CHEMISTRY 351

LABORATORY MANUAL

FALL 2024

FALL LABORATORY SESSIONS
START SEPT 9th, 2024

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DEPARTMENT OF CHEMISTRY

UNIVERSITY OF CALGARY

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WHY ONLINE PDF ?

The CHEM 351 student laboratory manual is available as a series of linked PDF documents. We certainly don't expect you to print all the pages, some can just be read online (*e.g.* the background information), but **you will need** paper copies of the pages that describe the **experimental techniques** and the **individual experimental procedures** during the laboratory sessions (no devices are allowed at the benchtop).

There are several sections to the manual:

- **Introduction** – that's this large document which outlines the laboratory part of Chem 351 including information on how to prepare for each experiment, keeping notes as well as grading and report writing issues.
- **Experiments** - each experiment has *at least two parts* to it:
  - **background** information on the basis and context of the experiment.
  - **experimental procedure -** describes the work to be carried out.
  - **report templates -** are provided for *some* experiments where you "fill in the blanks" to complete your report.
- **Techniques** – these provide stand-alone descriptions of how to perform the various experimental techniques that are integral to each of the experiments. In some cases, you will use a particular technique several times across the semester (e.g. melting point determination).

NOTE : For safety reasons, no electronic devices (other than a simple calculator) are allowed on the laboratory benchtop work area.

1. **<u>OUTLINE</u>**

Quick link to Chem 351 experimental schedule.

CHEM 351 is the first half course of an introduction to modern organic chemistry. Since organic chemistry is a practically based subject, the laboratory component forms an integral part of the course as it introduces basic laboratory techniques while also investigating some of the modern concepts of structure, reactivity and reaction mechanisms of organic molecules in both simple systems and in the often more complex biological context. Special attention has been paid to the selection of experiments in an attempt to clearly present and simplify some of the concepts of organic chemistry that students find difficult, to illustrate the role of organic chemicals in our everyday lives all while learning fundamental laboratory techniques. We hope that one of the outcomes from the laboratory component of the course it that you learn more about the safe handling and management of chemicals which can help you in everyday life, including at home where most families have chemical products that they tend to treat in a far too casual fashion. We know and understand that many of you don't want to be chemists, and you are required to take this course for your program or career aspirations because it was decided by others (not us) that the course is required. One of the reasons for this is that it is believed that the organic laboratory is a place where key tactile skills are developed.

For the experiments, please note that all the experiments **are known to work when carried out correctly and with good technique**. In general, they have been tried and tested over many, many years, so if the experiment doesn't work, it is most likely due to experimenter error **but that's OK** *as we all learn from our mistakes, and over time, hands on laboratory skills should improve which is one of our goals.*

Please note that the style of this manual, it is written in a scientific style and NOT as a "point form". You are taking a **University** science course and the procedures are written as you would find them if you were to go to a scientific journal or a standard laboratory textbook. A "point form" style generally discourages students from thinking for themselves, something that is both very dangerous in a laboratory, very boring for the student and doesn't promote skill acquisition.

2. LABORATORY COORDINATOR

If you have questions, concerns or issues with the laboratory component of the course, then you should contact the Laboratory Coordinator. While the laboratory TAs can help with questions about the in laboratory activities including experimental work and the reports, it is the Laboratory Coordinator who is responsible for decisions about the laboratory in general such as safety concerns *etc*. and it is your individual responsibility to communicate with the Laboratory Coordinator in a timely manner. **Questions concerning the CHEM 351 laboratories in the Fall 2024 semester should be directed to the Laboratory Coordinator, Dr. Ian Hunt mailto:irhunt@ucalgary.ca.**

3. ACADEMIC INTEGRITY

We expect and require all students to conduct themselves in an appropriate academic and ethical manner in the laboratory and when completing laboratory related work such as writing reports, quizzes or tests *etc*. Therefore, we have a **zero** tolerance for "cheating" on reports and quizzes *etc*. This includes but is not limited to copying reports, submitting reports that are not your own work *etc.,* This also means we expect students to be committed to diligently working on completing the practical work themselves.

We also have a **zero** tolerance for any attempts to interfere with the grading or the grades assigned as any part of the laboratory component. In the same manner as at an airport, where it says don't even joke about having a gun or a bomb, *please don't even think about joking with your TA about changing your grade in exchange for you buying them a beer or a cooler*, *etc*. We regard all such matters very seriously and they could result in an "F" in the course or worse.

4. ATTENDANCE AT THE LABORATORY

Students are required to attend the scheduled weekly laboratories and we expect this to be your registered laboratory section. This means that we expect you to be committed to attending your registered laboratory section.

Students are expected to be punctual for their laboratory sessions. **Students who arrive late** for a laboratory session will not be allowed to start the experiment and will be required to request a missed laboratory make-up session in accordance with the information below.

The laboratory sessions will close promptly at the end of the laboratory period since in many cases, the next section starts in just 30 minutes. This means that you will only be allowed 10 minutes "overtime" to complete laboratory tidy up.

5. MISSED LABORATORY SESSIONS

As stated on the course outline, students **are expected** to notify the laboratory coordinator (Dr Hunt, irhunt@ucalgary.ca) **ideally within 48 hrs.** of missing a laboratory section or a deadline for laboratory work. If you know you are going to miss a laboratory session ahead of time, then it is in your best interests to report the absence **5-10 business days** before the session takes place, ideally before the start of that week of laboratory activities. Please note that making the request too far in advance is not advised as we process them one week at a time.

In order to request a make up a missed laboratory section, you should complete the online "**Missed Laboratory Work**" survey.

Based on the information you provide in the form, the laboratory coordinator will determine if the reason is valid or not. Students with valid reasons (*e.g.* legitimate reasons that are appropriately reported in a timely manner to the Laboratory Coordinator) are normally **required** to make up the laboratory sessions. Valid absences can **only** be made up during the week of the corresponding experiment, ***and by prior arrangement*** with the Laboratory Coordinator. Absences can't be made up after the experiment has completed for the week. Due to resource limitations, each student is typically only allowed **one laboratory make up opportunity per semester** (except where *prearranged* special permission from the laboratory coordinator has been granted).

Students *without* valid reasons are expected to perform the experiment during their scheduled laboratory time and those that don't will get a zero for the experiment (*i.e*. it is an "unexcused" absence) as per UofC Calendar regulation G.2.3.a If you don't perform the actual experiment, then you can't submit any of the laboratory components that are based on that laboratory work for grading (*e.g.* quiz, notebook, report). If a student is granted an excused absence for an experiment, then they are automatically exempt from all components of that experiment.

The Chem 351/353 laboratory activities are based a series of experiments that gradually introduce fundamental laboratory techniques that will likely be used again in future experiments. Therefore, missing laboratory work and hence the opportunity to acquire the skill, may make it more challenging to complete some of the later experiments. This is why we expect students to attend their registered laboratory sessions and why we facilitate some make up laboratory sessions opportunities.

The ability to offer the laboratory make up process during F24/W25 depends on factors such as:

- The numbers of student and/or TA absences in a given week
- The nature of the experiment for that week
- The availability of space in laboratory sections that are running in each time slot

We have a finite weekly capacity and resources to run the make-up process. Therefore, if it becomes impractical to continue to run the laboratory make up process, the make-up process will be suspended. If this happens, then students who miss laboratory work for valid reasons will be awarded an "excused absence". The implications of the excused absence are described on the current Chem 351 course outline. Note that because laboratory work and laboratory principles can be tested on examinations, you need to make sure you familiarize yourself with the details of any missed experimental work. It may also mean that in the future you need to "fill in the gaps" !

6. <u>GRADING FOR THE LABORATORY</u>

As per the course information sheet, the Laboratory component will account for 20% of the course mark of CHEM 351. The laboratory course grade will be based on the grades accumulated from each of the following laboratory components in the manner stated below:

Each "report" (*i.e*. the "primary graded activity" for each experiment) = 10 points
Each quiz (pre-lab quiz) = 2.5 points
Preparation (pre-lab summary) = 2.5 points
Laboratory notebooks = 2.5 points

*The letter grade for each conventional written report is converted to a mark out of 10 based on the scale shown in the table below.

At the end of the semester, the total number of points obtained out of the maximum number of points possible for the specific semester will be converted to the corresponding grade out of 20 as the laboratory component of the course grade.

Please note that at the end of the semester, the typical laboratory average grade when all parts of the laboratory are considered is about 80%. Despite this, we often hear of students complaining about tough laboratory grading. **Sorry, but an 80% average laboratory grade simply doesn't match that sentiment !** Maybe you need to shift your expectations to match ours and with being in the 2nd year of a university program ?

Each laboratory activity will have as "primary graded activity" (typically completed after the laboratory session). It could be a short "template" report, a formal report, or it might be a set of questions to be completed in Moodle. If you are in any doubt, make sure to confirm with your laboratory TA during your specific laboratory session.

The laboratory course mark is based mainly on your laboratory reports (*i.e.* the primary graded activity, see above) but with contributions from your preparation work, your notebook mark and the pre-lab quizzes. **It is important to think about the laboratory work and put it into context in terms of the big picture, that is, the course as a whole. Look for the relationships between the practical work and the other course components (*e.g.* the lecture materials).**

A student with PERFECT laboratory and CAL marks would have a maximum of 40 course marks. That is not enough *on its own* to get a C- (50%)! It is important that students do well on examinations in order to get good overall grades in Chem 351. The examinations are the part of the course where we find out what an individual is capable of and it is the examinations, in general, and especially the key performance indicator - *the cumulative FINAL examination* - that have the most significant influence on the course letter grade. Less than 1 student in 5000 is likely to fail the course because they fail the laboratory, but maybe 1 in 5 will fail due to poor examination performance.

IMPLICATIONS…

So, for example, if you spend 5 hrs. working on a laboratory report worth about 1.5 course marks, then you should be spending about (35/1.5) x 5 hrs. = 116.7 hrs. preparing for the FINAL!
What does this mean?

PUT THE MOST EFFORT WHERE IT IS MOST CRITICAL!

MOST STUDENTS...

- **SPEND TOO MUCH TIME ON THE LABORATORY REPORTS AT THE EXPENSE OF EXAMINATION PERFORMANCE.**
- **ARE MORE ANXIOUS ABOUT LABORATORY MARKS THAN THEY ARE ABOUT MAKING SURE THEY CAN DO WELL ON THE EXAMINATIONS.**

Also, note that if you know the course content and materials well, you will be able to write good laboratory reports with minimal effort *and* do well in examinations. Students with good examination scores typically do well enough on reports but the opposite is not necessarily true.

In order to obtain a prerequisite pass in the course (*i.e.* C- or better), a **minimum** passing mark in the Laboratory (50%) is **required**. Students are also required to **complete a minimum of 5 (*i.e.* at least 60%) of the** experiments (*i.e.* attend the in-person laboratory session, perform the experiment, and submit the associated "primary" graded work *e.g.* report). If a student misses the laboratory session, and then submits the "primary" graded work, that work will not be graded. Students who fail to complete the minimum number of experiments will be judged to have failed the laboratory component and therefore will be ineligible for a prerequisite pass in Chemistry 351. **Therefore, any student who has concerns about satisfying this requirement should talk to the laboratory coordinator as soon as possible, but NO LATER FRIDAY NOVEMBER 1st, 2024.**

Laboratory reports will be graded using the letter grade scale shown in the table below (for reference see University Calendar). Note that these letter grades are the **only** possible grades available for reports. If a report gets BC it means it is slightly above the "C" (average), but not good enough for a "B".

"Calendar" description	Letter grade	Marks earned
Excellent performance – superior quality	A	10
	A/B	9
Good performance - clearly above average	B	8
	B/C	7
Satisfactory performance - basic understanding	C	6
	C/D	5
Marginal Pass – insufficient, lacking some key elements	D	4
Failure – very poor, lacking many key elements	F	2
Does not hand in report or does not perform experiment	--	0

The grade of the reports will be assigned based on the overall QUALITY of whole report: your work and results, scientific (*e.g.* chemical) and technical content of the report and the answers to any questions posed. The style, presentation, language, spelling and grammar used in your report are also important and will be used to evaluate the reports. Having the "correct answers" alone in a report is *NOT* sufficient to guarantee an A.

It is ALWAYS best to think of grades as being gained and awarded for what has been done well and correctly rather than see grades as being deducted for errors. *After all, a blank piece of*

paper has no mistakes, but would not get an "A"! **Grades should be viewed as a reward system NOT a penalty system and better work deserves better grades, with good answers and excellent answers.**

FOLLOW THIS LINK FOR FURTHER INFORMATION

For example, **a student who just adequately covers the minimum expectations in a report in an acceptable style will get a "C" letter grade – it is an average report!** General expectations are described below and more specifically at the end of each experiment.

The **Laboratory notebook** is fundamentally important to all scientists, and you will be required to maintain one for this course and it will be graded for each in-person experiment. For more details see the section below on "Laboratory notebooks".

You should expect a **pre-laboratory quiz** for each experiment since quizzes help promote preparation. The quizzes will be completed in Moodle and need to be completed at least 30 mins before the start of your laboratory session. The quiz questions will be based on *any of the information in the laboratory manual materials* related to that experiment and / or the *references* cited in relation to the experiment to be performed that week and / or *lecture materials* that relate to the experimental work.

If you disagree with any of the marks you have been awarded, you may appeal within **TEN BUSINESS DAYS** of being notified about the mark or of the items return to the class (as per the University Calendar "Reappraisal of Graded Term Work"). Based on life experience AND academic experience, we strongly recommend that you reflect on the grade for at least 24hrs and review the feedback provided before taking any further action. Remember that the grade is assigned to the document as a whole which means not only does it depend on the content, but it also depends on the quality of the presentation too. So before you request a regrade, reread section 9 about laboratory reports AND remember to consider the "big picture" rather than individual "small items".

In the case of laboratory work, the first step of the "appeal" process is to your home laboratory TA who should be able to **explain** (justify) the assigned grade to you. Note that your home laboratory TA can't regrade the item or change the grade for the item. If after talking to your home TA you still have cause for concern, then you will need to contact the laboratory coordinator. **If you need to appeal to the laboratory coordinator, then you need to provide a detailed rationale that outlines where and for what reason an error is suspected (*i.e.* clearly stating the details of your concern) from your University of Calgary email (all to be completed within the 10 day business period). The laboratory coordinator will then review the request. If appropriate, then the item will be regraded**

by a senior TA and / or by the laboratory coordinator. The laboratory coordinator will then provide a response to your University of Calgary email address. No such appeal will be considered after the 10 business days have elapsed. As per the University Calendar, "the reappraisal of term work may cause the grade to be raised, lowered or remain the same". For laboratory reports, since the grade is assigned to the whole report, the whole report must be considered in the regrade.

Each TA is provided with a marking guide for each of the reports, grading is discussed regularly at the regular TA meetings, and the grading is rotated so that different TAs grade the reports. In terms of rotating graders, we don't do it because it is easy, we do it because it helps many facets of the grading process. Each of these actions helps to standardise the grading process by the end of the semester. However, due to the large number of laboratory sections and hence TAs for these courses, **the laboratory coordinator reserves the right to adjust (*i.e.* normalise) the marks for each section *if needed***. This is to ensure parity of marks for ***every student*** in the course, but it Is rarely required because of all the other actions that are in place.

7. PREPARING FOR THE LABORATORY

This is an area of concern as too many students are typically arriving at the laboratory SERIOUSLY under-prepared.

In order to be able to complete the experiments in the scheduled time slot available (2h 50 min) and in order to get the ***most*** from the experiments in CHEM 351, ***you*** will need (and the TAs and laboratory coordinator will expect) that you arrive at laboratory session ***effectively*** prepared. This means you should have read through AND understand the experiment background, the procedure and any required techniques (including watching any videos that may have been provided). You should have looked at the references, and maybe the additional laboratory information on the course homepage. This will allow you to prepare for each experiment so ***you*** know what ***you*** will be doing and will enable you to complete the required work. Remember that each of the steps in the experiments is there for a *chemical* reason, and not simply to occupy your time! Try to think about these reasons and the procedures will all make more sense, and the experiments and the science will be more fun!

PRE-LABORATORY QUIZ

For most experiments, there will be a short pre-lab quiz to be completed online in Moodle **before** you attend your laboratory session. If there is a pre-lab quiz, then it will be accessible from 06:00 the day before your laboratory session until 30 mins. *before* the start of your scheduled laboratory session, 5-10 time limit, see Moodle for the specific details for each quiz. details. This pre-lab quiz is based on the materials described above in the preparing for the laboratory section. Late quizzes are not accepted for grades. We recommend that you write your pre-lab summary *before* you complete the pre-lab quiz.

PRE-LABORATORY SUMMARY

In addition, we typically require a “pre-lab summary” to be written and the pdf file to be submitted to the appropriate D2L Dropbox prior to the laboratory session (check D2L for specific details). Summaries that are longer than about 100 words will not get full marks

PRE-LABORATORY SUMMARY: **IN NO MORE 4 or 5 sentences (about 100 words), identify the purpose of the experiment, how you will achieve that purpose, (*e.g.* what will you be looking for *etc.*) and how you will establish success.**

TAs will be monitoring student preparation and reporting it to the laboratory coordinator. If there are concerns, then the laboratory coordinator will meet with the student to review the situation. We reserve the right to suspend students who are under prepared from the laboratory.

While writing the pre-lab summary before the laboratory period will help you prepare for the experiment, ***it should also help you write your report***, since these questions relate to various parts of a formal report (*i.e.*, introduction, results and conclusions). This “pre-laboratory summary” will be evaluated and will form part of your laboratory mark (see above in section 6).

FOR YOUR OWN USE, you may also find it useful to write your own point form summary (or a plan) of what you are going to be doing - an example is provided (**see below**). Many students in the past have written out the whole procedure ahead of time – it is probably **not** the best use of your time. For in-person laboratory work, it is NORMALLY what you actually **DO IN THE LABORATORY** that is important and needs to be recorded in the notebook AS YOU PERFORM THE EXPERIMENT.

As indicated above, <u>before</u> each experiment you may wish to <u>prepare a point form plan</u> of what you will be doing based on the laboratory manual and other relevant materials an example is provided (**see below**). It is a good idea to list the properties of reagents, products, and solvents such as molecular weights, weights needed, molar quantities, melting points, boiling points, densities, and solubility properties beneath an equation that represents the preparative or kinetic experiment to be carried out. This information is easily expressed in table form and could be entered into the notebook before you come to the laboratory, so it serves as an easy reference for the laboratory operations and helps you to minimise chemical waste. The "plan" may also include a sketch of any important apparatus and may contain a stepwise analysis of the procedure to be carried out that can be referred to during the laboratory period. Think about what will work best for you. An example of a "plan" for a typical organic chemistry experiment is provided on the following page.

8. LABORATORY NOTEBOOKS

A good laboratory notebook is fundamental to good science in general, not just organic chemistry, so you are required to keep a notebook for CHEM 351. A paper copy of your notebook work for an experiment will need to be handed in for review and grading at the end of your laboratory session. We strongly recommend a self-duplicating laboratory notebook because in our experience over many years, this is the easiest and most efficient way to do this. Self-duplicating laboratory notebooks should be available from the University Bookstore. Feel free to use the notebook in other courses too (including Chem 353).

The primary use for your laboratory notebook is to record **what you do as you do it** and observations **as you make them**. Laboratory notebooks should be written in **ink**. Any mistakes should be neatly crossed out ("White-out" **must not** be used). The information in your notebook can then be used to form the basis of your experimental report (we expect it to be consistent).

The notes in your laboratory notebook should be detailed enough to allow another chemist of your level to pick up your laboratory notebook and then repeat the experiment as you did it **without** the aid of this manual. Each experiment should start a new page that is dated and has the title of the experiment. If reactions are involved, then show the balanced equations.

Once you are in the laboratory, the notebook should be used to record *all* your **actual measurements** (crude weights, purified weights, percentage yields, b.pts. m.pts. distillation temperatures *etc.*) and observations (colours of solutions, precipitates *etc.*) as you perform the experiment. This means you should have your notebook with you at the balance when weighing things, by your side when you are measuring results and in general as you do the experiment.

Make sure note any deviations from your point form summary and the procedure at stated in the laboratory manual. Separations / extractions are often easily described in simple flow-chart format. Difficulties encountered with laboratory operations and any accidental loss or contamination of product should be recorded.

IMPORTANT:

A duplicate paper copy of your notes **must be handed-in to be graded and your notebook signed by laboratory instructor before you leave the laboratory each week.** It is your responsibility to make sure the TA for the laboratory period you attend has these notes at the end of the laboratory period. Don't expect us to try to track you down for them!

Examples of a typical pages from a laboratory notebook are provided for reference.

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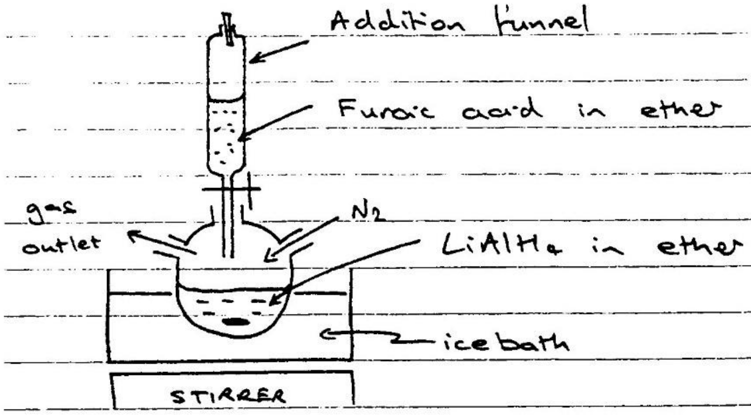
PLAN (SAMPLE)



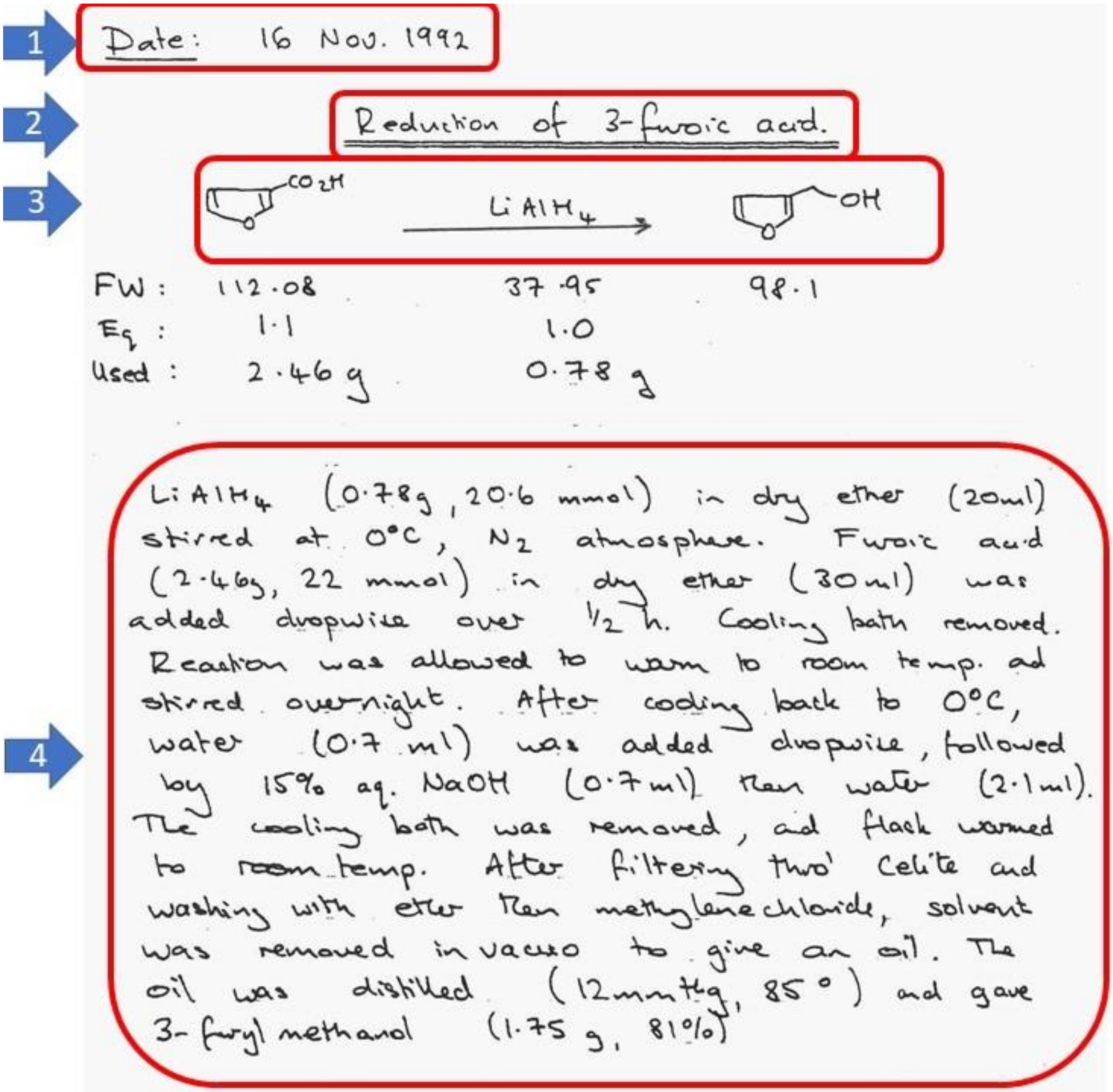
PROCEDURE

1. Dry glassware (100 ml, 3 neck RBF)
2. Set-up for N_2 atmos + addition
3. Weigh $LiAlH_4$ (CARE!)
   @ 20 mmol = 0.76 g → into flask
4. Add 20 ml DRY diethyl ether
5. Cool to 0° C.
6. Weigh acid
   @ 22 mmol = 2.47 g → funnel
7. 30 ml DRY diethyl ether → funnel
8. Add acid soln. slowly ~ 30 min.
9. Warm to RT, Rem overnight stir
10. WORK-UP
    0°C, add H_2O, NaOH, H_2O
    (.7 .7 2.1 ml)
11. Filter, remove solvent.

NOTE:

This page that is an example of what an organic chemist might write in their notebook as ***preparation*** for carrying out an experiment. This is optional, it will not be graded as part of the laboratory mark. It is simply to help you with preparation and give you a clear picture of the work you will need to do.

LABORATORY NOTES (SAMPLE)



1 Date: 16 Nov. 1992

2 Reduction of 3-furoic acid.

3 CO_2H $\xrightarrow{LiAlH_4}$ OH

FW :	112.08	37.95	98.1
Eq :	1.1	1.0	
Used :	2.46 g	0.78 g	

4 $LiAlH_4$ (0.78g, 20.6 mmol) in dry ether (20ml) stirred at 0°C, N_2 atmosphere. Furoic acid (2.46g, 22 mmol) in dry ether (30ml) was added dropwise over ½ h. Cooling bath removed. Reaction was allowed to warm to room temp. and stirred overnight. After cooling back to 0°C, water (0.7 ml) was added dropwise, followed by 15% aq. NaOH (0.7ml) then water (2.1ml). The cooling bath was removed, and flask warmed to room temp. After filtering thro' Celite and washing with ether then methylene chloride, solvent was removed in vacuo to give an oil. The oil was distilled (12mmHg, 85°) and gave 3-furyl methanol (1.75 g, 81%)

NOTE:

This page would be used to generate your **laboratory notebook mark**. This page reflects the work done during the laboratory period. It would allow someone to repeat your experiment.

Key components :

1. Date the work took place
2. Title of the experiment
3. Equation representing the reactions that were involved
4. Procedure describing the work done in sufficient detail to allow someone to repeat the work.

9. LABORATORY REPORTS

General Statement: We are more interested in **QUALITY** than quantity. More is **NOT** necessarily better! **In Science, it is important to be accurate, concise and to the point**. We will be building on report writing skills that you should have acquired in first year chemistry (which are the prerequisites for this course). This means we will be assuming that you already have some understanding of what goes into scientific laboratory reports. In turn, now that you are taking a 2nd year course, we will not need to be as specific in our report writing guidelines as to what is required (based on first year, you should already have a feeling for that). A key issue here is we want you to start to get used to making decisions for yourselves and understanding why / how *etc*. rather than just doing something because someone told you to do it. That being said, we need to strike a balance because many students spend too much time writing reports primarily because of poor background / content knowledge. Therefore, in terms of the "big picture" you should commit to improving your background knowledge (which is probably going to improve your examination grades too). In terms of **quality**, this means that the presentation of the work is important. All too often we see almost unreadable "scribble" or needlessly untidy figures, diagrams and documents. We expect you to show some pride in your work.

You need to treat your report a "professional" document, not a simple "memo" or a quick communication to a close friend or a high school piece of work.

As a rough guideline, once you are used to writing reports, we would expect that an average student should be able to complete a *reasonably good weekly report* (*i.e.* B grade) in an average of about 2 hours. If it is taking you longer, you may be writing too much, or you are not working effectively, and you should reconsider how much time you are putting into the reports. Note that in our experience, the main reasons students do not do as well as they would like on reports are:

- poor presentation and / or discussion of the scientific (especially chemical) issues pertinent to the experiment, likely a sign that your chemistry background knowledge is weaker than it should be, and may be that your organic chemistry knowledge is not at the level that is should be (*i.e.* you need to do more work maybe with a change of approach)
- incorrect writing style (*see guidelines below*)
- "claims" must be backed up with references, *e.g.* the statement "the boiling point of water is 100 C at sea level" should be supported with a reference (see below for acceptable reference styles).
- incorrect terminology (use the e-text, lecture notes, *etc*.) to make sure you are using the appropriate terms at the right times and in the correct manner.
- writing and / or work that is below a 2nd year University level (is it presented as a "professional document or does it feel like high school work or a note to a friend or coworker ?)
- not answering the required additional questions being asked

When writing reports:

- Think scientifically about what you are writing; analyse your results.
- Don't expect to get "excellent" (A) marks for "average work" (C)
- Read over your report one last time before you submit it

Generally, (*unless specifically stated otherwise e.g. check D2L News and laboratory related emails, do not base it on D2L Dropbox deadlines which might reflect the timings for the last laboratory sections of the week*), **laboratory reports are due by the start of your next scheduled laboratory period of your registered section** and that is *usually* **ONE WEEK AFTER** the experiment was carried out. **It is your responsibility to make sure that you have submitted your completed report as a pdf file to the correct D2L Dropbox by the appropriate deadline.**

Notes:

- **Most of these reports are to be completed individually: copying and plagiarism will NOT be tolerated and will be reported for appropriate disciplinary action. We strongly recommend that you consult the Academic Integrity Student Handbook. See page 4 for "Using sources ethically: avoiding plagiarism" and the next few sections for useful ideas and guidelines.**
- **It is your responsibility to ensure that your laboratory reports have been submitted to the appropriate D2L Dropbox by the start of your next scheduled laboratory session. Reports should be submitted as a SINGLE PDF document (make sure to make a print to PDF copy).**
- **The document needs to be submitted in a logical and organized format. Presentation, grammar and spelling are all important and can impact the grade assigned.**
- **It is your responsibility to check the document before submitting to ensure all parts are present, in order and can be read. If you submit document files that can't be read, then they will not be graded.**

Any report not received by the TA by the appropriate deadline will be judged as "**LATE**" and **WILL NOT BE GRADED BY THE TA**. **ALL** late reports submissions will need to be approved **by the Laboratory Coordinator of the CHEM 351 Laboratory Course** who will decide if there are acceptable extenuating circumstances.

There are two general formats for the CHEM 351 reports: on-line "template" reports ("fill-in-the-blank"), and the full reports (see below for more details).

Style guidelines

The laboratory report is a formal description of your account of the experiment. As such, it **must be written in a formal style and use proper English**. No slang or unacceptable abbreviations should be used ("lab" and "xs" are not allowed, but "*e.g.*" is). If you are in any doubt which abbreviations are acceptable, then you should consult a dictionary. You should use the past passive tense as this tends to be a more readable style for the next person (or yourself) reading your report. **The report should be written using sentences and paragraphs** and **NOT** in point form.

Consider each of the following examples, which one is the appropriate style ?

a. **1.** Place 0.78g of $LiAlH_4$ in 20ml of dry ether in a round bottom flask
2. Cool to 0 ^oC and stir under N_2 atmosphere
3. 2.46g of furoic acid in 30ml of dry ether
4. Furoic acid solution added dropwise over 0.5 hr.
5. Cooling bath removed
6. Reaction allowed to warm to room temp. and stirred overnight

b. Place 0.78g of $LiAlH_4$ in 20ml of dry ether in a round bottom flask. Cool to 0 ^oC and stir under N_2 atmosphere. Add a solution of 2.46g of furoic acid in 30ml of dry ether in a dropwise manner over 30 mins. Remove the cooling bath and allow to warm to room temp. and stir overnight.

c. I added 0.78g of $LiAlH_4$ to 20ml of dry ether in a round bottom flask. I cooled this to 0 ^oC and set up a N_2 atmosphere. I added a solution of 2.46g of furoic acid in 30ml of dry ether in a dropwise manner over a period of 30 mins. Once complete, I removed the cooling bath and allowed the reaction to warm to room temp. and stirred overnight.

d. In a round bottom flask under a N_2 atmosphere, $LiAlH_4$ (0.78g, 20.6 mmol) was combined with dry ether (20ml). The mixture was cooled to 0 ^oC. A solution of furoic acid (2.46 g, 22 mmol) in dry ether (30ml) was added dropwise over a period of 30 mins. The cooling bath was removed, and the reaction was allowed to warm to room temperature and then stirred overnight.

In the examples above, (a)-(c) each contain writing style errors:
(a) is in point form rather than paragraphs and has the wrong tense, and incorrect style for amounts
(b) has the wrong tense and has the incorrect style for amounts
(c) is written in the first person, is not the past passive and has incorrect style for amounts
(d) is the correct format and tense and represents the style that should be used.

Reports will need to be prepared and submitted electronically to the appropriate D2L Dropbox as **PDF documents**. Given that any report templates are provided as WORD docx, the easiest way to prepare your report would be to use WORD to complete the report rather than printing it out and then handwriting the document. The word processed document then **NEEDS TO BE** printed as a PDF (this "locks in" the structure and content and helps ensure the content is preserved in D2L). In terms of filenames, note that D2L does not allow "special" characters in filenames (*e.g.* / & : ; ? " # * etc.…). If one uses these characters, then the file upload will fail (and D2Ls error reporting isn't exactly useful for troubleshooting). D2L will also not allow you to upload a file with same name as an existing file. When uploading files, it is best to use only alpha-numeric characters, underscores and dashes in the names. We recommend using a simple format such as:
351Expt1_report_YourName_BXX.pdf

So, for example, if your name is "Mickey Mouse" and you are in laboratory section 1, the report for experiment 1 would be:

351Expt1_report_MMouse_B01.pdf

FYI : The experiment numbers are listed in the experiment schedule that is posted on the course website. Having a good file naming system that is logically organized is likely an important skill and it will likely help ensure that you submit the right file to the right course. Naming your files in a logical and systematic way is likely a useful and transferable skill.

IT IS YOUR RESPONSIBILITY to ensure that the correct "final" document is submitted to the correct D2L Dropbox, before the deadline and that the submitted documents are of a suitable standard (*i.e.* the document can be read, it is complete *etc.*). Note that as soon as you submit a document to a D2L Dropbox, D2L sends you an email receipt that indicates the name of the file that was submitted and to which specific D2L Dropbox it was submitted. We expect **YOU** to check that email and correct any errors you have made immediately (note that changes can not normally be accepted after the submission deadline). Any documents that are accepted would be subject to the standard course late penalty policies.

Documents that are not submitted to the correct D2L Dropbox by the appropriate deadline will NOT be graded.

Documents should be written or printed in **blue or black ink**, **line spaced at 1.5, using Arial font size 12**. These settings help with the expected professional presentation and help ensure that the submitted

PDF documents are legible in D2L. Many students try to cram in more material than is needed (don't). There is enough space on the template pages to write what is needed.

Do not change the format and/or length of the template to create more space and don't simply add more! We will NOT grade the "extra" beyond the given page limit.
If any part of a report is illegible, needlessly untidy, disorganised, uses poor English spelling or grammar or is in the wrong style then the report grade will be affected.

All tables, figures, graphs diagrams *etc*. should be given a title. Drawn graphs, figures and diagrams need to be neat and tidy (we see a lot of poor quality images or diagrams that could easily be better). Poor quality "unprofessional" images / documents impact the overall quality of the report and, therefore, the grade. There are several ways to include good quality diagrams:

1. use software (such as ChemDraw) or an app to create the image to insert into your document, then convert to pdf
2. hand draw the image (carefully) using a tablet device to insert into your document, then convert to pdf
3. print out the report, then hand draw (carefully) the image on the paper, scan document to pdf (*e.g.* using copiers on campus such as in the library)

Since the UofC Chemistry department has a lisence, ChemDraw is available for free download. You will need to register on the subscription website: https://informatics.perkinelmer.com/sitesubscription/. Find the University of Calgary in the list (painful… alphabetically or by typing in the search bar) and click on the University of Calgary's link to be taken to the account registration page where you identify yourself as a member of the institution. The Account ID field in the form will auto-populate with your Account ID. Individual users will receive a welcome email with URL's and log in instructions.

Key message: you don't *need* a tablet device to create your reports !

We see a lot of reports that have lots of words but actually say very little (*i.e.* they are verbose and or repetitive). This is not a feature of a good report! As examples:

1. if you include a reaction scheme, then there is likely no need to describe that reaction verbally
2. there is typically no need to repeat the same sentences multiple times in different parts of the report.

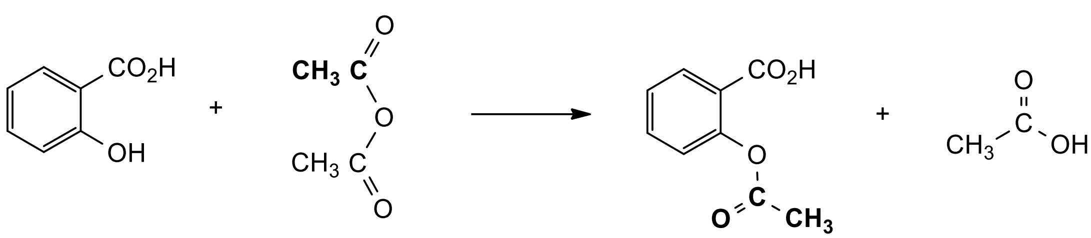
The **quality** of what you write AND the document as a whole is **very important** - in the future it may help you get a job - for example, a Calgary company once estimated that 25% of job applications go straight into the garbage because of the quality of the writing in the resume or the covering letter.

A general format for a full laboratory report would typically include:

a) A brief descriptive **__title__** and the **__date__** the experiment was carried out and the full names of any **__co-workers__**. You should also state your TAs full name (*e.g.* Mickey Mouse) and your laboratory section (*e.g*. B01).

b) **__Introduction__** (*the purpose and context of the experiment*).

The introduction briefly describes the *__purpose__* and *__context__* of the experiment (do __not__ simply restate the title). In most cases for Chem 351 and 353, this will be no more than a few lines. An introduction should not contain any results! The introduction should also include balanced reaction **__equation(s)__** if appropriate; these will accurately summarise the chemical reactions (*e.g.* using **drawn** structures rather than just "$C_7H_6O_3$" *etc.*) and show the basis for stoichiometric calculations. An example of how it should be done from the synthesis of aspirin experiment is shown below. If you take an equation from another source, it should be referenced, if you create it yourself from scratch, then it doesn't. The introduction provides some background to the work, the objectives and goals for the experiment.



c) **__Procedure__** (*what did you do and how it was done*).

A detailed description ***in your own words (and in the appropriate style, see above)*** of the experiment as you carried it out and that is sufficient in detail to *__allow another chemist to repeat your experiment__*. Be sure to describe, in sufficient detail, any departures from the procedure in the manual *e.g*. time and temperature of reaction, spillages, or loss through leakage from a distillation assembly *etc*.

d) **__Results and Discussion__** (*what did you find out and what does that mean?*)

Present your experimental results in a suitable format, such as tables and diagrams where appropriate and effective. Where appropriate, report the amount of product obtained both in grams and as a percentage of the theoretically expected yield. Record any physical properties you determine or observe and compare them with literature values (these values should be referenced - see below). If you measure a physical property more than once you should record __all__ the individual values determined (*e.g.* for melting points). Show **all** your calculations with appropriate significant figures. Include curly arrow mechanisms where appropriate (note: *all mechanisms covered in the laboratory work can be found in the etext*). **Discuss your results with a scientific (chemical) basis**. For chemistry, that probably means explaining things at the molecular level - what's happening and why. Here are some general suggestions that might be applicable (they will not apply to all of the experiments all of the time). Did you achieve the purpose of the experiment? How do you know that? To what extent? How do all of your results support this (relate to course content where possible)? Are there any discrepancies? Describe any sources of loss or error *etc*. that have contributed to your experimental results. Describe any difficulties you encountered or mistakes you made. Assess your performance in carrying out the experimental operations. **Most students write far too much about sources of errors** - essentially making things up (remember that the experiments are known to work as presented in the manual and therefore you don't need to recommend alternate procedures). Students then write too little on the discussion of the real science and the conclusions. The most likely sources of error will be because of what *you* have or haven't done rather than a problem with the experiment itself.

e) **<u>Conclusion</u>** (*what has been learnt?*)

Scientifically assess your experiment and your results in general. Highlight any deductions (answers) you have established. The conclusion should be a <u>*summary*</u> of how things turned out and will probably refer back to points in your introduction and discussion.

f) **<u>References</u>**. (*what sources were used?*)

Any claim you make must be supported by a reference indicating the source of your information. If you use the words, images, figures or ideas of others, then those sources need to be cited (otherwise you could be plagarising their work and/or ideas). The references you cite should be in the public domain and available at no charge.

Note Chemical Supplier catalogues (*e.g.* Sigma-Aldrich) are **NOT appropriate references** for data as they are usually the values for their products rather than the pure materials. In addition, the citation of fee-charging "course work" websites or documents is not acceptable.

There are many ways to present references, but two common formats we recommend are illustrated below:

EITHER ACS style using numerical references in-text, with a numbered reference list at the end in the order in which they were cited (see example excerpts below)

Excerpt from text could read:

"Acetaminophen was first synthesised by Morse[1] from <u>p</u>-nitrophenol by reduction with tin in glacial acetic acid. Acetaminophen is one of several analgesics that have been produced using organic synthesis since the late 1800s.[2]"

Excerpt from accompanying reference list would look like:

References

1. H. N. Morse, *Chem. Ber.*, **11**, 232 (1878).
2. H. Haas, *Am. J. Med.*, **75**, 1 (1983).

OR CSE style using name-year references in-text, with an alphabetical reference list at the end. (see example excerpts below)

Excerpt from text could read:

"Acetaminophen was first synthesised by Morse (1878) from p-nitrophenol by reduction with tin in glacial acetic acid. Acetaminophen is one of several analgesics that have been produced using organic synthesis since the late 1800s (Haas 1983)."

Excerpt from accompanying reference list would look like:

References

Haas H. 1983. History of antipyretic analgesic therapy. The American Journal of Medicine. 75(5): 1-3.

Morse HN. 1878. Ueber eine neue darstellungsmethode der acetylamidophenole. Berichte der Deutschen Chemischen Gesellschaft. 11(1): 232-233.

The American Chemical Society (ACS) style is commonly used by chemists and biochemists, and the Council of Science Editors (CSE) style is more commonly used by biologists. **It does not matter which one you choose if you are consistent.** Note that each reference should only appear in the reference list once and if it is cited more than once, the same reference number must be used.

For book citations, cite the author, book title, edition, publisher, place of publication and year of publication, *e.g.* Carey, Organic Chemistry, 4th edition, McGraw-Hill, New York, 2000, pp 800-850.

If you need to cite information from the WWW (including the Chem 351 or 353 laboratory manual documents), you should give the title of the page, the http address (the URL), and the date it was accessed, for example:

Organic Chemistry 351 Homepage, https://www.chem.ucalgary.ca/courses/351/index351.html, (July 16, 2019).

Chemistry 351 Laboratory Manual Fall 2024,
https://www.chem.ucalgary.ca/courses/353/laboratory/351outline-F24.pdf, (Sept 2, 2024)

g) **Questions.** Some experiments have extra questions included in the experimental / report documents for you to answer in the report - these are usually there to help you recognise some of the science that you need to be thinking about, *i.e.* they are really hints.

Note: We often get asked to provide sample reports but we don't. Why? Well, first it's difficult for a student to get anything out of a report about an experiment that they have not performed or written up themselves. In addition, the nature or the focus of each of the experiments are different and therefore so are the reports to some extent. Some experiments are focused on learning a technique, others are about reactivity while others are about the synthesis of a compound.

10. SAFETY AND WASTE MANAGEMENT

Safety should be of the utmost concern in every chemical laboratory, and every place where chemicals are used. The chemicals used in the laboratory may pose a variety of hazards. For students to be familiar with the safety issues related to working with chemicals in a chemistry laboratory, ALL STUDENTS are required to complete (if they have not already done so) the University of Calgary, Department of Chemistry / Safety Office course "Chemistry Laboratory Safety", read the information provided here, and completed and passed the on-line safety assessment. If you have not previously completed the Department of Chemistry/Safety Office course AT THE UNIVERSITY OF CALGARY (within the last 5 years), YOU ARE **REQUIRED** TO DO SO BEFORE YOUR FIRST LABORATORY SESSION. This is federal, legal requirement. The Department of Chemistry/Safety Office course is run online, see below.

The Chemistry Online Safety Course is a mandatory component for all students taking chemistry laboratory work. The safety course takes about 50 minutes to complete online (no preparation required).

- Log on to https://my.ucalgary.ca in a separate tab before trying to access the course.
- To enroll in the course, click on this link.
- To return to the course after enrolling, click on this link, then click the course title.

Log in using your my.ucalgary.ca credentials and click on the *Enroll* button at the bottom of the screen to join the class. Use the *Launch* links to open each section of the course. You must complete each section and pass each quiz (minimum 80% score) before you gain access to the next section. **Enable pop-ups in your browser** or you may not be able to see some of the content.

You are required to complete this course before your first laboratory session. **This is a federal requirement.**

Keep in mind that you cannot do any laboratory work without having done this Safety course. If you have completed this Online Safety Course at the University of Calgary within the last 5 years, you do not have to re-do the course. We monitor student compliance with this important requirement, but you may have to show your Completion Certificate to your TA or laboratory coordinator as proof.

Troubleshooting

- **Problem**: On login, you receive an "Invalid Learner ID" error.
  Solution: email Dr. Simon Trudel (trudels@ucalgary.ca) to have your access reset.
- **Problem**: After clicking "Launch" for a video, nothing happens.
  Solution: You need to allow pop-up windows from your browser. (Google e.g., "disable popup Firefox" to solve this).

- **Problem**: When trying to return to the course, you receive a "duplicate enrollment" error.
  Solution: Use the second course link above; or visit https://learning.my.ucalgary.ca/, and find the Chemistry Safety Course listed under "My Learning".
- **Problem**: After logging in, you get an "Error Getting Content" warning.
  Solution: Log into my.ucalgary.ca before trying to access the course.
- **Other problem?** email Dr. Simon Trudel (trudels@ucalgary.ca) with as much information as you can provide about the error.

Please be aware that if you have not passed the course, you will not be allowed to participate in the laboratory experiments and will be required to leave the laboratory and will be suspended from laboratory work until you have completed the course. You will receive a zero for all experimental work missed as a result of the suspension.

If you have done the Online Safety Training within the past 5 years at University of Calgary, you do not need to re-take the course. If your last training was over 5 years ago, you need to take it again.

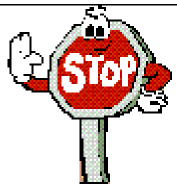
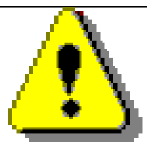
a) Regulations

Summary safety poster

The following regulations must be rigorously adhered to. Failure to follow the guidelines below and those provided for each experiment could lead to you be asked to leave the laboratory. In more serious cases, this could result in you being suspended from the laboratory sessions as your actions could be unsafe to other users of the laboratory environment:

- Students are **not** allowed in the laboratory unless an instructor is present.
- Students entering the laboratory must be compliant with current UofC health requirements.
- Safety glasses with side shields **must be worn at all times** when in the laboratory.
- Laboratory coats **must be worn at all times** when in the laboratory.
- Shorts and sandals are **prohibited** in the laboratory.
- Food, beverages and smoking are **prohibited** in the laboratory.
- The University of Calgary Chemistry Laboratory safety course must have been completed and passed (see D2L Laboratory content folder for the details) before you can participate any laboratory activity.
- The safety training record form (available from your TA) **must** be completed, signed, and turned into your laboratory instructor **before** beginning any laboratory work during the first experiment of the semester.
- **Never** pipette by mouth.
- **All unauthorized experiments are prohibited**.

- Experiments in progress **must not** be left unattended.
- **No** overnight experiments are allowed.
- Noxious or odorous chemicals are to be handled only in a fume hood. Generally, if you find a chemical in the fume hood, use it in the fume hood.
- If you have a small chemical spill or leave a "bottle ring", clean it up **immediately and dispose of the contaminated materials safely it the appropriate waste container**. For larger spills, talk to your TA. If you find a spill, report it to your TA.
- **Pay attention** to ***any hazards and warnings*** given in each experiment.

	If you see this symbol, you **must** talk to your TA before you continue. It means that there are significant hazards and that your TA needs to check before you continue.
	If you see this symbol, you should proceed with extra caution after checking your safety precautions. Check with your TA if in any doubt.

- **Never** handle or pour flammable liquids near an open flame or other ignition source.
- All accidents must be reported **immediately** to the laboratory TA and the appropriate accident reporting procedures followed as required, via the online accident reporting system, OARS.
- At the end of each laboratory, **you are required to ensure your personal work area is clean and tidy**.
- MSDS sheets are good sources of information concerning the hazards of a particular chemical and are available for all chemicals used in the laboratory. The MSDS sheets are available online in the laboratory at the TAs desk; they are also available online if you want to check prior to coming to the laboratory.
- To avoid the possibility of chemical spills, you should keep your work area clean and free of clutter. In the event of accidental spillage, inform your laboratory instructor immediately, and then clean up immediately. For larger spills, a spill kit is available from the laboratory technician.
- In the event of accidental skin contact, ask a fellow student to inform your laboratory TA while you immediately start to wash the area with copious amounts of cold water (*i.e.* under a continuous stream of cold water from the tap).
- It is generally good practice to wash your hands frequently when working in any chemical laboratory.
- In addition, you should learn the locations of the nearest telephone, fire extinguisher, eye wash station, emergency shower, and first aid kit, as well as exit routes from the laboratory and the building.

- **In case of a serious emergency, the primary respondent should use the laboratory telephone to dial 911 to report the incident and secure the appropriate emergency service. Once that call is complete dial 220-5333 to report to campus security then contact a laboratory technician.**
- At the end of the laboratory period, make sure that the fume hood, benchtop work area and sink area are clean and tidy. Equipment should be put away and all chemicals stored in a safe manner.
- Wash your hands before you leave the laboratory.

b) <u>Workplace Hazardous Materials Information System (WHMIS)</u>

WHMIS provides that, by federal law, students are entitled to information concerning any materials used in the laboratory. This information is available on a Materials Safety Data Sheet (MSDS). If you need access, then talk to your laboratory TA.

c) <u>Safe Laboratory Practice</u>

i) <u>Fire</u>

Know the location of the safety shower, the fire blanket and the fire extinguishers.

If the vapours from a flask ignite, a small fire can be extinguished by turning off the burner and gently placing a notebook or wire gauze over the top of the vessel containing the burning solvent. Under no circumstances move the flask. If it is necessary to use the fire extinguisher always take the fire extinguisher to the fire.

If solvent spilled on bench tops ignites, if possible, move bottles and flasks of unspilled solvent away from the area and then use the fire extinguisher. Never use the fire extinguisher wildly or more bottles may be knocked over and broken.

If your clothing is set on fire, move under the nearest safety shower and pull the chain.

ii) <u>Burns</u>

Beware of burns from hot glassware, hot iron rings or hot plates / heating blocks. Remember that the tip of a thermometer in the melting point or boiling point apparatus is at the temperature it records! If a hot plate / block has been used to heat a flask for an hour, then they are going to be hot until they have had a chance to cool.

iii) <u>Explosions</u>

Explosions are very fast exothermic reactions and are usually given by substances which can undergo internal redox reactions *e.g.* polynitro compounds such as T.N.T. (trinitrotoluene), picric acid (trinitrophenol) or nitroglycerin, or by substances which can decompose to give much more stable compounds such as peroxides, azides, fulminates and diazo compounds. You will be warned if any particular explosion hazard exists with the compounds that you are using. One of the commonest causes of unexpected explosions is as a result of distilling solutions to dryness leaving behind traces of explosive residues such as peroxides. This is one reason why a distillation vessel is never heated to dryness.

iv) Poisoning, Skin Contact, Chemicals in the Eyes

Several of the compounds you will use are poisonous *e.g.* most alcohols, amines and nitriles. The regulations concerning eating and drinking in the laboratory **must** be strictly adhered to. Do not pipette liquids by mouth. You should always remove your gloves and then wash your hands before leaving the laboratory.

Certain harmful substances can be absorbed relatively easily through the skin *e.g.* dimethyl sulphate, nitrobenzene, aniline, phenol, and phenylhydrazine. Aromatic amines are serious carcinogens. Always minimise contact with any chemical and wash with water after accidental contact. You may wear latex surgical gloves, if you wish. These will be available in the laboratory, or for improved protection, wear a pair of heavy duty gloves.

If harmful, irritating or flammable gases are used for, or produced during a reaction, then that part of the experiment should be conducted in the fumehood.

If you do splash something into your eyes wash them thoroughly (for about 15 minutes) by means of the eye wash station. Contact lenses *must be removed immediately* in order to wash the eyes.

v) Mixing Chemicals

Extreme caution should be used when mixing certain chemicals. If in doubt, ask your laboratory instructor. Be especially careful when dealing with waste as certain types of waste are incompatible (*e.g.* concentrated H_2SO_4 and organic waste).

Carefully observe the warnings about mixing water with certain reagents *e.g.* water should never be poured into concentrated H_2SO_4, or brought into contact with sodium. Chlorinated hydrocarbon solvent residues should be placed in the bottle provided. These should never be mixed with alkali. Never put concentrated H_2SO_4 in the organic waste containers. Specific warnings will be issued as required.

vi) Protection of Skin and Clothing

Acids and alkalis are extremely corrosive and will damage skin and clothing. Certain organic compounds are excellent dyes. This is the reason that you are required to wear a protective laboratory coat and safety glasses. Sandals or shorts must not be worn in the laboratory.

vii) Handling Reagents

Take great care to avoid contaminating reagents by:

- Always replace bottle tops as soon as you have finished dispensing reagents since many compounds react with moisture in the air, with oxygen or with carbon dioxide. Others are quite volatile. Do not mix the tops. If you do remove the reagent from the common work area temporarily, replace it as soon as possible.
- Never put the “wrong” pipette into the “wrong” reagent bottle.
- Always pour from the side of the bottle away from the label so that the name is not rendered unreadable. A reagent with no label at best is useless; at worst is dangerous.

viii) Tidiness

In order to work well, one of the first essentials is to keep your own, and the common areas such as reagent benches and fume-hoods, both clean and neat. Wipe up any spillages **immediately**. Care and tidiness in work is a large factor in safety, accuracy and efficiency.

d) <u>Waste Disposal</u>

This is a **VERY SERIOUS ISSUE**. When disposing of any waste material, **THINK** ABOUT WHAT YOU ARE DOING. This is for YOUR SAFETY and to PROTECT OUR ENVIRONMENT. Failure to comply with the waste rules could lead to you being suspended from the laboratory sessions and hence the loss of laboratory marks. IF YOU HAVE ANY DOUBT ASK !

Appropriate waste disposal is important, for example, chemically contaminated glass such as Pasteur pipettes cost $1100 per m^3 as it is buried, whereas clean glass costs $30 per m^3 and is recycled. **So, PLEASE MAKE SURE** the waste is going **into** the appropriate container. Here are some the rules:

- SPECIFIC INSTRUCTIONS FOR THE DISPOSAL OF CHEMICAL WASTES ARE GIVEN AT THE END OF EACH EXPERIMENT.
- NEVER POUR WASTE CHEMICALS DOWN THE SINK other than **very** dilute aqueous, non-toxic solutions.
- ANY BROKEN GLASSWARE SHOULD BE COLLECTED INTO A BEAKER AND CLEANED (RINSED WITH SOLVENT AND THEN WATER) BEFORE TAKING IT TO THE TECHNICIAN FOR REPLACEMENT, WHEN IT IS THROWN AWAY in the BLUE PAILS provided. (Chemically contaminated waste is costly to dispose of and can pose an environmental hazard).
- MAKE SURE WASTE IS PUT **IN** THE CORRECT CONTAINER.
- NEVER OVERFILL CONTAINERS (THERE **MUST BE** AT LEAST 5cm AIR SPACE AT THE TOP).
- IF A CONTAINER IS FULL, THEN ASK FOR A REPLACEMENT.
- Broken glass or other sharp objects MUST BE **CLEANED** then placed in the BLUE PAILS specifically marked for this purpose.
- **Solid chemical waste should be placed in the container provided in the fumehood.**
- **Organic solutions or solvents should be placed in the organic waste container at the waste station (different from the container for *solid* chemical waste).**
- **Aqueous solutions should be placed in the aqueous waste container at the waste station.**
- **Sample vials should be placed in the special boxes provided.**
- **Pasteur pipettes should be placed in the container assigned specifically for them.**
- **"Non-chemical" solid wastes should be placed in the waste bins.**

IF IN DOUBT, check your laboratory manual, and if you are still unclear, then ask your laboratory TA or consult a copy of Aldrich Chemical Co. catalogue (this includes disposal information on all chemicals they sell) for appropriate procedures. The waste disposal area must be kept clean. If it is not, then expect your laboratory TA to ask you to help clean it up.

11. CHECK IN / CHECK OUT PROCEDURES

Time has been allocated in the laboratory schedule for both **check-in** and **check-out** of your laboratory equipment. You will be assigned a laboratory equipment drawer. Please don't leave any personal items in your equipment drawer (this includes your laboratory safety coat and your safety glasses).

Using the equipment list, identify the contents of your laboratory drawers (your TA will help you do this if needed). If any glassware isn't clean, remove any organic residue using an organic solvent (try acetone first). Once the organic residue has been removed, wash the glassware using soap and water in the sink and then rinse it with cold water and then acetone. If it doesn't come clean, check with your laboratory TA. When you have determined that the equipment is clean, see your TA to sign the check-in form. You will not be allowed to perform any experimental work until the check-in form has been signed.

YOU ARE RESPONSIBLE FOR CLEANING YOUR EQUIPMENT, LOOKING AFTER IT, REMEMBERING TO PUT IT AWAY AFTER EACH LABORATORY SESSION.

During the semester, if you need to replace a broken or missing piece of equipment, go to EEEL 208, where the technician will issue a replacement.

Please note that malicious, careless or improper use of equipment or excessive breakage *etc.* may result in you being billed for the replacement cost of the item(s). Equipment costs are provided on the equipment list (in the course student laboratory manual).

You may also be required to pay for the replacement of broken communal equipment (*i.e.* not part of the normal locker equipment) if the breakage resulted from careless or improper use on your part.

It is important that you remember that you are also required to check-out of your drawer by the end of the course **EVEN IF** you drop or withdraw from the course.

ALL **STUDENTS WHO CHECK-IN (INCLUDING THOSE THAT DROP OR WITHDRAW) ARE REQUIRED TO CLEAN THEIR EQUIPMENT THEN CHECK-OUT. FAILURE TO CHECK-OUT BY THE LAST WEEK OF THE LABORATORY COURSE MAY RESULT IN A LATE CHE*CK-OUT FEE*.**

If you drop or withdraw from the course during the semester, you should check-out of your drawer straight away before you forget !

12. USEFUL REFERENCES FOR PRACTICAL ORGANIC CHEMISTRY

The results of chemical and biochemical research usually appear as notes, communications, or papers in the various chemical and biochemical journals. The easiest access to this information is through one of the electronic databases.

However, if all the information you require is a melting point or a good recrystallisation solvent a much simpler source of information would be one of the specialised secondary sources that have been developed. There are collections of methods of synthesis, methods of analysis, physical properties including melting point, boiling point, solubility, refractive index, vapour pressure, and also ultra-violet, infra-red, nuclear magnetic resonance (NMR) and mass spectra, and many others.

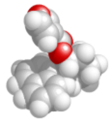
Listed below are a few of the more useful references that you might need to consult.

1. Handbook of Chemistry and Physics, 63rd edition, R.C. Weast, editor, Chemical Rubber Company, 1973. Contains physical and chemical properties for about 14,000 organic compounds in addition to a host of other physical and chemical data.
2. Dictionary of Organic Compounds, Heilbron, Oxford University Press.
   This dictionary is an alphabetical listing of over 25,000 organic compounds. It contains information concerning physical properties, recrystallisation solvents, reactions, derivatives, and literature references.
3. The Merck Index, 8th edition, Merck and Co. Inc., New Jersey, 1968
   This index lists about 10,000 compounds in alphabetical order and contains information concerning physical properties, solubility, hazards, and medicinal uses.
4. A Textbook of Practical Organic Chemistry Including Qualitative Organic Analysis, 3rd edition, A.I. Vogel, Longmans, 1961.
   An enormously useful volume which includes a detailed discussion of the theory and techniques of practical organic chemistry, and detailed preparations of nearly 500 representative organic compounds.

You will also find the following laboratory texts available in the University Library:

1. A Textbook of Practical Organic Chemistry, Vogel, 4th ed. Longmans, 1978.
2. Organic Experiments, Fieser & Williamson, 4th ed. D.C. Heath, 1979.
3. Introduction to Organic Laboratory Techniques, Pavia, Lampman & Kriz, Saunders, 2nd ed., 1982.

13. CHEM 351 Homepage



CHEM 351 maintains a Homepage on the WWW at:

https://www.chem.ucalgary.ca/courses/351/index351.html

The Homepage can also be accessed via the University of Calgary homepage and following the "path" to the Faculty of Science, Department of Chemistry, and Courses.

The CHEM 351 web pages contain information on all aspects of the course, such as schedules, a weekly bulletin of what's going on, past exam solutions, sample questions, a link to our version of the publishers (McGraw-Hill) On-Line Learning Center for the Carey textbook, plus links to other potentially useful WWW sites etc.

Within the Homepage is a section devoted to the laboratory that includes information, colour pictures and/or movies of important laboratory techniques and experimental set-ups, plus a few details of each week's experiment. This material is designed to help you prepare for each experiment ahead of your normal laboratory period.

14. TA OFFICE HOURS

TA contact information is available (provided by TA in first lab session, and on course website) if you need to talk about your laboratory work or your report. To use this resource optimally however, you should not expect the TAs or the instructors to just tell you answers to questions we've posed to you (as this is not their role, and they will not do this). They will try to encourage you and guide your thinking process. They will be able to help you best when you have thought about things yourself first, and you have attempted to piece things together. They will expect you to have thought about things yourself first - if you haven't, don't expect them to help you and don't complain about it!

15. INTRODUCTION TO THE EXPERIMENTS Online experimental schedule

The CHEM 351 laboratory provides:

- An opportunity to apply the various essential techniques of practical organic chemistry,
- The opportunity to further develop the skills required to work with the course material in general.

Chemistry is a practical subject. In particular, for an organic chemist much of the time, effort, and skills are devoted to the isolation of a product from a reaction mixture and its subsequent purification.

The practical materials in the manual are divided into two sections. The first part contains general information on the important experimental techniques and the second part contains the actual experiments. You will need to refer back to the techniques in order to complete the experiments. Each of the experiments covers a particular "task" or "tasks".

"Experiment #0" is an orientation session for the laboratory component and a chance to see your laboratory space, meet your TA, get a sense of laboratory expectations before starting the "real".experimental sessions.

Experiment #1 is an investigation of the solubility of organic compounds in common solvents, to probe the important connection between structure and function.

Experiment #2 introduces the technique of recrystallisation for purifying solids (you will use this technique several times during 351 / 353)

Experiment #3 introduces the techniques for measuring melting and boiling points – standard organic laboratory practices for characterizing/identifying solids and liquids (you will use it several times during 351 / 353)

Experiment #4 is a “dry” experiment investigating principles of molecular structure and shape.

Experiment #5 & #7 prepare and purify (by recrystallization) the very well-known analgesics (pain killers) aspirin and acetaminophen.

Experiment #6 work on developing spectroscopic problem-solving skills to help you learn how to solve a spectroscopy problem by approaching it as a puzzle.

Experiment #8 introduces the technique of liquid-liquid extraction to extract the naturally occurring stimulant, caffeine from tea leaves. Standard isolation and purification techniques will be used: liquid-liquid extraction and recrystallization.

Experiment #9 brings together some of the previous experiments in a chromatographic analysis of drugs.