# **Writing CHEM350 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 the mab manual.
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 the 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 the Report Book (see above for url) 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 it 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 the Report Book (see above for url).

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 the 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 must 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 TemplateOrganic 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.



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



***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.

## 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/mLmelting point:57.2 °Cboiling 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/mLmelting point: 103 °C – 106 °Cboiling point: approximately 300 °C | Causes serious eye and skin irritation. |
| isopropanol | 20 mL | 60.094 | -- | -- | density: 0.786 g/mLmelting point: - 89 °Cboiling 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/mLmelting point: -97.6 °Cboiling 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 °Cboiling 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**



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

**Nuclear Magnetic Spectroscopy (NMR) Spectra**



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



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

1. ChemSketch is freely available at: https://www.acdlabs.com/resources/free-chemistry-software-apps/chemsketch-freeware/ [↑](#footnote-ref-1)