-chemistry-software-apps/chemsketch-freeware/ [↑](#footnote-ref-1)
2. Include a copy of your IR spectrum. [↑](#footnote-ref-2)