Cooperative Organic Chemistry Student Laboratory Manual

COOPERATIVE ORGANIC CHEMISTRY STUDENT LABORATORY MANUAL

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PEER REVIEW STATEMENT

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INTRODUCTION

Dear student and researchers,

We're glad you're here. Welcome to CEM 255! This course is an evolving product of design-based educational research. We welcome your feedback on the materials and projects.

The development of this transformed laboratory course has been supported by a Howard Hughes Medical Institute (HHMI) grant and the ongoing optimization of the design is part of a National Science Foundation (NSF) grant. This adaptation of the original *Cooperative Organic Chemistry* features Green and Sustainable Chemistry, an urgent need for progress in science.

The basis for this laboratory curriculum is to experience science from the perspective of an organic chemist. If you took CEM 161 or 162 here at MSU, you would find many elements of the curriculum (especially the assignments and rubrics) to be very similar. You and your group members will be tasked with a project that will require you to design and carry out an investigation centered around an organic chemistry reaction that you are familiar with from CEM 251 and 252.

Within that investigation, each team member will use organic chemistry techniques and instrumentation to contribute to the overall argumentation needed to answer the project's scenario. These intellectual practices of designing and carrying out investigations, collecting and analyzing data, and engaging in argumentation from evidence are a few of the key Scientific Practices, i.e., the performances through which scientists "do" science. This course will give you opportunities to plan investigations, engage in green decision-making, use scientific models of phenomena, construct explanations, analyze data to generate evidence, and use that evidence in argumentation communicated in classically scientific ways—reports, scientific papers, posters, and oral presentations.

One "novel" portion of this course is the concurrent 50-minute "recitation", in which you and your team will meet in an online meeting to work through case studies of sustainability issues that require analysis and decision-making from a green and sustainable chemistry lens. As a unique communication opportunity, these case studies will give your team an opportunity to communicate scientific understanding and sustainability solutions in a policy context—through a technical report and constructing a policy paper. In these case studies, you will use your organic chemistry knowledge from lecture and your understanding of safety and green chemistry metrics from laboratory to make sustainable decisions and communicate those to important stakeholders: the public and public servants. The recitation content may subject to changes depends on different semesters.

You will sometimes make mistakes, but you will not be penalized by a "one-shot" opportunity to complete an experiment. Instead, in these activities you will have time and space to rectify any mistakes throughout the

multi-week projects and case studies. Not everything you try will work, and while this may be frustrating, keep in mind that **temporary frustration is a natural state of problem solving**.

PART I

PART 1: GREEN & SUSTAINABLE CHEMISTRY

1.

TWELVE PRINCIPLES OF GREEN CHEMISTRY

In the early 1960s, prominent reports and crises[1] prompted concern over the effects of chemical substances on the health of the environment and human populations. The logic behind disposal of chemical substances at this time had been "dilution as the solution to pollution," which (now debunked) posited that decreasing the substance's concentration would mitigate the harmful impact. The public outcry and expense of remediation of these environmental disasters led to legislation to mandate the manufacture, use, and disposal of chemical substances. By the 1990s, it was clear that the solution was to prevent pollution at the source, reducing the need for treatment of chemical wastes. One such type of pollution prevention scheme is the approach to synthesis that has been dubbed "Green Chemistry"[2]. The focus of this approach is to design and redesign chemicals and/or processes to prevent pollution and favor chemicals that pose less of a risk to human and environmental health. The ultimate goal of this "benign" approach to chemical synthesis is "to design synthetic methodologies that reduce or eliminate the use or generation of toxic feedstocks [reagent sources], by-products, [and] solvents."

1. Warner, John & Cannon, Amy & Dye, Kevin. (2004). Green Chemistry. Environmental Impact Assessment Review. 24. 775-799. 10.1016/j.eiar.2004.06.006.

2. Developed by Paul Anastas and John Warner. Anastas, P. T.; Warner, J. C. Green Chemistry: Theory and Practice. Oxford University Press: New York, 1998. More information available online through the American Chemical Society's Green Chemistry Institute.

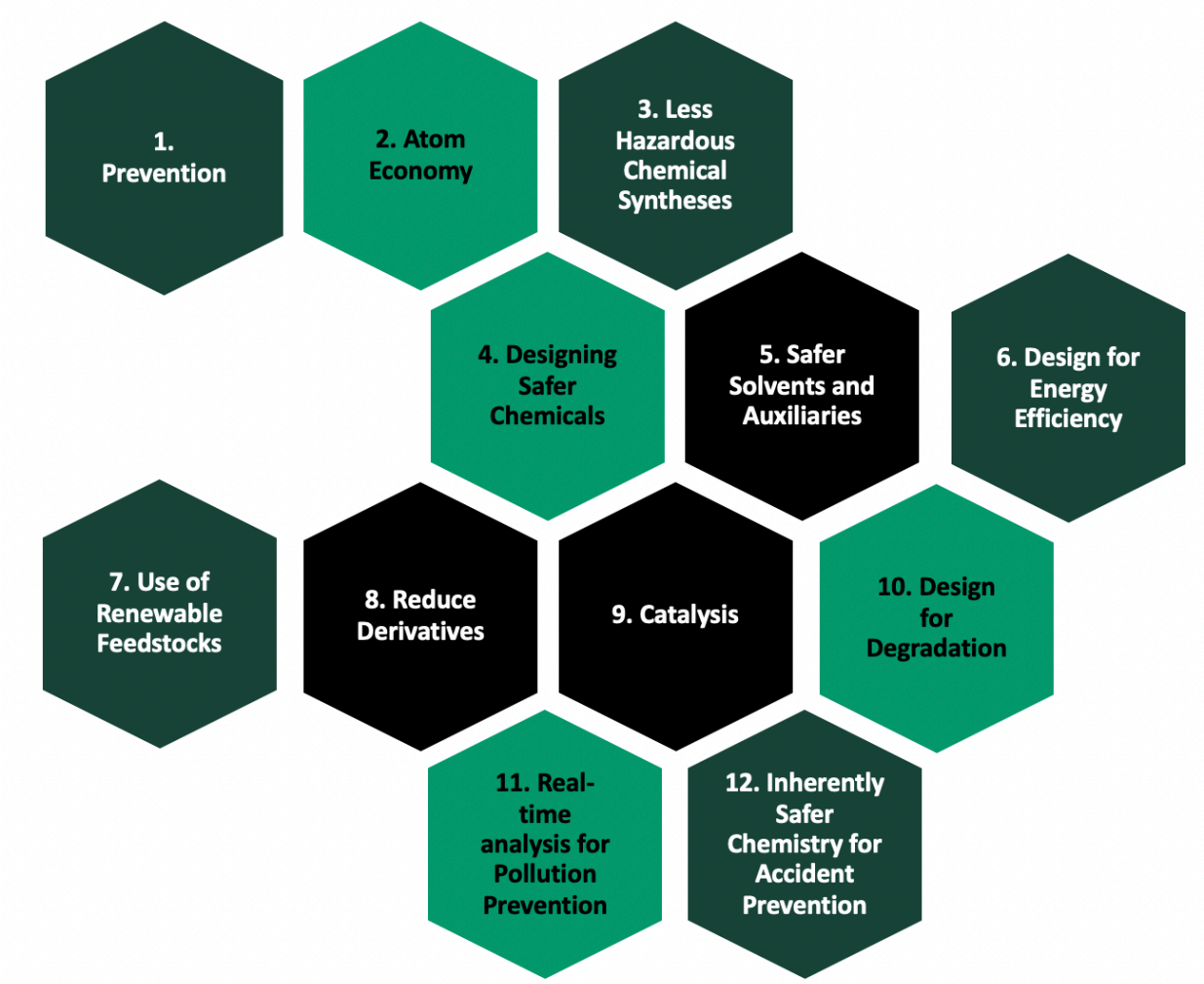

Figure 1. The 12 Principles of Green Chemistry were developed by Paul Anastas and John Warner in 1998. The figure was created by Mengqi Zhang, CC BY-NC.

Pictured in **Figure 1**, the culmination of innovation in the 1990s was the **12 Principles of Green Chemistry**. The twelve principles are explained in greater detail below, and the list includes: (1) prevention, (2) atom economy, (3) less hazardous chemical synthesis, (4) designing safer chemicals, (5) using safer solvents and auxiliaries, (6) designing for energy efficiency, (7) using renewable feedstocks, (8) reducing derivatives, (9) catalysis, (10) designing for degradation, (11) real-time analysis of pollution prevention, and (12) inherently safer chemistry for accident prevention.

These 12 principles represent the considerations and strategies that chemists should employ to analyze and optimize a chemical process as well as the chemicals throughout the entire life cycle. We will explore these principles, their associated metrics, and the broader impacts of the chemistry in the case studies associated with

this course. The individual green chemistry principles are enumerated below, but they follow overarching themes of safety, efficiency, and eco-consciousness through the prevention of pollution and attention to renewable sources of materials.

Principle 1: Waste Prevention

It is better to prevent waste than to treat or clean up waste after it has been created. [3]

This principle embodies much of the clarity gained in the 1990s around the problem with pollution. Rather than expensive and less effective remediation projects, it is far more efficient to design processes to be benign, especially by preventing the production of waste. While simple and clear, such an endeavor requires careful planning and analysis of the whole chemical process.

As we will cover in greater detail in the Handling Waste section, waste includes everything but the desired product—excess or used reagents and solvents or consumable materials such as plastic pipettes and weigh paper—but a more expansive way of viewing chemical processes is to consider the generation of waste at all time points in a chemical's life cycle.

A general Life Cycle for a material is pictured in **Figure 2**. From a chemical's extraction to its transformation in industrial processes to its shipping, intended use and end-of-life (reuse, recycling, disposal), there is waste generated at every step in the process.

3. This builds on work by Berkeley W. Cue, Jr., Ph.D., BWC Pharma Consulting, LLC. 12 Principles of Green Chemistry

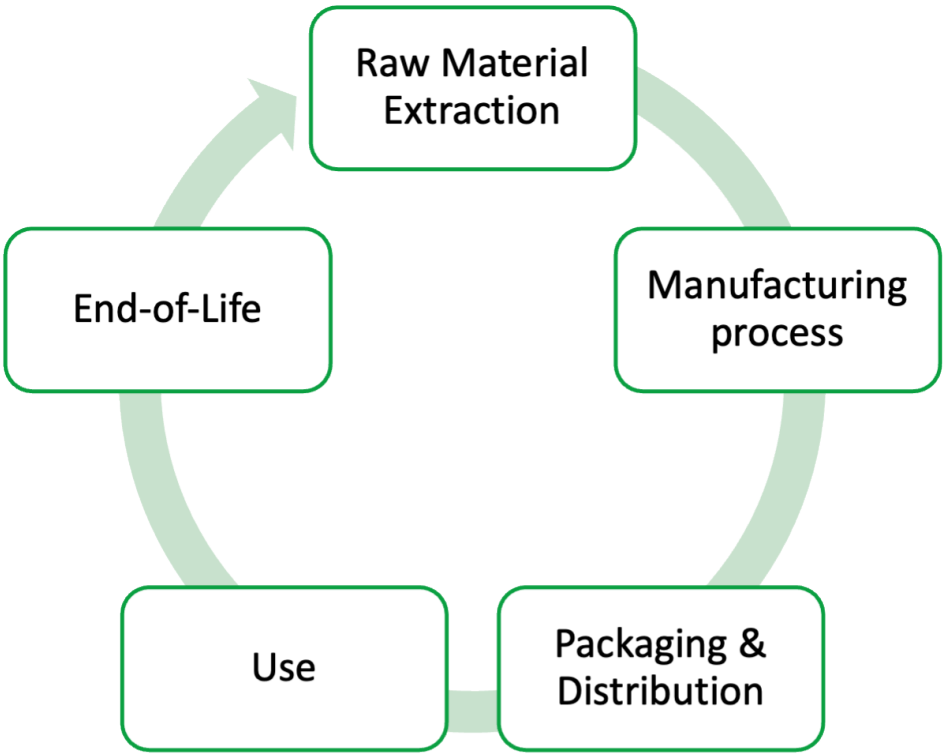

Figure 2. A visualization of the life cycle of a material by Elizabeth L. Day, CC BY-NC.

For the sake of the projects in this laboratory course, and for specific chemical processes in the case studies, we will focus on the waste generated by one component of this entire system: the laboratory, specifically at the scale of the laboratory bench. To aid our evaluations of waste generation for various processes, we can employ two metrics: e-factor and process mass intensity (PMI).

E-Factor

One classic metric of waste generation in organic chemistry is the e-factor[4], a ratio of the mass of waste produced in the process to the mass of desired product. This metric is encompassing in that it takes into account the yield of desired product (giving us a sense of the relative cost per unit mass of product) as well as the masses of actual waste: reagents, solvents (including loss of solvent), any auxiliary compounds, and potentially the amount of

4. Sheldon, R. A. "The E Factor: fifteen years on" Green Chem. 2007, 9, 1273-1283, doi: 10.1039/B713736M

fuel needed (albeit not an easy thing to calculate). However, because of the perceived "green-ness" of water-based reactions, water used in the process is **not** included in this calculation.

$$e - \text{factor} = \frac{\text{amount (kg) of waste}}{\text{amount (kg) of desired product}}$$

A higher e-factor indicates more waste generation and a greater environmental impact. Ideally, the e-factor would be zero. Oil refining industries report e-factors <0.1 whereas pharmaceutical industries report a range from 25-100.[7]

Process Mass Intensity (PMI)

This metric provides a more fine-grained analysis of the individual steps within a chemical process. The materials included in this mass ratio include reactants, solvents for reaction and purification, and catalysts.[5] As with e-factor, a lower PMI is desirable; the lowest possible PMI is 1. PMI can be calculated using the equation below.

$$PMI = \frac{\text{total (mass) in a process or process step (kg)}}{\text{mass (of) product (kg)}}$$

Overall, both metrics assess the amount of waste generated relative to the desired product yield. Neither metric makes a distinction about the hazards and relative risks of the waste in these calculations, which is an assessment that we will discuss more in the Classifying Hazards section.

Principle 2: Atom Economy

Synthetic methods should be designed to maximize incorporation of all materials used in the process into the final product.[6]

Atom economy is both a principle and a metric by which to judge a chemical process. As a principle, starting materials that closely resemble the structure of desired product will produce the highest atom economy,

5. Jiménez-González, C. et al "Using the Right Green Yardstick: Why Process Mass Intensity Is Used in the Pharmaceutical Industry to Drive More Sustainable Process" Org. Process Res. Dev. 2011, 15 (4), 912-917, doi: 10.1021/op200097d

6. This builds on work by Michael Cann, Ph.D., Professor of Chemistry, University of Scranton. 12 Principles of Green Chemistry

maximizing the number of atoms of starting materials in the desired product and therefore minimizing by-products as waste.

Atom Economy

The atom economy metric[7] is expressed as a percentage of the weight of all atoms of the reactants used (i.e., incorporated into the desired product) to the sum of the weights of all reagents. In an efficient, atom-economical reaction, this metric would be 100%, indicating that every atom of the reagents was somehow incorporated into the desired product. (Recall that FW is the formula weight, the sum of atomic weights of all atoms in a given chemical formula).

$$\text{Atom Economy } (\%) = \frac{\text{FW of atoms utilized}}{\text{sum of FW of all reactants}}$$

In your argumentation for reporting your findings for each lab project, you may be asked to calculate atom economy as a metric to judge reaction schemes. This metric could be used in contrast to one you are more familiar with: percent yield.

$$\text{Percent Yield } (\%) = \frac{\text{actual mass of desired product (g)}}{\text{theoretical mass possible based on reaction stoichiometry (g)}}$$

However, notice that yield doesn't incorporate the masses of any material besides the desired product, for example, catalyst. However, use of catalyst will impact the atom economy value of a synthetic route. Relying on this metric alone, one could have a reaction that is very efficient at generating a desired product, but inefficiently incorporate starting material into that product which leaves much of the starting mass to be relegated as waste.

Principle 3: Less Hazardous Chemical Syntheses

Wherever practicable, synthetic methods should be designed to use and generate substances that possess little-to-no toxicity to human health and the environment.[8]

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7. Trost, B. "The Atom Economy-A Search for Synthetic Efficiency" Science 1991, (254), pp. 1471-1477.

8. T**his builds on work by by David J. C. Constable, Ph.D., Director, ACS Green Chemistry Institute® 12 Principles of Green Chemistry**

The metrics discussed in Principles 1 and 2 help serve as benchmarks for efficiency and minimization of the **quantity** of waste; this principle attends to the **identity** of that waste, its inherent hazards, and the toxicity it may pose to human health and the environment. Although, as noted by the "wherever practicable", we recognize that there may not be preferred substitutions for certain reagents, solvents, or auxiliaries. In cases where a substitution is possible, there are practical concerns such as efficiency and cost to consider.

For the context of this course, several of these decisions have already been incorporated into the design of the projects your team will engage in. There will be opportunities for your team to engage in green decision-making surrounding solvent use for certain techniques. This Principle 5: Safer Solvents and Auxiliaries will provide you with a Greener Solvent List to guide those decisions.

EcoScale

The primary source material of hazard information for chemicals used and generated will be discussed further in Classifying Hazards in the next section. The metric we will use to incorporate hazard information into our assessments will be EcoScale.[9] EcoScale take penalty points off six categories: yield, price of reaction components, safety, technical setup, temperature/time and workup and purification, as shown in Table 1. A high EcoScale score is preferred. EcoScale metric favors high yields, low-cost, less reaction time and safer reaction conditions with low energy consumption and easier processing methods. The penalty points to calculate the EcoScale is summarized in the table below. Several online EcoScale calculators are available including: The Ecoscale

9. Aken, K. V.; Strekowski, L.; Patiny, L. Beilstein J Org Chem 2006, 2 (3), doi: 10.1186/1860-5397-2-3

Table 1: The Penalty Points to Calculate the EcoScale[10]

Parameter	Penalty points
1. Yield	(100 – %yield)/2
2. Price of reaction components (to obtain 10 mmol of end product)	
Inexpensive (< $10)	0
Expensive (> $10 and < $50)	3
Very expensive (> $50)	5
3. Safety[a]	
N (dangerous for environment)	5
T (toxic)	5
F (highly flammable)	5
E (explosive)	10
F+ (extremely flammable)	10
T+ (extremely toxic)	10
4. Technical setup	
Common setup	0
Instruments for controlled addition of chemicals[b]	1
Unconventional activation technique[c]	2
Pressure equipment, > 1 atm[d]	3
Any additional special glassware	1
(Inert) gas atmosphere	1
Glove box	3
5. Temperature/time	
Room temperature, < 1 h	0
Room temperature, < 24 h	1

Heating, < 1 h	2
Heating, > 1 h	3
Cooling to 0°C	4
Cooling, < 0°C	5
6. Workup and purification	
None	0
Cooling to room temperature	0
Adding solvent	0
Simple filtration	0
Removal of solvent with bp < 150°C	0
Crystallization and filtration	1
Removal of solvent with bp > 150°C	2
Solid phase extraction	2
Distillation	3
Sublimation	3
Liquid-liquid extraction[e]	3
Classical chromatography	10

[a] Based on the hazard warning symbols. [b] Dropping funnel, syringe pump, gas pressure regulator, etc. [c] Microwave irradiation, ultrasound or photochemical activation, etc. [d] $scCO_2$, high pressure hydrogenation equipment, etc. [e] If applicable, the process includes drying of solvent with desiccant and filtration of desiccant.

Principle 4: Designing Safer Chemicals

Chemical products should be designed to preserve efficacy of function while reducing toxicity.[11]

From a green chemistry perspective, safer chemicals are those that are designed and produced with a focus on minimizing negative impacts on the environment and on human health. Safer chemicals exhibit characteristics such as low toxicity, safe handling and use, high degradability, minimal waster generation and low energy requirements during production, etc.

Designing chemicals is beyond the scope of this course, but one goal of this course is to provide opportunities to use these principles and metrics to evaluate sustainability problems. As we will unpack further in the section Toxicology and Green & Sustainable Chemistry, careful consideration of the hazards inherent to certain chemicals (and processes) and how these chemicals interact with human health and the environment can be a powerful tool for identifying necessary areas for change and innovation. Tasks within this learning goal will incorporate some basic ideas of toxicology and environmental science.

Principle 5: Safer Solvents and Auxiliaries

The use of auxiliary substances (e.g., solvents, separation agents, etc.) should be made unnecessary wherever possible and innocuous when used.[12]

As you've covered in lecture, the solvent has an important role in the rate of the reaction as it interacts with the reactants. After the reaction, the solvent and other auxiliary compounds play an important role in the **workup**. The workup includes all process steps after the reaction step with the broad goals of separating, purifying, and confirming purity and yield of the target product. As you may discover in your projects and case study #1, the workup can often be the step in the entire chemical process that is the least green, requiring energy input and/or toxic or hazardous solvents to separate the desired product from by-products or impurities. If practicable, eliminating solvents altogether and making use of mechano-chemistry or other solvent-free setups is highly desirable.

On basic level, as this course was designed to feature Green and Sustainable Chemistry, a lot of substitutions for safer, less hazardous chemicals have already been made for you. However, there are explicit opportunities for you to make sustainable decisions within the context of your investigations. When appropriate, you will be

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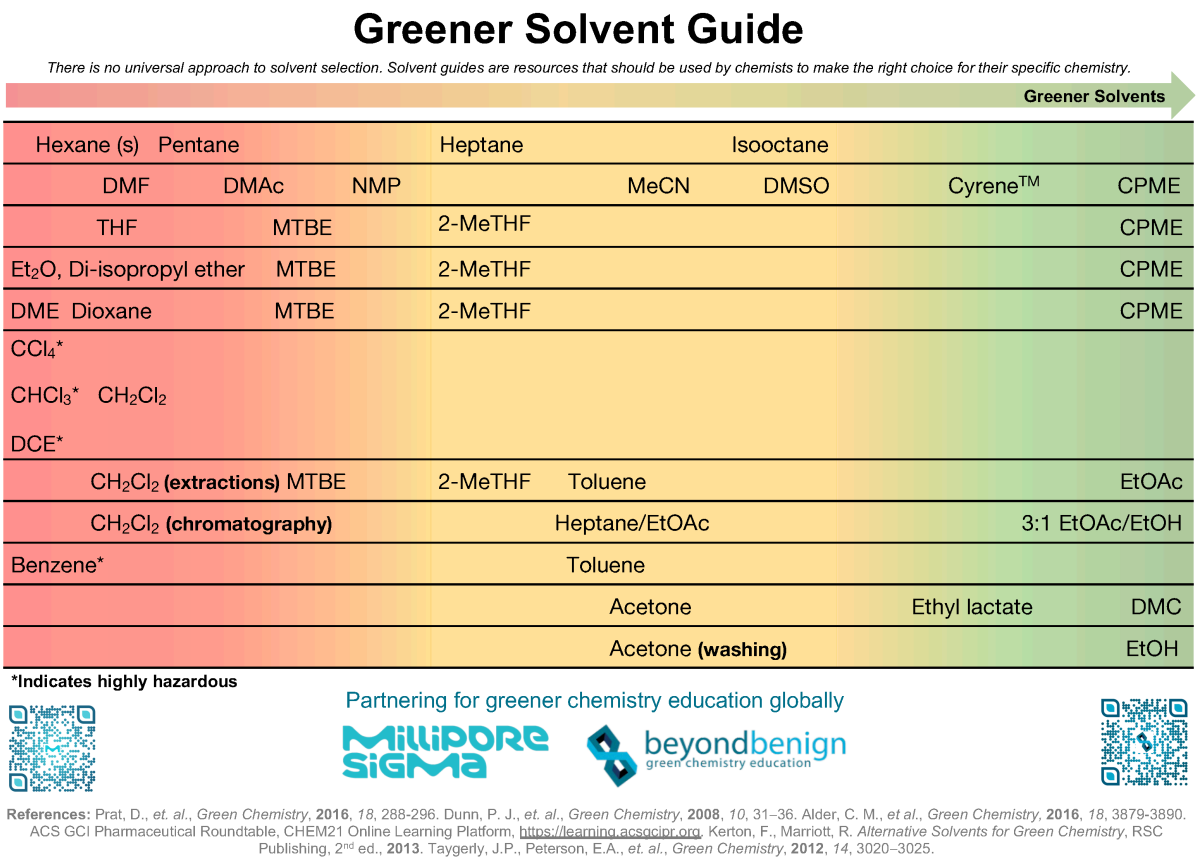
11. This builds on work by Nicholas D. Anastas, Ph.D., U.S. Environmental Protection Agency- New England 12 Principles of Green Chemistry

12. This builds on work by Dr. Concepcíon (Conchita) Jiménez-González, Director, Operational Sustainability, GlaxoSmithKline 12 Principles of Green Chemistry

directed to choose a solvent with the characteristics (polarity, basicity, etc.) from the Greener Solvent List in Table 2.

Greener Solvent List

Table 2. Greener Solvent List from Beyond Benign (V.1 March 2020)



The Greener Solvent Guide. Image courtesy of Beyond Benign (Beyond Benign), CC BY-NC-ND 4.0.

As noted, there's no universal approach to solvent selection. In the sections following, instructions for how to test solvents on a small scale are given. To help interpret this solvent guide table, each bullet point below discusses a line in Table 2.

- If seeking to replace hexane(s) and pentane as nonpolar solvents, this solvent guide suggests heptane and isooctane as problematic, yet greener alternatives.
- If looking to replace Dimethylformamide (DMF), dimethylacetamide (DMAc), or N-

methyl-2-pyrroliodone (NMP), the greenest alternatives available are cyrene, cyclopentyl methyl ether (CPME), or dimethyl carbonate (DMC); more problematic alternatives include dimethyl sulfoxide (DMSO) and acetronitrile (MeCN).

- If looking to replace Tetrahydrofuran (THF), the best alternative is CPME, although Methyl tert-butyl ether (MTBE) or 2-methyltetrahydrofuran (2-MeTHF) are viable yet problematic alternatives.
- If trying to replace Diethyl ether (Et_2O) or Di-isopropyl ether, the best alternative is CPME, although Methyl tert-butyl ether (MTBE) or 2-methyltetrahydrofuran (2-MeTHF) are viable yet problematic alternatives.
- If trying to replace Dimethoxyethane (DME) or dioxane, the best alternative is CPME, although Methyl tert-butyl ether (MTBE) or 2-methyltetrahydrofuran (2-MeTHF) are viable yet problematic alternatives.
- For highly hazardous solvents chloroform ($CHCl_3$), dichloroethane (DCE), or carbon tetrachloride (CCl_4), the only alternative is also undesirable, dichloromethane (DCM).
- For replacing pyridine in applications as a base, triethylamine (Et_3N) is a problematic alternative.
- DCM is commonly used in extractions despite its undesirability, the greenest alternative is ethyl acetate (EtOAc), but MTBE, 2-MeTHF, or toluene are viable yet problematic alternatives.
- For DCM's use in chromatography, the ideal replacement would be a 3:1 ratio of EtOAc to ethanol (EtOH); a 50:50 mixture of EtOAc to heptane could be used, although is less green.
- Highly hazardous benzene is best replaced by toluene, which is a problematic alternative.
- Finally, one can "green" their procedure by replacing acetone for ethyl lactate or DMC, or with EtOH in the context of washing the product.

Principle 6: Design for Energy Efficiency

Energy requirements should be recognized for their environmental and economic impacts and should be minimized. Synthetic methods should be conducted at ambient temperature and pressure.[13]

As noted in the previous Principle, there are energy-expensive steps in the workup (and in the reaction step itself) procedures following a reaction. The use of electricity, energy in the form of heat, or water to cool condensing setups all have an economic cost, and this use of energy does contribute to the generation of greenhouse gases (GHGs) and the effects of climate change. While calculating energy usage (and the relative GHG creation) can be tricky in the laboratory classroom, we can prioritize reactions that run at ambient (room) temperatures and pressures. Energy consumption in an academic environment may be negligible. The energy usage and associated cost can be significant on a large industrial scale, while extreme temperature and pressure

13. This builds on work by Dr. David Constable, Director, ACS Green Chemistry Institute® 12 Principles of Green Chemistry

conditions are prevalent in various industries. Design for energy efficiency is imperative to reduce economic and environmental costs.

Principle 7: Use of Renewable Feedstocks

A raw material or feedstock should be renewable rather than depleting whenever technically and economically practicable.[14]

As noted in the Life Cycle in **Figure 2**, an important sustainable consideration is the source of the chemicals and materials we use at the bench. Historically, since the 1800s, chemicals have been refined and transformed from petroleum. Most chemicals we use are extracted from oil, a non-renewable resource! Even hydrogen peroxide (H_2O_2)—which is considered a greener oxidizing agent, especially compared to heavy metals such as chromium or manganese—derives from oil refineries rather than one of the pharmacy shelves at the store.

This principle highlights the urgent need to develop renewable sources of chemicals. However, as we will explore in Case Study, some renewable sources are also food sources (such as sugar cane) and removing those materials from the food supply to serve the chemical industry could have detrimental impacts on UN Sustainable Development Goal #2 – Zero Hunger.[15]

The 17 UN Sustainable Development Goals (SDGs), shown in **Figure 3**, was adopted by the United Nations Member State in 2015, calling for action by all countries in a global partnership towards peace and prosperity for people and the planet, now and into the future.

14. This builds on work by Dr. Richard Wool, Professor of Chemical and Biomolecular Engineering and Director of the Affordable Composites from Renewable Materials program, University of Delaware. 12 Principles of Green Chemistry

15. United Nations Sustainable Development Goals (SDGs), 2015, United Nations Sustainable Development

Figure 3. UNESCO Sustainable Development Goals, by United Nations Educational, Scientific and Cultural Organization, 2015, CC BY-SA 3.0 IGO.

Principle 8: Reduce Derivatives

Unnecessary derivatization (use of blocking groups, protection/deprotection, temporary modification of physical/chemical processes) should be minimized or avoided if possible, because such steps require additional reagents and can generate waste.[16]

In research and industrial laboratories, the use of blocking, protecting/deprotecting, or other temporary modifications to reagent molecules is common to drive the reaction in a desired direction. This derivatization of molecules in a reaction scheme is useful, but it introduces more steps and uses more energy and material resources.

16. This builds on work by Peter J. Dunn, Green Chemistry Lead, Pfizer 12 Principles of Green Chemistry

Designing reactions and processes to avoid derivatization is beyond the scope of this course. However, as you take future chemistry, biochemistry, or biology courses, be aware that the solution to this issue likely lies in enzymatic or biological processes to carry out these chemical transformations.[17]

Principle 9: Catalysis

Catalytic reagents (as selective as possible) are superior to stoichiometric reagents.[18]

In the general and organic chemistry lectures, we have explored the effect of a catalyst on a reaction's rate. These substances—chemicals, enzymes, or even surfaces—accelerate a reaction without serving as a reactant by providing an alternate pathway for the reaction to occur, resulting in a lower activation energy than the uncatalyzed pathway (which is the "stoichiometric" ratio of reagents).[19] With this lower activation energy, ambient temperatures (and pressures) are more feasible, and a catalyzed-reaction pathway may be more selective, require fewer process steps, require less reagent, produce less waste, and result in a higher atom economy.[20]

Principle 10: Design for Degradation

Chemical products should be designed so that at the end of their function they break down into innocuous degradation products and do not persist in the environment.[21]

This green principle challenges molecular designers (expert chemists) to consider end-of-life when choosing a target molecule. Referring to the Life Cycle in **Figure 2**, we can leverage the structure-property relationships to

17. Beyond Benign Green Chemistry: Principles and Lab Practices 2020 A Guide to Green Chemistry Experiments for Undergraduate Organic Chemistry Labs (PDF)

18. This builds on work by Roger A. Sheldon, Ph.D., Emeritus Professor of Biocatalysis and Organic Chemistry, Delft University of Technology and CEO of CLEA Technologies B.V. 12 Principles of Green Chemistry

19. Cooper, M. M.; Klymkowsky, M. "Chapter 8: How far? How fast?" CLUE: Chemistry, Life, the Universe, and Everything 2019. https://openbooks.lib.msu.edu/clue/

20. Beyond Benign Green Chemistry: Principles and Lab Practices 2020 A Guide to Green Chemistry Experiments for Undergraduate Organic Chemistry Labs (PDF)

21. This builds on work by Rich Williams, Founder and President at Environmental Science & Green Chemistry Consulting, LLC 12 Principles of Green Chemistry

design molecular targets that will be effective for their use but at the end-of-life stage will "readily degrade and [won't] persist and accumulate in the environment."[22]

While the work of designing products to be effective yet degradable is beyond the scope of this course, we will use this principle in the Case Studies as we explore current, urgent sustainability problems that illustrate how useful chemical and material products can have long-lasting and negative effects in their end-of-life.

Principle 11: Real-Time Analysis for Pollution Prevention

Analytical methodologies need to be further developed to allow for real-time, in-process monitoring and control prior to the formation of hazardous substances.[23]

While this principle suggests an area of innovation and investment for industry, pollution prevention is a challenge if we don't have accurate and timely data to evaluate processes. As you may find as your team collects data for the green metrics and tools described above, some data may be challenging to collect with accuracy and precision.

Principle 12: Inherently Safer Chemistry for Accident Prevention

Substances and the form of a substance used in a chemical process should be chosen to minimize the potential for chemical accidents, including releases, explosions, and fires.[24]

This principle is well-aligned with Principle 3: Less Hazardous Chemical Syntheses and the RAMP safety approach described in the next section. Designing and carrying out greener chemical investigations requires attention to minimizing risk of hazards by choosing substances that are more benign. Coupled with a culture of safety and thoughtful preparation for emergencies, the green chemistry laboratory is inherently safer.

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22. Beyond Benign Green Chemistry: Principles and Lab Practices 2020, p. 13, A Guide to Green Chemistry Experiments for Undergraduate Organic Chemistry Labs (PDF)

23. This builds on work by Douglas Raynie, Assistant Professor, Chemistry & Biochemistry, South Dakota State University. 12 Principles of Green Chemistry

24. This builds on work by Shelly Bradley, Campus Chemical Compliance Director, Hendrix College; Dr. David C. Finster, Professor of Chemistry, Wittenberg University; and Dr. Tom Goodwin, Elbert L. Fausett Professor of Chemistry, Hendrix College. 12 Principles of Green Chemistry

Other Metrics

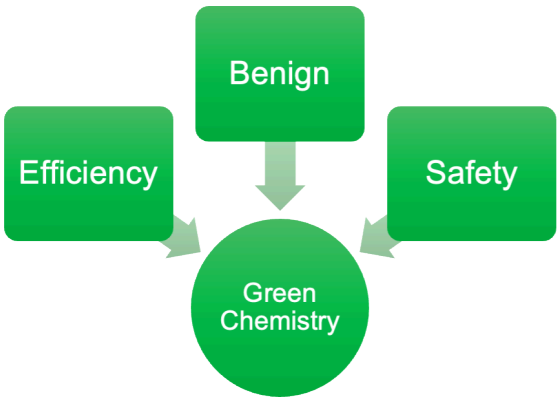
Green Chemistry is a rapidly evolving field with new developments emerging every day. Other green chemistry metrics are available to evaluate the greenness of a production process, each offering unique insights into different aspects of environmental impact and sustainability.

- Lice Cycle Assessment (LCA): Focuses on evaluating the environmental impacts associated with all stages of a product's life, from raw material extraction through manufacturing, distribution, use, and disposal or recycling, in the hopes of improving environmental performance.
- Reaction Mass Efficiency (RME): Provides a quantitative measure of how effectively starting materials are converted into the desired product, considering both the yield and the mass of reactants used. The calculation is typically expresses in percentage.
- DOZN quantitative Green Chemistry Evaluator: A quantitative, industry-first tool that uses the 12 Principles of Green Chemistry for comparing the relative greenness of similar chemicals, synthetic routes and chemical processes. More information is available via: DOZN™ Quantitative Green Chemistry Evaluator.
- And more...

By employing a combination of the metrics, chemists and industries can make informed decisions to design and implement greener processes that align with sustainability goals and regulatory requirements.

Three Themes on Green and Sustainable Chemistry

These twelve principles could be grouped into three overarching themes: a commitment to efficiency, safety, and overall benign chemistry. As you plan and carry out your investigations for each project, the planning questions will guide you to consider the efficiency—in terms of e-factor, PMI, and atom economy—benign-ness of the substances in the reaction—using the hazard information and EcoScale—and uphold a culture of safety within your team and course. The next section will detail how a RAMP approach to safety will be enacted in the planning process.



Three themes on green chemistry principles: efficiency, benign, and safety by Elizabeth L. Day, CC BY-NC.

2.

SOCIAL & ENVIRONMENTAL JUSTICE

> "Environmental justice is defined as the fair treatment and meaningful involvement of all people regardless of race, color, national origin, or income with respect to the development, implementation, and enforcement of environmental laws, regulations, and policies."[1]

Social and environmental justice are intertwined concepts that address fairness and equity in the distribution of resources, opportunities, and burdens within society, while also considering the impact of human activities on the natural environment. When viewed through the lens of social and environmental justice, green chemistry also serves to highlight several common concerns:

1. **Equitable access to safer products and eliminate exposure to harmful chemicals:** Green chemistry aims to ensure that all individuals and groups have fair access to products that are benign for human health and the environment. This involves taking into account the requirements of marginalized communities and underrepresented groups which might suffer from exposure to harmful chemicals. The overarching aim is the equitable allocation of environmental benefits and burdens, as seen through the lens of environmental justice, and the enhanced empowerment of marginalized communities and individuals, as seen from the perspective of social justice.
2. **Protection of Vulnerable Groups**: Pollution prevention goes hand in hand with the focus on protection of vulnerable populations, such as low-income communities and minority groups, from environmental hazards and ensuring their right to a healthy environment.
3. **Sustainable Development**: Green chemistry promotes the efficient use of renewable resources and the reduction of waste in chemical processes. This can contribute to sustainable development by conserving natural resources and minimizing the environmental impact of chemical production. This objective aligns with environmental justice, which advocates for sustainable development practices that minimize environmental harm and long-term well-being of both people and the planet.
4. **Global Impact**: Green chemistry recognizes that environmental issues and social injustices often have global implications. By promoting sustainable practices and technologies, it aims to address challenges

1. R. Bullard, Environmental Justice, International Encyclopedia of the Social & Behavioral Sciences, 2001, 4627-4633, ISBN 9780080430768, https://doi.org/10.1016/B0-08-043076-7/04177-2.

such as climate change and biodiversity loss on a global scale.

Several states in the United States have proposed laws, executive orders, or initiatives to promote green chemistry and encourage the inclusion of sustainable practices into manufacturing and industry. For instance, Executive Directive 2006-6[2] from the State of Michigan emphasized the importance of implementing green chemistry principles to reduce the use and generation of hazardous substances and encourages state agencies to promote and support research, education, and the implementation of green chemistry practices. These efforts reflect a growing recognition of the importance of green chemistry in addressing environmental and public health concerns.

2. State of Michigan, Governor [Jennifer M. Granholm]. Executive Directive No. 2006-6 (28): Promotion of green chemistry for sustainable economic development and protection of public health, 17 Oct 2006.

PART II

PART 2: SAFETY INFORMATION

In a laboratory space—especially instructional or learning laboratories—safety and management of risk is paramount. As the discipline and industry reorients to green and sustainable chemistry, one key theme across the Twelve Principles of Green Chemistry is a commitment to safer chemistry. Similarly, one of the goals of this course is to develop your capacity to recognize, assess, and manage risk and prepare for emergencies within a culture of safety.

3.

USING A RAMP APPROACH TO LEARN ABOUT HAZARD AND RISK

R: Recognize hazards

A: Assess the risks of hazards

M: Minimize the risks of hazards

P: Prepare for emergencies

This RAMP framework for laboratory safety is derived from the work of Hill and Finster[1] and supported by the American Chemical Society (ACS), a US-based professional network of chemists. For this course, as a part of the Planning and Carrying Out Investigations (aka Planning Documents) portion of each session within a course project, your team will engage directly with these principles in your planning as well as uphold them during your team's investigations.

R: Recognize Hazards

The first principle is to recognize the presence of hazards and the risks that they pose. This section will differentiate hazard and risk, identify the key sources of hazard information, and characterize different classes of hazards.

Defining a Hazard and a Risk

A **hazard** is any source of potential damage or harm to a person's health.[2] It is an intrinsic property of the material (and/or process). These hazards will be classified into broad groups below. It is important to realize that hazards are not limited to chemicals (i.e., non-chemical equipment may potentially pose hazards), such as the potential damage from broken glass or the damage over time from carrying out repetitive motions, such as

1. Hill, R. H.; Finster, D. C. Laboratory Safety for Chemistry Students. 2nd edition, 2016.

2. ACS Chemical & Laboratory Safety. "Basics & RAMP" ACS Institute Safety Basics & RAMP

lifting or small movements as pipetting. **We can never eliminate a hazard, but we can manage the risks that hazards pose.**

A **risk** is the probability of harm or damage from a hazard.[3] It is the combination of the likelihood of an event (the exposure) and the severity of the hazard or damage that event poses.[4]

As we noted above, the hazard is an intrinsic property and cannot be eliminated, but we can minimize the likelihood of the event in which the hazard can cause damage, thereby minimizing the risk. Therefore, the emphasis is on probability because the risk can be managed through careful planning, preparation, and Personal Protective Equipment (PPE).

Where to Find Information About Hazards?

(Materials) Safety Data Sheets (M)SDS

Safety Data Sheets (SDS), formerly called Materials Safety Data Sheets (MSDS), provide information including properties, handling, and hazards of materials. As you plan your investigations, be sure to thoroughly read the SDS for each new chemical you use. In the appropriate space on the Planning Documents, you should list each chemical or material necessary for your plan, and explicitly recognize the hazards these materials pose on the appropriate line.

There are numerous sources of (M)SDS on the internet. Using a search engine, input the chemical name plus the term MSDS or SDS (e.g., "MSDS Sodium Chloride" or "SDS Isopropyl Alcohol"). If you always want to use SDS from the same source for consistency of SDS layout, you may want to search for the SDS from a particular chemical manufacturer, such as Flinn Scientific or Sigma-Aldrich. While every manufacturer may have a slightly different arrangement to their (Materials) Safety Data Sheets, or (M)SDS, each should contain all

3. ACS Chemical & Laboratory Safety. "Basics & RAMP" ACS Institute Safety Basics & RAMP

4. American Chemical Society. Free Online Course: Foundations of Chemical Safety and Risk Management. American Chemical Society Login

of the same sections of information. The Hazard Communication Standard Safety Data Sheets link should help you to navigate any SDS: OSHA Brief Hazard Communication Standard: Safety Data Sheets (PDF).

As you peruse the SDS for the chemicals and materials that your team decides that is necessary for your investigation, use these sheets to collect the relevant hazard information (explicated below) as well as information about proper PPE and waste disposal. You will be required to note any PPE necessary, even if it's "appropriate goggles and nitrile gloves". Your instructor will review your chemical safety plan and make recommendations.

Note: To foster a culture of safety in the laboratory, safety information about each project will be outlined in the following format (yellow background, red border) in the scenario. This information will cover the known, required chemical and physical materials that the project calls for. Your team—with the support of your instructor—will add to this information during your Planning Documents to account for the specifics of your team's unique investigation and choices.

Safety notes:

- Be sure to consult the MSDS/SDS for any compound that you might work with.
- Wear safety goggles, gloves, and appropriate clothing at all times in the laboratory.
- Dispose of waste in the labeled containers. **Do not** pour any waste down the drain unless you check with your instructor first.
- Use great care when transferring solutions of acids and bases.
- If you spill a strong acid or base on your clothes or skin, rinse with large amounts of water immediately and ask one of your team members to tell your TA.
- All of the unknown compounds that you will work with in this project are Generally Recognized as Safe, but normal safety precautions should be observed.

Classifying Hazards

Of the hazard information contained on an SDS, there are two broad systems of communicating hazard information you should be aware of: the NFPA diamond and the GHS labels. These systems should help you enumerate the hazards posed by the materials in your Planning Documents. In the green chemistry case studies associated with this laboratory course, the following information is helpful for evaluating sustainability solutions using green chemistry tools and metrics (described in Twelve Principles of Green Chemistry).

National Fire Protection Association (NFPA) Diamond

This ubiquitous NFPA[5] diamond summarizes major classifications of hazards in a visual manner. The individual diamonds will have numbers of symbols in them to communicate at a glance which hazards are present and the severity of the risks that they pose. The health (blue), fire (red), and reactivity (yellow) diamonds communicate hazards on a scale of 0 (least hazardous) to 4 (most hazardous) within their respective categories, and the white diamond uses abbreviations to communicate Special hazards.

5. National Fire Protection Association. NFPA 704: Standard System for the Identification of the Hazards of Materials for Emergency Response. NFPA 704

Blue – Health Hazards

4	Deadly
3	Extreme Danger
2	Hazardous
1	Slightly Hazardous
0	Normal Material

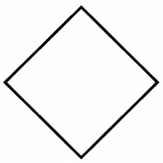
Red – Fire Hazards

4	Below 73°F
3	Below 100°F
2	Above 100°F
1	Above 200°F
0	Will Not Burn

Yellow – Reactivity Hazards

4	May Detonate
3	Shock & Heat May Detonate
2	Violent Chemical Change
1	Unstable if Heated
0	Stable

White – Special Hazards



ACID	Acid
ALK	Alkali
COR	Corrosive
OXY	Oxidizer
☢	Radioactive
W	Use **NO** Water

Globally Harmonized System (GHS)

The GHS labels are an internationally adopted system[6] of graphic labels intended to provide hazard information at a glance. The base template (shown at the right) of a white diamond with a thick red border will be filled with a specific graphic to indicate the class of hazard that is posed by the material.

Note that the numbering system for GHS is opposite of NFPA: a 1 is more hazardous than a 4.

The labels could be grouped into broader categories of hazards, although there are more sophisticated ways of classifying these hazards. For this course, one major category of hazards will be those posed by the chemicals or physical materials used to carry out reactions, workups, and overarching investigations. The chemical hazards are inherent properties, and include toxicity, flammability, corrosivity, and reactivity. The first group of GHS symbols below emphasis categories of hazards that fall under the broad umbrella of hazards posed by reactivity or conditions like the high pressure of compressed gas canisters. These properties have implications for shipping, storage, use, and waste generation.

6. UNECE. Globally Harmonized System of Classification and Labelling of Chemicals, 2015. UNECE GHS

Chemical Hazards

Explosive:
reacts spontaneously, exothermically & uncontrollably

- Explosives
- Self-reactive substances
- Organic peroxides

Flammable:
ignites and burns if exposed to source of ignition

- Flammable gases, aerosols, liquids, and solids
- Pyrophoric liquids or solids
- Self-heating substances
- Self-reactive substances
- Substances that emit a flammable gas upon contact with water
- Organic peroxides

Corrosive:
reacts and damages materials and body tissues

- Skin corrosion/burns
- Eye damage
- Corrosive to metals

Oxidizer:
reacts readily with organic compounds or reducing agents without energy input

- Oxidizing gases, liquids, and solids

Compressed Gas:
canisters of highly pressurized gas

- Gases under pressure

Health Hazards

Health hazards include the biological and chemical materials that pose a hazard to human or environmental health. These hazards are an issue all along the life cycle of a chemical, but for our purposes we are most concerned about safe use and proper disposal. As you compile the SDS for the necessary chemicals for planning your investigations, pay special attention to the following GHS symbols. Notice how corrosion is also on this list, as it poses a human health hazard.

Toxic:
poisonous or capable of damage or death if ingested

- Acutely toxic substances may be fatal or toxic if inhaled, ingested, or absorbed through the skin

Irritant:
causes reversible inflammation or irritation to body tissue

- Irritant (skin and eye)
- Skin sensitizer
- Acute toxins
- Narcotic effects
- Respiratory tract irritants
- Hazardous to the ozone layer (non-mandatory)

Corrosive:
able to burn or corrode organic tissue by chemical action

- Skin corrosion/burns
- Eye damage

Environmental Hazard:
poses a known hazard to the environment

- Acute aquatic toxins
- Chronic aquatic toxins

Health Hazard:
poses a known hazard to human health

- Respiratory sensitizers
- Carcinogens
- Mutagens
- Reproductive toxins
- Target organ toxins, single or repeated exposure
- Aspiration toxins

Note: All GHS pictograms Copyright © United Nations, 2023. Reprinted with the permission of the United Nations.

Waste Disposal

After you have finished your experiments, dispose of waste and excess as instructed. **Do not put anything down the sink unless specifically told to do so by your instructor.** It is your responsibility to indicate on the planning document the appropriate waste disposal methods for each chemical; this information is available on the Safety Data Sheet (SDS). The stockroom will provide the appropriate waste containers for the relevant categories of hazards listed above.

A: Assess the Risks of Hazards

While some of these terms may be (incorrectly) used interchangeably, each class of hazards could co-occur. Some **toxins**—proteins from plants, animals, or bacteria that are **toxicants** or toxic substances—are also listed as human health hazards because of their carcinogenicity, mutagenicity, or targeting of specific organs. However,

some toxins are only **acute** and are classified as irritants. Other toxic substances pose a **chronic** hazard; this distinction between "acute" and "chronic" will be explore further in the next section.

Toxicology and Green & Sustainable Chemistry

As we explore sustainability issues within the course and apply our chemistry knowledge to our everyday lives, it's important to recognize that the environmental and human health hazards have a **dose-dependent** relationship to the deleterious (negative) effects observed. The field of study that explores these relationships is the field of Toxicology. We will use a few of their foundational ideas in our investigations and explorations within this course.

Dose: How Much

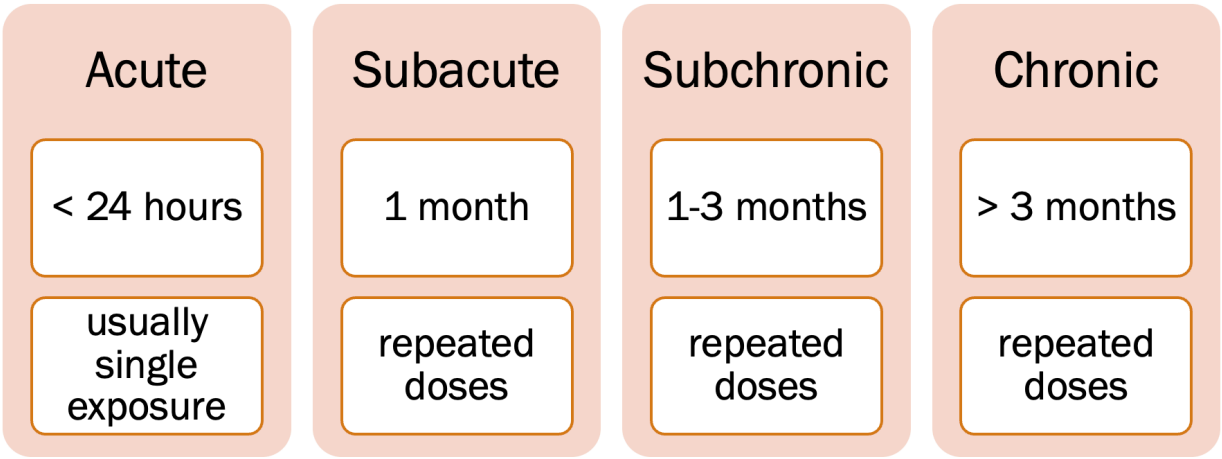
"Dose makes the poison" has become a guiding principle in toxicology. When determining whether a material is toxic, data on the route on ingestion (inhalation, ingestion, or contact with skin) and the dose relative to bodyweight are important qualifications to the assessment. In this section, we will briefly define terms to describe a lethal dose of a toxic substance.

Table 3 summarizes the toxicological definition of toxic and highly toxic, according to OSHA standards. A (median) lethal dose is abbreviated as LD_{50} and a median lethal concentration is abbreviated as LC_{50} (for toxic gases). Notice that highly toxic substances have a lower median lethal dose/concentration than toxic substances.

Table 3. OSHA Definitions of Median Lethal Dose/Concentrations for Various Absorption Methods.[7]

	Administered Orally$^{\alpha}$	Continuous Contact with Skin$^{\beta}$	Continuous Inhalation (in air)$^{\alpha}$
Median Lethal Dose/ Concentration	LD_{50} (mg/kg bodyweight)	LD_{50} (mg/kg bodyweight)	LC_{50} (ppm)
Toxic	50 – 500	200 – 1000	200 – 2000; 2-20 mg/L
Highly Toxic	<50	<200	<200 2 mg/L
administered to: $^{\alpha}$ albino rats (200-300 g); $^{\beta}$ albino rabbits (2-3 kg)			

Duration: how long and how often[8]



On the spectrum from acute to chronic toxicity, one goal of this course is to distinguish between the two extremes. Acute toxicity occurs less than 24 hours after exposure, usually a single dose but possibly multiple or repeated doses within a 24-hour period.[9] Chronic toxicity is the cumulative damage to specific organ systems over a period of greater than three months through repeated exposures (doses). Some substances—such as alcoholic beverages—can induce both acute toxicity (depression of the central nervous system) and chronic toxicity (cirrhosis of the liver).

M: Minimize the Risks of Hazards

Personal Protective Equipment (PPE)

Proper Attire (Required)

Students are required to wear lab-appropriate attire to be permitted in the laboratory:

- **Tops: Shoulders, midriffs, and backs must be fully covered.** Prohibited items include, but are not limited to, muscle tanks or other sleeveless tops, crop tops, backless shirts, and tops with mesh or other

7. Interactive Learning Paradigms Incorporated (ILPI) "The MSDS Hyperglossary: Toxic" 11 Nov 2020 MSDS Hyperglossary: Toxic

8. Nardei, S. "Toxicology Basics" from University of Arizona Southwest Environmental Health Sciences Center. Toxicology Basics

9. Beyond Benign. "Introduction to Toxicology" Beyond Benign: Introduction to Toxicology

holey fabrics.

- **Bottoms: Legs, including ankles, must be fully covered.** If your pants are not quite long enough to cover your ankles, you must wear socks that are long enough to cover the exposed skin. Prohibited items include, but are not limited to, shorts, cropped pants (e.g., capris), jeans with holes, and sheer tights.
- **Shoes: Feet, including toes, must be fully covered.** If shoes do not completely cover the feet, socks must be worn. Prohibited items include, but are not limited to, sandals, flip-flops, and other shoes that are not well-secured to the foot.

Goggles (Required)

Students are required to protect their eyes with **splash-proof safety goggles (OSHA-ANSI Z87.1-2010 standard or later)**, which can be purchased at the local bookstores serving MSU.

- Students who wear glasses should be sure to purchase goggles that fit over their glasses.
- Splash-proof safety goggles with **indirect venting** are recommended to reduce fogging.
- **Under no circumstances should you wear contact lenses in the laboratory, even under goggles.** Chemical vapors may dissolve in liquids covering the eye and concentrate behind the lenses. "Soft" contact lenses are especially dangerous as chemicals can dissolve in the lenses themselves and be released over a period of several hours.

Goggles should be worn at all times in the laboratory, unless told otherwise by a laboratory instructor. "At all times" includes periods when you personally are not experimenting; others may be conducting experiments, which still presents a hazard to others in the room. If you need to take off your goggles to adjust them or because they have fogged up, you should step outside the laboratory room to do so.

Gloves (Required)

Nitrile gloves are available in the laboratory in various sizes. You should wear the glove size that best fits your hands—too small, and the gloves are prone to tearing (leaving parts of your hand unprotected); too large, and you lose a great deal of dexterity (making it difficult to do fine tasks). If your size is not available, you can replenish the supply by taking the empty box to the stockroom. If there is no empty box corresponding to the size that you need, please alert your TA.

Wearing gloves at all times is not mandatory. However, for most experiments, such as those involving organic compounds, dyes or acids and bases, you are encouraged to wear them when working with these substances. Used gloves are disposable and shall be thrown to contaminated lab debris bin.

In order to avoid contaminating door handles or other surfaces, do not wear gloves outside the lab. If you must go into the hallway while handling something that requires a gloved hand, keep one hand ungloved, and use that hand to open doors. (Your TA may refer to this as the "one glove" rule.)

Lab Coat (Required)

Required laboratory attire includes a knee-length laboratory coat. Posters and guidelines on proper attire are available in CEM 255 laboratories. Here are some things to keep in mind:

- Be sure that your lab coat fits properly. They come in multiple sizes.
- Snap-front closures are preferred for easy removal in the event of significant contamination.
- Gathered sleeve cuffs are recommended to minimize wrist exposure and sample contamination.
- Lab coats and gloves should only be worn inside the lab.

P: Prepare for Emergencies

Preparation for emergencies involves proactive measures to minimize risk, plan for accidents and spills, and appropriate mediation measures. While your TA will be an invaluable resource in this preparation process, one key learning goal for the Planning Documents is to name the chemicals, the hazards they pose, and identify the major risks that your team will encounter and what necessary PPE will help mitigate that risk. If accidents (inevitably) occur, it is everyone's responsibility to contain the accident.

On the first day of laboratory, your instructor or TA will point out the safety features of the laboratory classroom. These features are summarized below, and each teammate should be able to locate and operate with assistance these safety features. The locations of these safety features are approximated in the **Figure 1** below.

The waste (WH) and supply (SH) hoods are on the north and south ends of the classroom. The safety shower (SS) is just inside the door next to the waste hood and the eye wash stations (EW) are next to the supply hoods and the sinks on the north and south ends of the room. In the event that a chemical splashes near your eyes, use the station before the material runs behind your safety glasses and into your eyes. **You should irrigate your eyes for at least five minutes and notify your laboratory instructor right away.**

In addition to the general waste garbage cans throughout the space, there are glass waste boxes are available for broken glass. Never place broken glass in the general waste can. Be careful to never place your hand inside the glass waste box, and to never overfill this box. If glassware breaks on the counter or floor, consult your TA regarding any chemical contamination of the glass waste before using the broom to scoop up and deposit the glass waste in the glass waste box. Glassware that is broken in the sink should also be handled with care at the TA's instruction. The final source of broken glassware is likely to be inserting glass tubing or thermometers into stoppers; to do so safely, lubricate both the tubing and the stopper hole with water. Wrap the tubing in a towel, grasp it as close to the end being inserted as possible, and push gently, using a twisting motion.

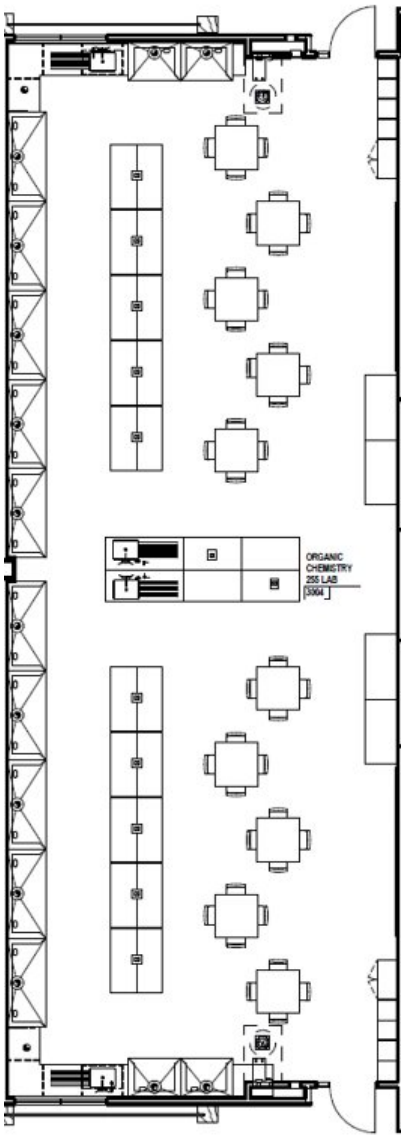


Figure 1. Overview of the CEM 255 laboratory space.

4. HANDLING WASTE

As a student in the chemistry lab, every time you perform an experiment, you will generate waste. Therefore, you have the responsibility to see that the chemicals are identified, handled, and disposed properly. The following information will help you meet this responsibility.

First, you must understand the difference between hazardous and non-hazardous waste:

- **Non-hazardous waste**: Non-hazardous waste does not cause harm to the environment and may be disposed of in the normal waste stream. Non-hazardous waste includes clean disposable pipets, vials, broken glassware, filter paper, gloves, etc. A small number of chemicals are considered non-hazardous. Where you dispose of these items depends on what it is:
    - **Glass disposal (blue broken glassware box)**: Broken glass and other potentially sharp debris must be placed in the blue broken glass box.
    - **Sink**: Some inorganic solutions and soap water are non-hazardous and can be flushed down the drain with excess water. These include some, but not all, solutions of sodium, potassium, magnesium, and calcium salts.
    - **Trash can**: Most of the remaining non-hazardous waste can go in the trash can.
- **Hazardous waste**: Most inorganic compounds and the vast majority of organic compounds are considered hazardous and should not be flushed down the drain or placed in the trash. These materials must be collected and handled in the hazardous waste stream:
    - **Hazardous waste carboys**: Each lab has at least one hazardous waste carboy. They are topped with large funnels in which you can pour chemical waste.
    - **Contaminated lab debris can**: Lab supplies contaminated with hazardous chemicals (e.g., filter paper, paper towels, gloves) can go into contaminated lab debris can.

Note: Sometimes the funnels get clogged or the carboy gets full. In this case, alert your TA and wait to dispose of more waste until the situation is resolved.

In order to comply with government regulations, the contents of hazardous waste containers must be known as accurately as possible. In addition, many wastes are incompatible and should never be mixed together. Mixing

of the wastes may cause a dangerous situation (e.g., an explosion or the formation of poisonous gas). Therefore, the hazardous waste containers in the lab are labeled by course number, experiment number, and name/type of waste. If everyone disposes of their waste in the proper container, the container's contents will be known, and incompatible wastes will not be mixed.

If you are directed to treat the used chemicals you generated to render them non-hazardous, detailed instructions will be given as part of the project. Follow those instructions carefully. With your help, the Department of Chemistry can manage the used chemicals generated in the teaching labs in a safe and environmentally responsible manner.

Note: Unless you are absolutely certain how to dispose of something properly, ask your instructor before you get rid of it!

PART III

PART 3: GLASSWARE & LABORATORY EQUIPMENT

5.

EQUIPMENT AVAILABILITY

In Team Drawers

Item Name	Size	Quantity
Adapter, Claisen	14/10	4
Adapter, thermometer	14/10	4
Adapter, stillhead	14/10	4
Adapter, vacuum	14/10	4
Beaker	30 mL	2
Beaker	50 mL	4
Beaker	150 mL	4
Beaker	250 mL	4
Beaker	600 mL	2
Condenser, air	14/10	4
Condenser, jacketed	14/10	4
Cork ring	3”	2
Dish, crystallizing	100 X 50 mm, 325 mL	2
Filter cone	#2	4
Flask, Erlenmeyer	50 mL	2
Flask, Erlenmeyer	125 mL	4
Flask, Erlenmeyer	250 mL	1
Flask, Filter	125 mL	4

Item Name	Size	Quantity
Flask, Volumetric	100 mL	2
Flask, Round bottom	10 mL	4
Flask, Round bottom	25 mL	4
Forceps		2
Funnel, glass	Various sizes	4
Funnel, Buchner	5.5 cm	4
Graduated cylinder, with base and collar	10 mL	1
Microspatula		2
Test tube	4”	8
Test tube	6”	2
Test tube brush	¾”	1
Test tube holder		1
Scoopula		2
Sponge		1
Stir magnet, micro		2
Stir magnet, large		4
Stirring rod, glass	6”	4
Stopper, polyethylene		4

Item Name	Size	Quantity
Vial, conical, heavy wall	8 mL	4
Watch glass	Any	4

Note: glassware may subject to changes from semester to semester.

6.

CONTAINERS

Containers can be used as reaction vessels or for storing samples.

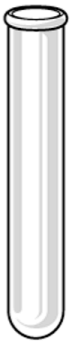
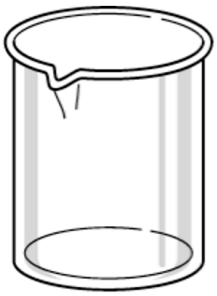
Image of Glassware	Name and Description of Glassware
 The figure was created by Dr. Erin Duffy, CC BY-NC.	Test Tubes There are two main types of test tubes: those made out of regular glass and those made out of Pyrex. Most of the test tubes in your lab drawer will be glass test tubes. These are fine but do not stand up to excessive and rapid heating and cooling as well as Pyrex test tubes do; however, they are cheaper. Test tubes may be used as containers for solids or liquids. They can be used as containers for quick tests for properties such as solubility, effect of heat, etc. Often, they are used to carry out reactions on a small scale and can also be used as centrifuge tubes when a separation of solid from liquid is necessary.
 The figure was created by Dr. Erin Duffy, CC BY-NC.	Beakers Beakers are larger containers than test tubes, available in various sizes (25-500 mL, typically), and usually have a pouring spout. These glass beakers are safe to use for heating, such as for water baths. Be wary of placing a hot piece of glassware (like a beaker) on a room temperature lab bench; thermal shock (causing the glassware to shatter) may occur. Those beakers with graduations on the side can be used as approximate measuring devices for liquids (see Measuring Devices if you need a specific volume).

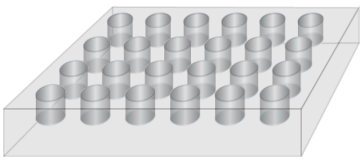
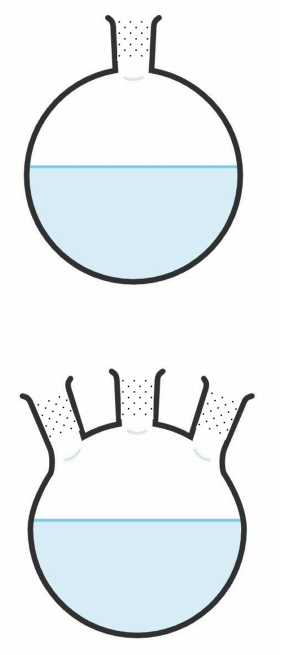
Image of Glassware	Name and Description of Glassware
 The figure was created by Dr. Erin Duffy, CC BY-NC.	Erlenmeyer Flasks Named for its creator, these conical-shaped flasks have a flat bottom and a narrow neck. They are usually glass and are safe for heating solutions. The narrow mouth helps prevent the solvent from escaping and makes the flask particularly useful for containing volatile solvents. These are often used for recrystallizations, titrations, or as reaction vessels. They are not ideal for making solutions or measuring.
 The figure was created by Dr. Erin Duffy, CC BY-NC.	Conical Vial A small, cylindrical glassware with a cylinder bottom, usually used for microscale reactions or storing and handling small volumes of liquid samples. It can stand up on the laboratory bench due to its flat bottom, and the interior of the vial tapers to a point to allow a pipet to extract liquids from the bottom of the vial. This glassware can be used for small-scale reflux, extraction, condensation or distillation reactions.
 The figure was created by Dr. Erin Duffy, CC BY-NC.	Watch Glass This is a very shallow glass bowl often used to allow crystals to dry after they have been filtered. This glass should not be heated to extremes.

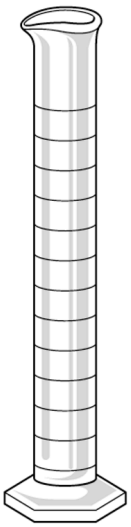
Image of Glassware	Name and Description of Glassware
 Created with Chemix	Round Bottom Flask A round bottom flask, or a boiling flask, is a container with spherical body, a flat bottom, and a narrow neck (or multiple necks). It is typically made of glass and is used for heating liquids and distillations. The round bottom allows for more even heating, while the narrow neck minimizes evaporation and allows for the attachment of various laboratory apparatus such as condensers, stoppers, and thermometers. The shaded area indicates ground glass joints.

7.

MEASURING DEVICES

Measuring Liquids by Volume

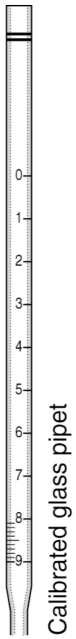
Liquids can be measured either by weight or by volume. It is often more convenient to measure the volume of a liquid and then convert it to mass if the density is known (remember, density = mass/volume, with unit).

Image of Glassware	Name and Description of Glassware
	Graduated Cylinders • Can be used to measure the volume of a liquid. • Range in size from 5.00 mL to several liters. • More accurate than beakers, but less accurate than volumetric glassware (see below). • Fine for most purposes, unless you are doing accurate volumetric measurements such as titrations.

The figure was created by Dr. Erin Duffy, CC BY-NC.

Volumetric Glassware

Volumetric glassware is used when great accuracy is required, for example, during titrations, or when making up standard solutions of known volume.

Image of Glassware	Name and Description of Glassware
	Pipets • Most are made to measure one volume only, most commonly 10.00 mL, 25.00 mL, or 50.00 mL. • Used to measure a specific volume of liquid and will usually have the accuracy engraved on the pipet. • Must be used with some sort of pipet filler for suction (e.g., a squeeze bulb). **Safety note**: Never pipet by mouth. **Other note**: Use the appropriate number of significant figures based on the precision of the measurement.

The figure was created by Dr. Erin Duffy, CC BY-NC.

Measuring Solids and Liquids by Mass

Laboratory Balance

A laboratory balance is used to obtain the mass of various objects. We have two different types of electronic balances in the lab:

- **Analytical Balances** are accurate to 0.0001 g (a tenth of a milligram) and are used to weigh out samples of less than 1 gram as the high accuracy results in low capacity.
- **Precision Balances** are accurate to 0.001 g (a milligram) and are used to weigh out samples over 1 gram.

In this course you will likely use the precision balances. These balances are accurate to 0.001 g and simple to use, but they are delicate and expensive. Please treat them with care and respect and leave them clean. If you

spill anything solid on the balance, use the attached brush to clean it. If you spill a liquid, notify the stockroom personnel immediately.

Created with Chemix (Chemix)

Note: The balances are fitted with draft shields to remove fluctuations of the readings. Please do not remove them.

Use of Laboratory Balances

The video Weighing Using An Analytical Balance provides an overview of using either type of balance (even though it talks only about the analytical type balances). The step-by-step instructions begin at approximately 1:09.

- **Never weigh anything directly on the balance**. Always use a weigh boat, beaker (for highly corrosive compounds), or some other container to contain the chemical.
- **Never pour anything into a container in the balance**. You should always remove the container to add or remove material. This should help keep the balance clean.
- **Never leave the balance dirty**. If you should spill something in the balance, be sure to clean it out. If you are not 100% sure how to do so, ask your instructor.

8.

TRANSFER DEVICES

Transfer devices can be either quantitative or non-quantitative and are used for moving substances from one container to another.

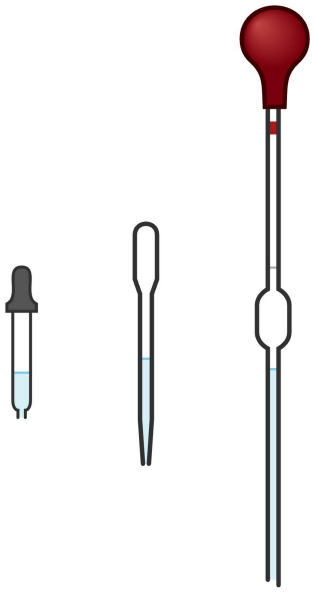
Image of glassware	Name of glassware
 Diagrams made in Chemix	Droppers and Pipets (Pipettes) • Used for transferring volumes of solutions or adding solutions dropwise to a reaction mixture. • Glass pipets (left) usually have a rubber bulb which attaches to the top and acts as the suction device to draw liquid up into the pipet. Glass pipets are non-quantitative and reusable. • Plastic pipets (middle) are non-quantitative and disposable. • Graduated pipets (right), sometimes called Beral pipets, have the bulb built in as an integral part of the pipet. Graduated pipets are quantitative and reusable.

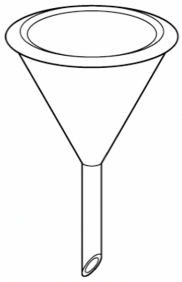
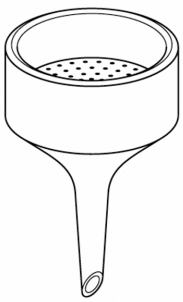
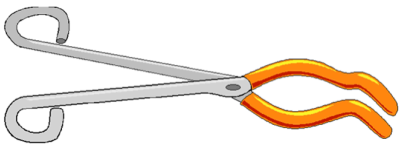
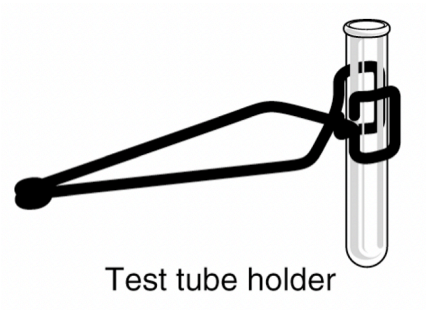
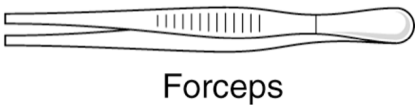
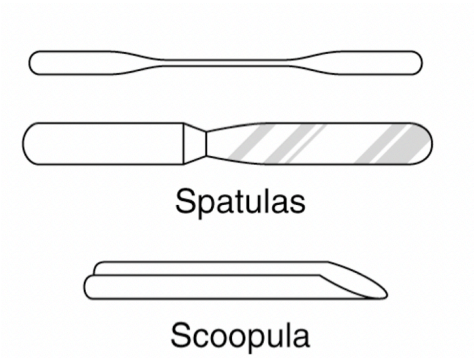
Image of glassware	Name of glassware
  The figure was created by Dr. Erin Duffy, CC BY-NC.	Funnels There are two types of funnels used in this course: **Conical funnels (short-stem funnels)** and **Buchner funnels**. Conical Funnel or Short-Stem Funnel • May be used as a guiding device to help when pouring liquid from one container to another. • With the aid of a filter paper, it can be used as a separation device to separate liquids from solids. • Gravity filtration is used when the **liquid** is desired. Buchner Funnel • Usually used with filtration flask under vacuum suction. • Vacuum filtration is used when the **solid** is desired and can be used for drying a solid.
 The figure was created by Dr. Erin Duffy, CC BY-NC.	Crucible Tongs Can be used to transfer hot objects from one place to another, such as an evaporating dish.

Image of glassware	Name of glassware
 The figure was created by Dr. Erin Duffy, CC BY-NC.	Test-Tube Holder Used to hold a test tube when it is being heated, or to move a hot test tube from one place to another.
 The figure was created by Dr. Erin Duffy, CC BY-NC.	Forceps Used to pick up relatively small objects.
 The figure was created by Dr. Erin Duffy, CC BY-NC.	Spatulas and Scoopulas • Used to transfer solids from one place to another. • Available in different forms and sizes.

9.

SUPPORT DEVICES AND DISTILLATION GLASSWARE

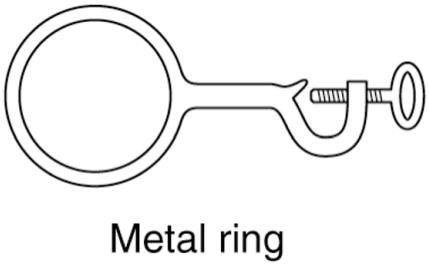
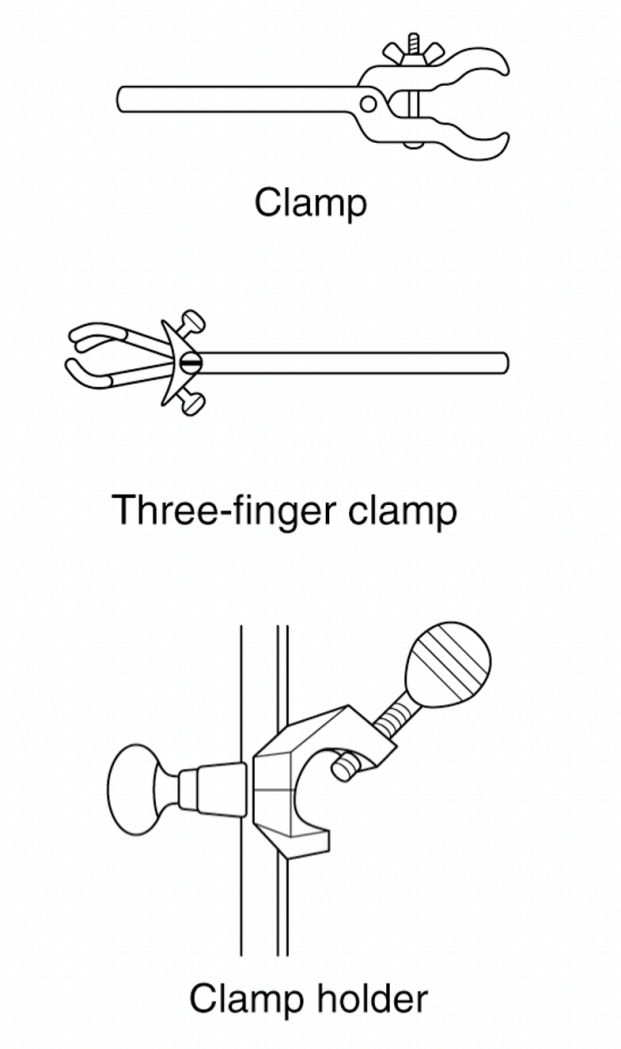
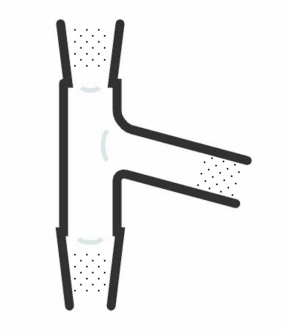
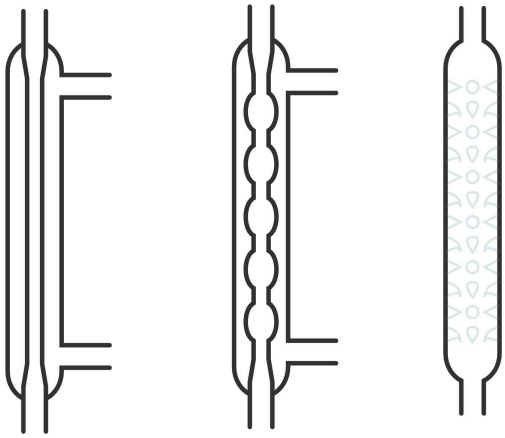
Image of glassware	Name of glassware
 The figure was created by Dr. Erin Duffy, CC BY-NC.	Metal Ring or Iron Ring A metal ring attaches to the stand on your benchtop. It can serve as a support for pipestem triangles and wire gauzes.

Image of glassware	Name of glassware
	Clamps and Clamp Holders • Used to hold equipment steady. • Usually held by a separate clamp holder attached to a stand on the benchtop. • A “universal clamp” is both a clamp and clamp holder in one piece.

The figure was created by Dr. Erin Duffy, CC BY-NC.

Image of glassware	Name of glassware
 Created with Chemix	Distillation Head A distillation head is a glassware used in distillation setups. It is typically a long glass tube with two openings. One opening is tapered to fit into the neck of a round-bottom flask, while the other opening is designed to attach to a condenser. The distillation head is placed between the boiling flask and the condenser in a distillation setup and serves to direct the vapor from the boiling flask to the condenser, where it is cooled and condensed back into liquid form.
 Created with Chemix	Condenser A condenser is a long glass tube with a coiled or straight inner tube that is surrounded by a larger outer tube. The inner tube is typically connected to a water source, such as a water tap or a recirculating water bath, which circulates cold water through the condenser to cool the vapor. As the vapor passes through the condenser, it loses heat and condenses back into liquid form, which collects at the bottom of the condenser and can be collected in a receiving flask. Condensers come in various sizes and shapes, with the most common types being the Liebig condenser, the Graham condenser, and the Allihn condenser, each with its own specific design and application.

PART IV

PART 4: SETTING UP THE REACTION

10.

INITIAL CONSIDERATIONS

Below are some questions and considerations for which you need to account in your team planning documents.

1. Whenever you do a reaction for the first time, always do it on a small scale (100-200 mg) using small-scale glassware.
2. Start with clean, dry glassware for your reaction. If your glassware is dirty, clean it with soap and water and air dry. Most of the stains can be cleaned with soap water. If your glassware is still dirty, use acetone rinse. If your glassware will have water on it, it may (detrimentally) affect the course of the reaction.
3. If any of your reaction components are water- or air-sensitive, you will need TA approval and instruction on carrying out reactions under an inert atmosphere.

A few minutes of forethought can save you many problems later. Your TA and the resources in this manual can help you design the appropriate procedure. Before you begin, think how you will address the following questions:

- **How will you get your reactants into the reaction mixture?**

  Will you mix them in the reaction vessel and then let the reaction occur, or will you need to add one component to the other as the reaction progresses? If a simple initial mixture is all that is required, a very simple reaction setup might be only an appropriately sized reaction vessel.

  If you will need to add a reagent during the reaction, you may need to set up a reaction with a dropping funnel or a syringe to add the second component.

  Weigh out one of the reactants in the clean, dry reaction vessel. The other reagent(s) can be weighed onto a piece of weighing paper or into a glass vial in the case of a liquid.

  Another option for obtaining the correct mass of liquid is to find the density of the liquid and measure out the appropriate volume (recall the formula for calculating density, below).

$$\text{density} = \frac{\text{mass}}{\text{volume}}$$

- **Does the reaction need a solvent?**

Most organic reactions are done in the presence of a solvent so that all the reactants are in the same phase. Because this course introduces principles of green chemistry, always consult the Greener Solvent List to ensure that the most appropriate benign solvent is used. The greenest choice is solvent-free reaction, also referred to as "neat reaction". Neat reactions are often used when the reactants are liquids or solids that can effectively mix and react without the need for a solvent.

- **Will you need to stir the reaction mixture?**

  If so, make sure you set the reaction up on a stirrer, and place a stir bar in the reaction vessel before you start adding reactants.
- **Will you need to heat the reaction mixture?**

  In many cases, you will not know whether you need to heat the reaction until you have monitored it for some time. If in doubt, set up the reaction mixture in a heating bath on a hot/stir plate. This will allow you to heat the reaction mixture more uniformly and in a more controlled fashion than direct contact onto the hot/stir plate.
- **How will you monitor the reaction?**

  Typically, you will use Thin-Layer Chromatography (TLC), Infrared (IR) Spectroscopy and NMR spectroscopy to follow the progress of the reaction. You need to prepare your chosen monitoring system before you begin the reaction.
- **How will you work up the reaction?**

  Make sure you understand how to isolate and purify the product before you begin. Consider Extraction, Distillation, Rotary Evaporation, Recrystallization, Precipitation, Drying, and/or Filtration techniques.

11.

SUGGESTED REACTION SETUPS

There are various ways to set up a reaction, and you need to be sure what you are going to do and what equipment you will need before you start. Each time you do a reaction for the first time, make sure you do it on the microscale using appropriately small-scale glassware. Once the small-scale reaction is successful and the conditions/setup are optimized, you may scale up the reaction.

The following are some suggestions (not an exhaustive list) for reaction setups for various common scenarios:

Set Up for Stirring Only

If you are **sure** that your reaction is not air-sensitive and will not need to be heated, it is probable that all you will need is a reaction flask of appropriate size, a stirrer bar, and a magnetic stirrer.

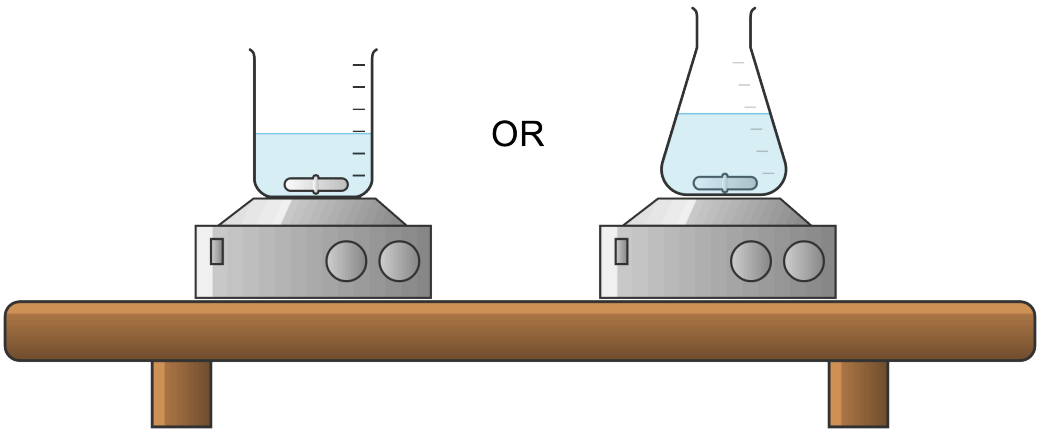


Figure 6. Two examples of a set up for stirring only. Place a flat-bottomed container (beaker, left, or conical flask, right) on a stir plate. Insert a magnetic stir bar of appropriate size into the container. Created with Chemix

Set Up for Heating and Stirring

Set up the reaction in a water bath (or sand bath) on a hot/stir plate. Since you will be heating up the reaction, you will need to attach a reflux condenser to the reaction flask so that no solvent or product vapors will escape.

Do not forget to run water through the condenser in at the bottom and out at the top. Make sure that the only outlet for vapors is through the condenser. If you have more than one neck on your flask, place a stopper in the other necks. Do not block the condenser top. Heating volatile liquids in a closed container is **strictly forbidden**.

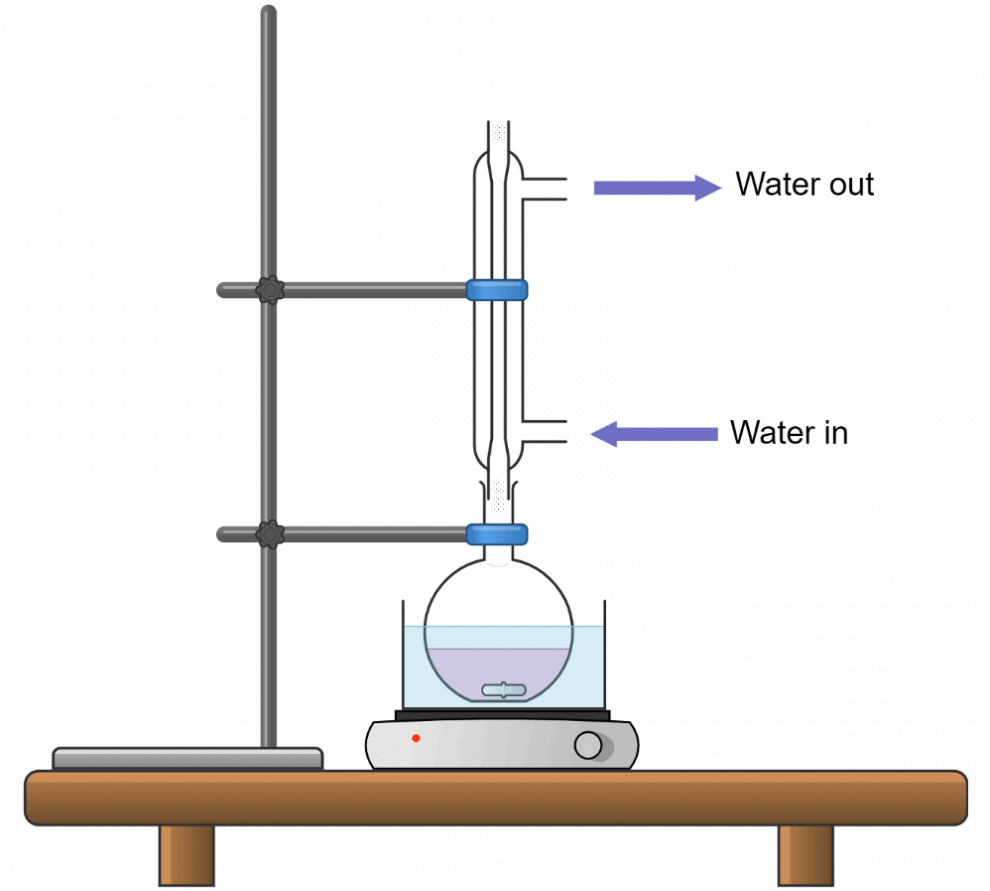


Figure 7. Reflux and stirring setup. Created with Chemix

Set Up for Mildly Water-sensitive Reactions

If you must perform a reaction that is somewhat water-sensitive, you can place a drying tube (a tube filled with a drying agent such as calcium chloride) in the neck of the reaction vessel or on top of the condenser if the reaction is to be heated.

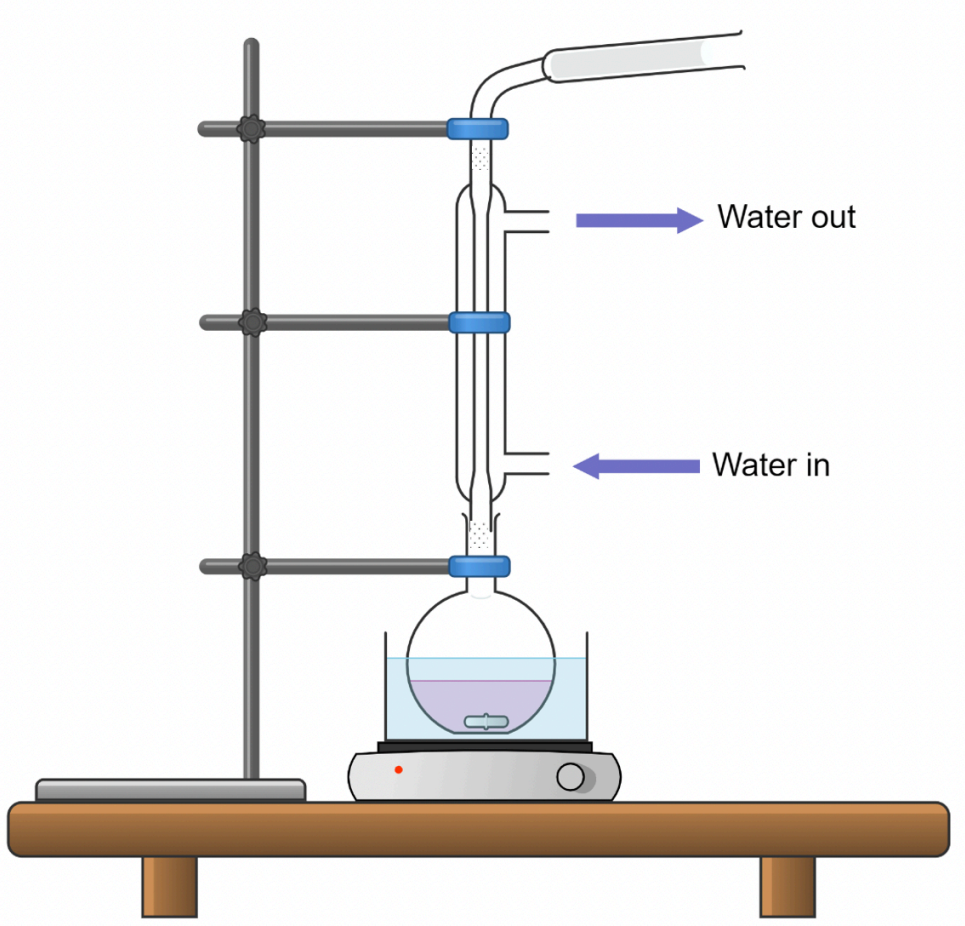


Figure 8. A round-bottom flask within a heating mantle, secured to a ring stand with a clamp on the neck of the round-bottom flask. The condenser is fitted in the mouth of the round-bottom flask and secured to the ring stand. A tube of drying agent caps the other end to prevent evolution of vapors. Note the position of the water intake and output hoses. Created with Chemix

The set up in **Figure 8** uses a condenser adapter and drying tube to ensure that vapors do not escape while minimizing the exposure to water. Keep in mind that all refluxes and reaction should be carried out in the fume hood, and in academic research, reactions that are sensitive may also require a glovebox. The condenser uses running water (note the positions of the water intake and output hoses) to cool any vapors arising from the reflux, and the drying tube fitted into the top of the condenser is filled with a drying agent to exclude water from the reaction apparatus. Be sure to carefully clamp and support each connection to a ring stand.

For reactions requiring a heating source, a heating mantle is a good option if available in an appropriate size for the reaction vessel. Other reactions may make use of water, oil, or sand baths, in which a large glass dish is filled with an appropriate medium that is heated either on a hot plate or with an immersion heater. When

using one of these mediums to heat a reaction, insert a temperature probe or thermometer into that medium to monitor the bath temperature. In this course, we will not use a Bunsen burner. If you do use one, always check to see that there are no flammable liquids in the vicinity, and never leave a Bunsen flame unattended.

In these reflux setups, it is important to use the magnetic stir bar in the reaction flask to stir the reaction in a controlled manner. Stirring (just like use of boiling chips) provides an essential control in the reaction setup: these materials can prevent **bumping**. Bumping is a phenomenon of violent eruption of a liquid, heated at its boiling point.[1] These bubbles contain superheated vapor of the liquid, and the uncontrolled manner by which they are discharged from the liquid phase is not well controlled by the reflux apparatus. Thinking about **P:** Prepare for Emergencies, we can use a stir bar or boiling chip to provide a surface for these bubbles to form in a smaller, steadier manner, thereby preventing bumping.

Set Up for Stirring With Dropwise Addition

Sometimes you will need to carry out a reaction in which one reactant must be added slowly to the reaction mixture. This is often the case with very exothermic reactions or very reactive systems. Sometimes this reactivity does not show up in small-scale reactions, so if you have done a reaction on a small scale and are scaling it up, it is a good idea to add one of the reactants in a dropwise manner. This can be accomplished by using a pippette, a dropper and/or a dropping funnel (also known as the separatory funnel or sep funnel), as illustrated in **Figure 9**.

1. Lehman, J. W. Operational Organic Chemistry

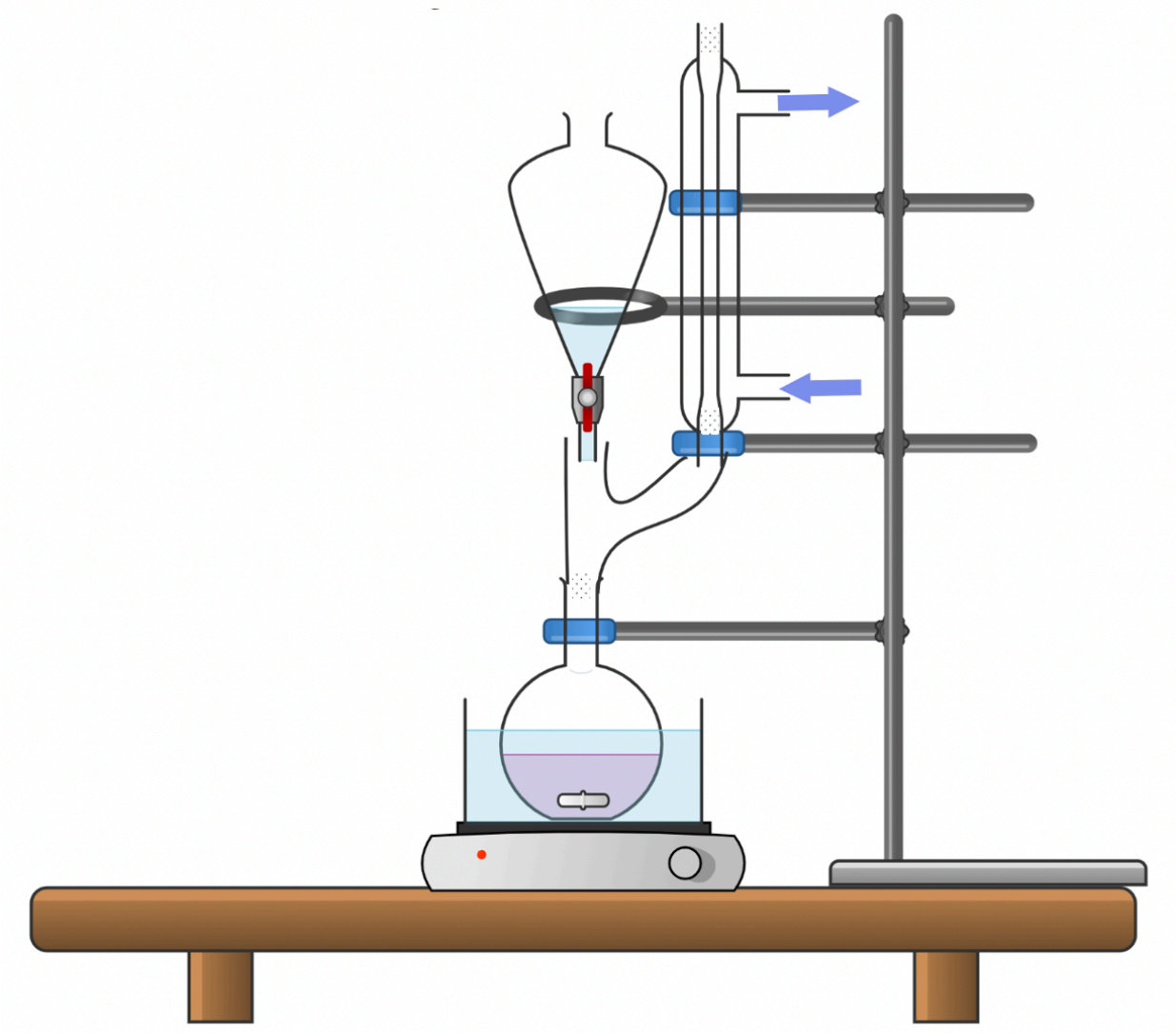


Figure 9. Set up for dropwise addition under reflux. A round-bottom flask (holding pink liquid and a stir bar) is secured to a ring stand and rests in a water bath on top of a hot/stir plate. A Claisen adapter is fitted into the neck (if only single-neck round-bottom flasks are available), and in this configuration the dropping (sep) funnel is situated directly above the reaction. The second neck of the Claisen adapter has a condenser (note the water intake hose is always below the water output hose) has been secured to condense and trap any vapors the reaction may produce. Multiple ring stands might be necessary to ensure all glassware is secured. Created with Chemix

Depending on which round-bottom flask you have available, you may need a Claisen adapter (y-shaped adapter) to fit the dropping funnel and a condenser if the reaction is likely to produce vapors. Remember that all set-ups should be done in the fume hood. Allow the reactant in the dropping funnel to add dropwise into the stirred reaction mixture. If the reaction starts to heat up or give off fumes, stop addition for a while until the reaction

returns to a less vigorous state. If a three-neck round-bottom flask is available, one neck could be used to monitor temperature with a probe or thermometer to monitor the temperature rise of any exothermic reactions.

12.

MONITORING A REACTION

How to Monitor a Reaction by TLC:

Find a Suitable Elution Solvent System for Your Reaction

The TLC chamber needs to be set up and ready to go before you start your reaction. Find a suitable solvent system that will make your starting material run to about $R_f = 0.5$ (or half-way up the plate). If this is the first time you have dealt with this compound, here are a few tips that will make it easier to find a suitable system:

- If the compound is polar (for example, an alcohol), you will probably need a fairly polar solvent system. For nonpolar compounds, a nonpolar solvent system is necessary.
- Do at least two solvent system trials at a time using solvents of different polarities.
- You may need to use mixed solvent systems to get a good R_f—many compounds can be separated by using some mixture of ethyl acetate (polar) and toluene (nonpolar), for instance. The solvents you choose in a binary or multi-solvent system must be miscible.
- Remember, it doesn't matter what you dissolve the starting material in—it will evaporate (unless you use water) so you should use the solvent with the lowest boiling point possible to dissolve the analyte. Dissolving solvent and elution solvent can be the same or different solvent (systems).

Remove a Sample

You can do this by taking a small aliquot from the reaction vessel with a capillary tube. You do not need much; a dip and drop is plenty.

Prepare the Sample for TLC

Make sure that you are sampling only the organic components of your reaction mixture. If your reaction is in an organic solvent with no inorganic materials present, you can spot the reaction mixture directly onto the TLC plate.

If, however, you have water, some other very polar solvent with a high boiling point, or inorganic materials present, you will perform a microscale work-up of the small sample you have taken.

Provide Comparison Samples on the TLC Plate

Thin layer chromatograms (TLCs) are difficult to replicate for beginners. The R_f value and TLC result of a particular analyte can vary considerably depending on the concentration of the spotting solution, the size of the spots, the temperature, the vapor pressure inside the developing chamber, and so on.

This lends itself to a more qualitative comparison in which you run the sample side-by-side with the starting material on the same plate. It is a good idea to always spot the starting material on the same place on each plate, such as on the left, so that if you were to forget to label the compounds you would still know what each spot was.

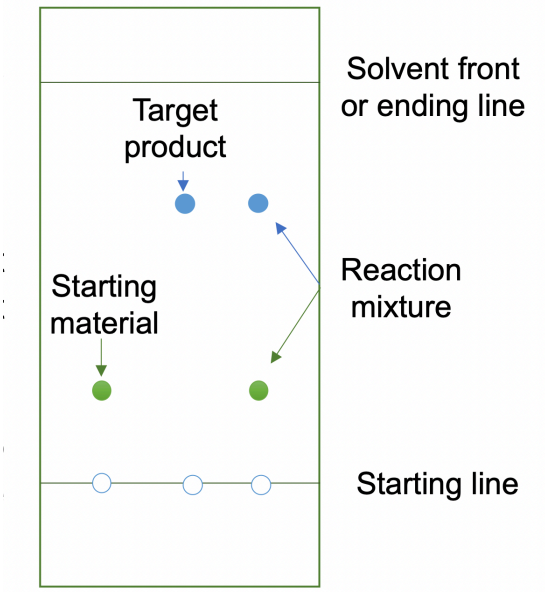


Figure 10. TLC plate example

It is also a good idea to spot the starting material, target product and reaction mixture on the same TLC plate for verification of each compound and monitoring reaction progress.

The starting line you draw must be above the solvent level in the TLC developing chamber. After you put the TLC plates into the TLC chamber with the bottom of the plate in contact with the elution solvent, you will see the solvent front rising along the plate due to capillary effect. Stop the TLC analysis when the solvent front is approximately 1 cm away from the end of the plate, or when you predict there is good separation already.

As you follow the reaction by TLC (roughly 15-minute intervals), you should see a new spot appear as the product is formed and the relative amount of the starting material should decrease. If more than one product is possible, yet you only desire one of the products, you may be able to stop the reaction when you see the formation of the secondary product.

Not all compounds will show up on TLC under UV-visible light. More information on TLC can be found in the next section.

13.

REACTION WORKUP

Before you begin any reaction, you need to think about how you will get product out at the end. The reaction mixture probably contains solvent, product, (hopefully) minimal amounts of by-products, and probably some unreacted starting material. The process of isolating the crude product is called "working up the reaction", and depending on the nature of the reaction and the product, this can be done by various methods.

If you have never done the reaction before, you need to do some microscale (test-tube sized) tests of how to work up your reaction. Consult the Greener Solvent List to test acceptable solvents.

- The first thing to try is pouring the reaction mixture onto ice water; if you are lucky, unreacted starting materials will be dissolved while your product will crystallize. When crystallization is complete, you can filter off your product. **Note**: You need to wait until the aqueous solution does not look cloudy before you filter your product; otherwise, you will lose product that is still in the process of crystallizing.
  If after ~5 minutes no crystals have formed, it is improbable that any will form during the lab period, so you need to move on to the next test.
  Also, in many reactions this method will not work for the following reasons:
  - If your product is a liquid at 0°C, it will (obviously) not crystallize.
  - If your reaction has formed by-products, these may prevent crystallization.
  - Your solvent may not be water soluble, and thus your product may be in the organic layer of the reaction mixture.

- If no crystallization is seen on pouring into water, try extracting the mixture with organic solvents. This will separate the desired product (which is in the organic layer) from the water (also called aqueous layer). When you do the full scale (macroscale) work-up, be sure to use two or **three small portions** of solvent, rather than one large portion. For example, if you have about 100 mL of reaction mixture and water, use three 15 mL portions of organic solvent to extract the product.
  As each organic layer is separated from the aqueous layer, combine it with the others in an Erlenmeyer flask or a beaker. Dry the combined organic layers by adding an anhydrous salt (a drying agent), such as calcium chloride or sodium sulfate. The amount of solid should be enough to cover the bottom of the flask; if the solid seems lumpy and wet, add some more salt until it is dry and powdery.
  Swirl the flask and filter off the drying agent through a gravity filtration apparatus into an already-weighed

round-bottom flask. Evaporate the solvent either by blowing compressed air into the flask or on a rotary evaporator if one is available. Weigh the flask and observe the product.

- At this point, it is a good idea to check the purity of your product by TLC. If the product seems pure (one spot), you can obtain an FTIR spectrum and try to crystallize it.

PART V

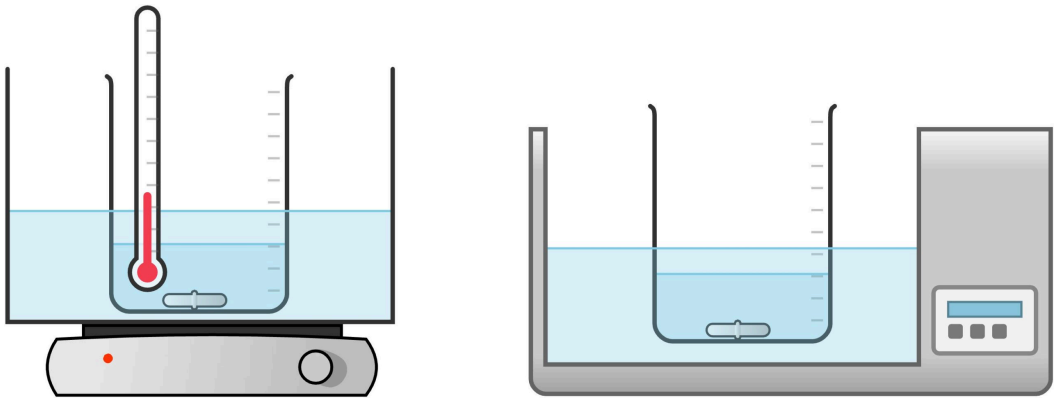
PART 5: ORGANIC LABORATORY TECHNIQUES

14.

HEATING AND COOLING

Heating Bath

It is often necessary to heat reactions or solutions in a controlled manner so that the temperature does not exceed a certain value. For example, many reaction mixtures might decompose when heated to very high temperatures. One way to avoid this problem is to use a water bath. As noted in the Suggested Reaction Setups, you should consider a heating mantle (if available) or make use of water, oil, or sand baths, in which a large glass dish is filled with an appropriate medium that is heated either on a hot plate or with an immersion heater. These methods allow for controlled heating and an easy way to monitor the temperature.



Created with Chemix

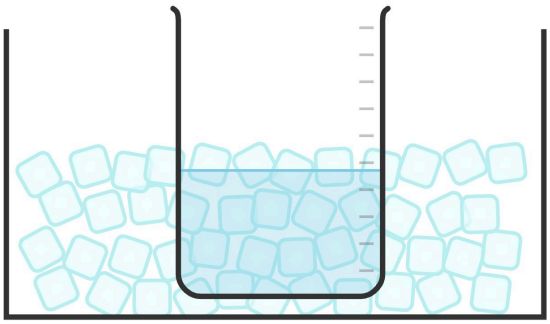
Cooling Bath

A cooling bath is used to cool a reaction or system under investigation.

Water-and-ice mixtures can reach temperatures as low as 0 °C.

For temperatures lower than 0 °C, a mixture of ice, water, and salt can be used to achieve a temperature of about -10 °C.

It is unlikely that you will need to reach a temperature even lower than -10 °C in the organic chemistry lab, but it is possible to do so using dry ice (solid carbon dioxide) in organic solvents.



Created with Chemix

Measuring Temperature

Thermometer

For most purposes, the classic thermometer is suitable for measuring temperature. Scientific thermometers typically measure temperature using the Celsius (°C) scale. Our course will make use of Vernier Temperature Probes instead.

Vernier Temperature Probe

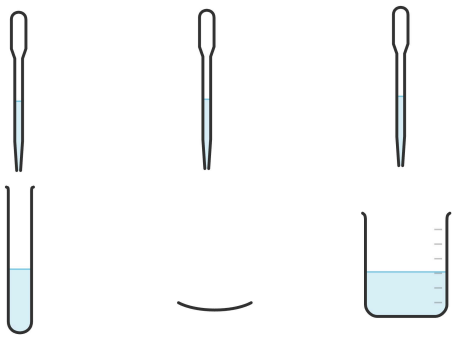
The stockroom has temperature probes that connect to your computer via USB. This allows you to monitor and record temperature in Vernier's LoggerPro software. This is especially useful if you want to record temperatures over time (i.e., make a temperature vs. time plot). By recording the temperature this way, it is easy to determine when a maximum or minimum temperature was reached, for example, during a chemical reaction or when a temperature stabilized for a certain amount of time.

Note: The majority of this chapter has been adapted from the CEM 161/162 manual: Cooper, M. M. *et. al. Cooperative Chemistry for Michigan State General Chemistry Laboratories*, **2019**.

15.

SMALL-SCALE CHEMISTRY

When performing an experiment for the first time, it is always a good idea to do the experiment on a small scale. This will give you some idea of what to expect, and should anything unforeseen happen, i.e., an accident, it is much easier to contain small amounts of chemicals. Small-scale experiments are safer, produce less waste, and are therefore preferred if possible. Obviously, you will not be able to do all your work on the microscale, since it is very difficult to do quantitative measurements on a small scale. However, microscale work is very useful when you want to see what will happen, e.g., to see if a precipitate will form, or a gas be evolved, etc.



Diagrams made in Chemix.

The pieces of equipment used for most microscale work are the test tube, watch glass or beaker and the plastic pipettes discussed in the laboratory equipment section. For semi-quantitative purposes the pipettes can be used to deliver a constant volume by marking a point on the stem of the pipette. If the pipette is then filled to this level each time, even though you do not know what the volume contained in the stem is, you will be able to control the relative volume of each solution that you mix (e.g., 2 pipette volumes of NaOH per 1 pipette volume of H_2O).

One drawback to using microscale techniques such as these is that it is difficult to heat the test solutions. If you find that this will be necessary, you will have to scale up your reaction perhaps to a test tube scale where you will be dealing with 1–2 mL instead of drops of solution. Material can be heated in a test in a water bath. You should always heat test tubes whilst pointing the opening away from yourself or any other person working in the lab.

Small-scale chemistry is also considered more green or environmental friendly because it reduces waste, improves energy efficiency, and is more cost-effective. Working on a smaller scale often means working with less hazardous materials and lower quantities, which can reduce the risk of accidents and exposure to hazardous substances.

Note: The majority of this chapter has been adapted from the CEM 161/162 manual: Cooper, M. M. *et. al. Cooperative Chemistry for Michigan State General Chemistry Laboratories*, **2019**.

16.

CHARACTERIZATION AND IDENTIFICATION

Thin-Layer Chromatography (TLC)

Chromatography is a technique that is used to separate mixtures. To separate large quantities, you would perform column chromatography. For qualitative testing of small amounts (a few drops), you can use TLC. All chromatographic techniques share the same underlying principles.

One component is the **stationary phase**, composed of substances ranging from cellulose (in paper chromatography) to silica gel or alumina layered onto of a glass or plastic plate (in TLC) or finely powdered silica or alumina packed into a glass column (in column chromatography), to any number of complex mediums used in biological separations. The stationary phase is usually fairly polar and experiences strong attractive forces with polar analytes. The analytes to be separated become adsorbed (i.e., stuck) onto the stationary phase and stay there until dislodged by some external force.

Once the analyte has been adsorbed, the stationary phase is brought into contact with the **mobile phase**, usually a liquid, although in some instruments the mobile phase can be gaseous. The mobile phase is drawn along the stationary phase by capillary action; when the leading edge of the mobile phase (called the **solvent front**) reaches the adsorbed analyte, the analyte is preferentially attracted to either the stationary or mobile phase, depending on the polarity of the analyte. Although the common adage is that "like" solvents dissolve "like" solutes, most analytes (whether they are ionic or molecular in nature) experience some degree of attractive forces to both the stationary and mobile phases. An equilibrium is established for the analyte between the two phases, as shown in Equation 1.

$$D = \frac{[S_{\text{org}}]_{\text{total}}}{[S_{\text{aq}}]_{\text{total}}} = K_{\text{D}} = \frac{[S_{\text{org}}]}{[S_{\text{aq}}]}$$

K_D is the partition coefficient, while D is the distribution ratio. S_{org} and S_{aq} represent solubility of a compound in organic and aqueous phases. This equation can be used to calculate for liquid-liquid extraction with no secondary reactions.

As the solvent front moves up the stationary phase, developing solvent that is "fresh" passes over the spot of analyte, and new equilibria are established. At the same time, any of the analyte that has dissolved in the mobile phase encounters fresh stationary phase, and new equilibria are established. The total effect of all these equilibria is that the movement of the substance depends on the nature of its relative attractions with the substances of

the mobile and stationary phases. We characterize this movement in terms of **retention factor** (R_f), defined in Equation 2.

$$R_f = \frac{\text{distance traveled by spot}}{\text{distance traveled by solvent}}$$

R_f values can be as high as 1.0 if the analyte moves with the solvent front and as low as 0 if the substance does not move at all. The R_f values are reproducible for a particular analyte and solvent system if the experimental conditions are closely controlled. One important variable is the composition of the developing solvent (the mobile phase). If one of the solvent components is volatile (has a low boiling point), it is possible that evaporation will change the percent composition of the solvent as you develop the chromatogram. You can avoid this complication by developing the chromatogram within a closed container so the air inside the container is saturated with solvent vapor.

A sample containing two or more components can be separated (or resolved) by choosing a solvent system for which the sample components have distinctly different R_f values.

Procedure for TLC:

- Prepare a developing chamber (beaker with watch glass as cover can be used) by placing about 0.5 cm depth of your chosen solvent system. It is recommended to line the beaker with filter paper or a paper towel, which will allow the vapor and liquid to equilibrate faster. In order to have reproducible TLC results, you need to have the solvent tank saturated with the vapor of the developing solvent(s). Let the solvent equilibrate for 5-10 minutes.
- Obtain a piece of chromatography paper or a TLC plate. If you use paper, you can fold it in half lengthwise so it will stand up in the tank. Draw a line lightly **with pencil** 1 cm from the bottom of the paper. If the starting line fell below solvents, sample dots will dissolve in solvents rather than eluting up the plate at designated spot.
- Make a dilute solution of the analytes of interest and place spots of each solution on the pencil line. For instance, a capillary tube can be used as a TLC spotter. You can place spots from several analytes (or standards, if you are running a comparison) on this line, as shown in **Figure 11**.

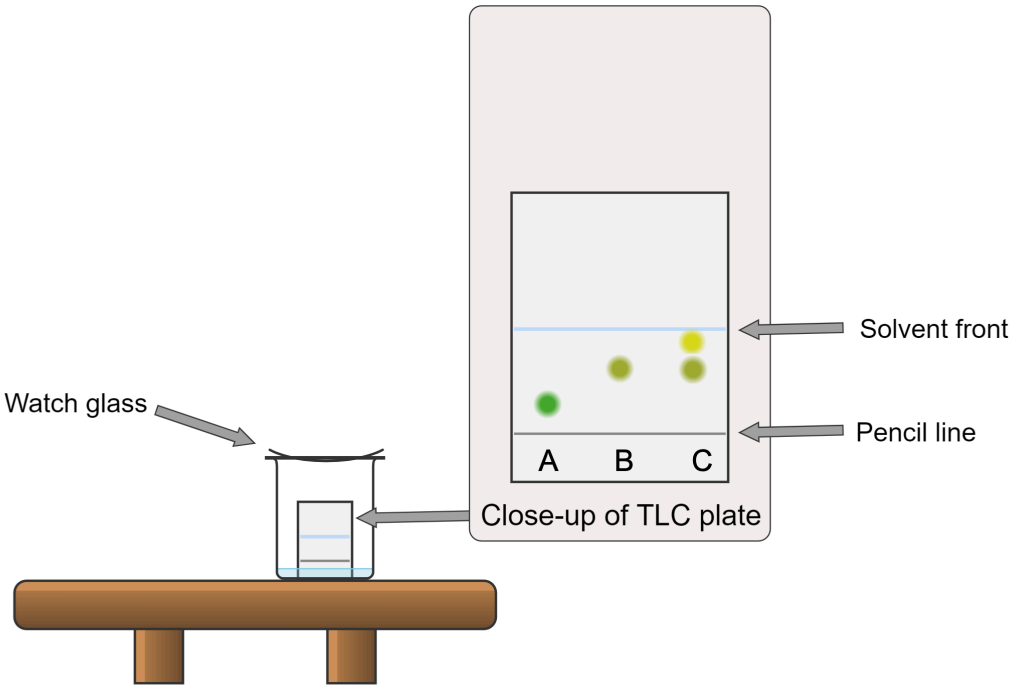


Figure 11. TLC development chamber consists of a beaker or other wide-necked container with a watch glass on top. The developing solvent sits in the bottom of the chamber. Created with Chemix

Note: The spot must be small; otherwise, the stationary phase will be overloaded, and the plate will be smeared. The best way to establish this is by trial-and-error. The pencil line must be above the solvent level in the TLC developing chamber. Use the UV lamp to do a preliminary check to see if you have any compound on the plate.

On the plate shown in **Figure 11**, three samples are spotted. Sample A (green dot) might be a reaction mixture, and sample B (hazel dot) might be a dilute solution of two starting materials. Sample C contains unreacted sample B and a new compound (indicated by a yellow dot) but showed no presence of sample A. The dots shall be sufficiently spaced apart for better separation and identification.

- Place the chromatography plate in the developing chamber, cover the chamber, and allow the solvent to rise up until it is about 1 cm from the top of the paper.
- Remove the paper and mark the level where the solvent has reached with a pencil. In your lab notebook, draw a picture of the resulting separation if spots are visible and note the solvent system used. If nothing is visible on the plate, several methods can be used to visualize the spots. An iodine chamber will make most organic species appear as brown spots. There are a number of specialized visualizing agents that can be

sprayed on the plate and heated. If the TLC plates have a fluorescent indicator in them, a UV lamp can be used and the plates will glow under the light, causing the spots to appear dark where they cover the fluorescent indicator. If you use a UV lamp, be sure not to look directly into the light. Your TA will help you determine the most appropriate method for your needs.

- Calculate the R_f of each spot as shown in Equation 2, reproduced below.

$$R_f = \frac{\text{distance traveled by spot}}{\text{distance traveled by solvent}}$$

- Repeat the process with another solvent, and note the difference in the R_f values of the spots.
- Repeat the process until you find the appropriate solvent.

TLC is a versatile and widely used technique in organic chemistry laboratories for identification of chemicals. TLC is particularly useful for monitoring reaction progress and completion. As a reaction progresses, the composition of the reaction mixture changes, leading to changes in the spots observed on the TLC plate. By comparing the spots from the reaction mixture to those of the starting materials and products, one can assess the extent of the reaction and the formation of intermediate products or byproducts.

TLC technique also has its limitations. It is not a quantitative tool and does not allow for the isolation of mixtures. TLC may not be able to separate compounds with similar size, structure, and polarity. Additionally, not all chemicals' spots show up under UV lamp, and some compounds may not be suitable for analysis by TLC due to their chemical properties or interactions with the stationary phase or mobile phase. Furthermore, TLC may not be as sensitive as other analytical techniques, such as mass spectrometry or UV-visible spectroscopy, which can limit its ability to detect low concentrations of compounds.

Remember to always follow safety precautions and proper procedures when handling chemicals and equipment.

Note: Part of this chapter has been adapted from the CEM 161/162 manual: Cooper, M. M. *et. al. Cooperative Chemistry for Michigan State General Chemistry Laboratories*, **2019**.

17.

MELTING POINT DETERMINATION

One measurement that can qualitatively determine purity is to take the relative melting point of your analyte (product, unknown, etc.) and compare to the melting point of a standard or a known melting point from the literature. Melting point of a compound will be a range. Presence of impurities will lower and broaden the melting point range. The narrower the experimental melting point range is, the purer the compound is.

To use the Melt Station with Logger Pro 3 Software[1]

1. Vernier. Melt Station manual (PDF)

Figure 12. The Vernier Melt Station (sometimes referred to as a "meltemp"). The figure was created by Gustavo Casanova, CC BY-NC.

Load a small amount of the solid substance into a capillary tube. This 3-minute video: Loading capillary tubes with benzoic and mandelic acids[2] demonstrates how to load a finely-ground solid into a glass capillary tube. Never load capillary tubes directly from the stock bottles to avoid introducing contamination.

Ensure that the power supply to the Melt Station, pictured in **Figure 12**, is off and connect the power supply. Connect the Melt Station sensor via Bluetooth or via cable to a computer that has installed Logger Pro

2. Gilchrist, M. "Loading capillary tubes with benzoic and mandelic acids" Published 16 July 2020.

3 software. Launch Logger Pro 3. A live temperature reading will display even though the Melt Station control knob is in the "off" position.

Carefully place the filled capillary tube into one of the three slots of the aluminum heating block within the Melt Station. Tilt the station toward you slightly for a better view of the slots. Once placed, adjust the tilt of the station to best position the viewing chamber (window at the top of the station) to observe the melting process.

In general, the station will collect 100 readings over a 20-minute period which should be sufficient to melt most substances in the course. Begin collection by selecting "Collect" in the Logger Pro 3 interface and turning the control knob on the station to "Rapid Heat" region. A red LED light will turn on to alert you that the unit is heating. Rapid heat indicates a rate of > 10°C per minute.

Note: When the red LED light is on or as the unit cools down, take care not to touch the hot Melt Station or to allow combustible materials to get close (paper, flammable cloth, chemicals).

When the sample approaches the temperature within 10°C of the literature value for the substance, slow the heating rate to 1.5°C/min. This slower rate allows the best precision in determining the point that the solid has completely liquified.

While the Melt Station is collecting data to populate the temperature vs time table, you should carefully observe your sample through the viewing chamber. At the first indication of the solid melting (it may appear "melted" or "liquified" around the edges, for example), click "Mark" (or press the D on the keyboard) to indicate to Logger Pro 3 that the melting has begun and to mark the temperature on your graph. Mark the temperature again when all the solid completely melted. The two temperatures you recorded are the melting point range of the solid sample.

18.

EXTRACTION AND DRYING

There are two different extractions: solid-liquid extraction and liquid-liquid extractions. Both techniques use solubility differences to transfer the target compound from one phase to another.

Solid-Liquid Extraction

Solid-liquid extraction is a method used to separate a compound or compounds from a solid matrix using a solvent. The solute is extracted from solid phase to the carrier liquid phase due to good solubility. The solid impurities residue is then removed by filtration. Carrier liquid will be removed by rotary evaporation (rotovap) or distillation.

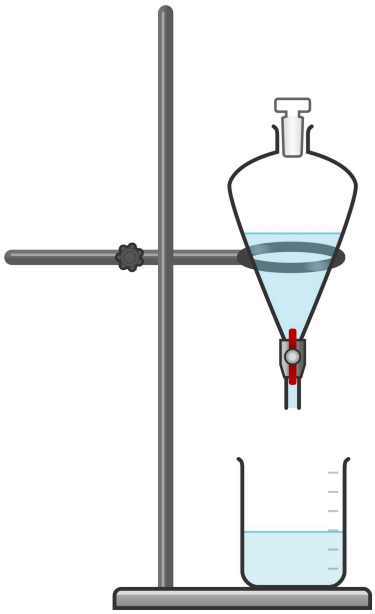
Solid-liquid extraction is commonly used in various industries, including pharmaceuticals, food processing, and environmental analysis, to extract and purify compounds from solid materials.

Liquid-Liquid Extraction

Liquid-liquid extraction is a method used to separate compounds based on their relative solubilities in two immiscible liquids. Generally, you will be doing an extraction to separate the organic product (in an organic solvent such as ether or ethyl acetate) from an aqueous layer. Depending on your product, you may want to retain either the aqueous layer or the organic layer. Use density to navigate which layer is which.

Large Scale

To separate volumes larger than a few milliliters, you will need a separatory funnel. This piece of equipment consists of a pear-shaped container with a stopcock at one end and a ground-glass opening at the other.

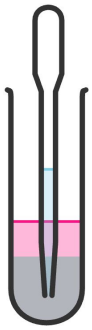


Diagrams made in Chemix

To use the separatory funnel (often referred to as just "sep" funnel) to perform an extraction or separation, place the two immiscible liquids you wish to separate in the sep funnel. Put the ground-glass stopper in the open end, and **ensure that the stopcock is closed** (the knob is horizontal, i.e., parallel to the floor); vigorously shake the funnel while firmly holding the glass stopper in place. Pressure will build up quickly if the solvent is volatile. **Making sure to point the sep funnel away from yourself and anyone else**. Vent the funnel by pointing the tip up and away from you and opening the stopcock. You should hear a "whoosh" or a release of pressure. Close the stopcock and repeat this process several times. The funnel can now be placed in an iron ring on a ring stand. **Remove the ground glass stopper before you open the stopcock.** If you forget and open the stopcock while the stopper is still in the funnel, the fluid will be trapped in the sep funnel because you have formed a vacuum. If this happens, close the stopcock and remove the ground-glass stopper. Now open the stopcock and allow the bottom layer to drain out into an appropriate container. Slow the rate of flow of the fluid as the interface of the two layers approaches the stopcock. At this point, you might achieve best results by draining the bottom layer dropwise. Close the stopcock when the last drop of the bottom layer is in the stopcock. You can then pour the top layer out of the open end of the sep funnel, so it will not be contaminated by the lower layer.

Usually, it will be obvious which layer is the organic layer and which is the aqueous layer. If you get confused, try adding a drop or two of water to one layer and see which layer the added water joins. In general, if the organic layer is in ether or ethyl acetate, it will be the upper layer (and the aqueous will be the lower layer). If the organic solvent is denser, such as dichloromethane or chloroform, the organic layer will be the bottom layer.

Small Scale



Created with Chemix

If the volume of solvent that you want to extract is only a few milliliters, the sep funnel is too big to be efficient. In this case, you should use microscale extraction vials.

Place the mixture that you are extracting into a conical vial; cap and shake to assure thorough mixing. Uncap the vial and let it stand to allow sufficient separation of the layers.

If the layer you are extracting is on the top, draw the lower layer into a Pasteur pipette; the conical nature of the vial will allow you to make a good separation. Pipette the layer you have drawn into another container.

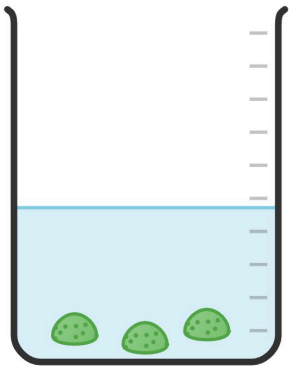
Alternatively, if the layer you are extracting is at the bottom, take all of the mixture up into the Pasteur pipette and gently tap the pipette to make sure the layers are well separated within the pipette. Then pipette the bottom layer (the desired layer in this case) into one clean vial and the top layer into another vial. Add more ether to the bottom layer and repeat at least twice more, combining the organic extracts into the other vial.

Drying Organic Extracts

When you have extracted an aqueous solution with an organic solvent, you need to remove any water that has dissolved in the organic layer or that may have been accidentally included into the ether extracts.

The easiest way to dry an organic solvent is to treat it with an anhydrous inorganic salt, such as magnesium sulfate, sodium sulfate, calcium chloride, etc. These salts must be anhydrous or else the water in the organic layer will not be absorbed.

The amount of drying agent needed depends on the amount of water residue in the organic mixture. For example, for 50 mL of solvent, try a couple spatulas of drying agent first. If the drying agent seems wet and clumpy, add some more until all the moisture is absorbed and some of the drying agent is freely moving. Allow it to sit for a few minutes and then filter off the drying agent.



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Note: The majority of this chapter has been adapted from the CEM 161/162 manual: Cooper, M. M. *et. al. Cooperative Chemistry for Michigan State General Chemistry Laboratories*, **2019**.

19.

FILTRATION

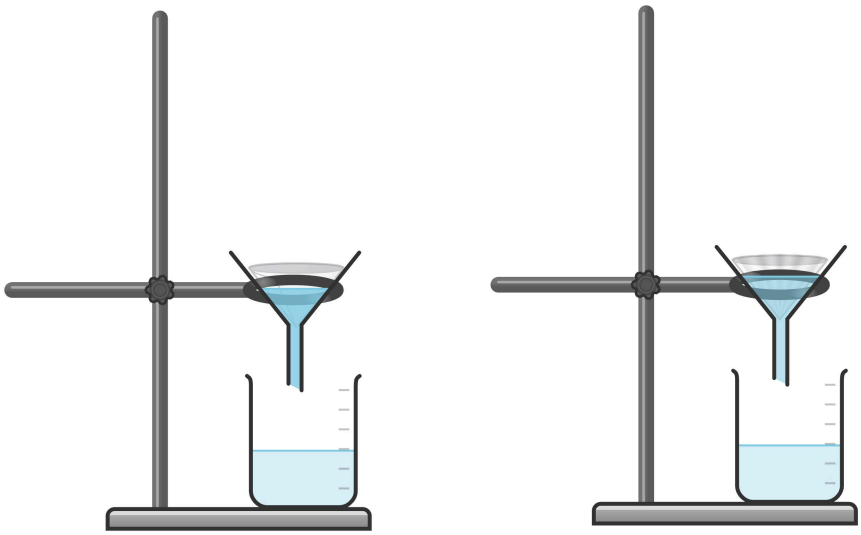
Filtration is a technique used for **separation of mixtures of solids and liquids**. The type of filtration apparatus used depends upon whether you want the solid you are filtering or the liquid (called the "filtrate") from the mixture.

- If the desired compound is the filtrate, a simple gravity filtration is the best method.
- If the desired compound is the solid, a vacuum filtration may recover more solid.

Find a video tutorial at Filtration | MIT Digital Lab Techniques Manual (Gravity filtration begins at 5:06).

Gravity Filtration

In gravity filtration, a conical funnel is fitted with a piece of filter paper that has been folded. In simple fold, shown below on the left, the filter paper is folded in half to form a semicircle. The folded edge is then folded again to form a quarter circle. "Fluted fold", shown below on the right, is also used for rapid filtration with maximized exposed surface area of the paper.



A gravity filtration setup with semicircle fold and fluted fold. Diagrams made in Chemix

Wet the filter paper with the solvent you are using; for example, if the mixture is a solid and water, use water to wet the paper inside of the funnel. Pour the mixture of solid and liquid through the filter paper and funnel and allow it to filter by gravity. The solid remains as the liquid passes through the filter paper. Keep adding the mixture to the funnel as the level drops. You may find it necessary to swirl the mixture as you pour so that the solid does not get left behind in the bottom of the flask.

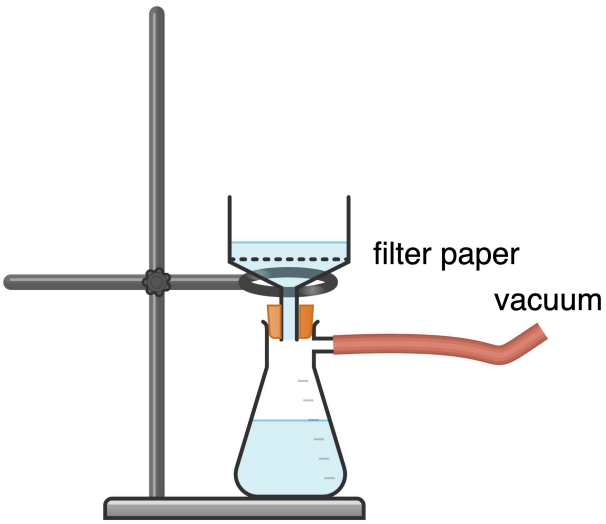
There are different grades of filter paper, so if the solid is finely divided, you may have to use a different grade of filter paper. Using the wrong grade of filter paper may result in poor or no separation, which may be visible from the appearance or testing of the filtrate. In general, the finer the filter paper, the slower the filtration.

When you have filtered all the material, you may find that there is some solid left in the original flask. Pour some of the filtrate back into the original flask with the remaining solid, swirl, and re-filter. Repeat this process until you have all the solid in the filter funnel.

Then, wash the solid by pouring a small amount (<5 mL) of fresh cold solvent through the funnel. Repeat the washing. (**Note**: for washing solids, two small portions are more effective than one large portion.) The filtrate is now ready for further use. If you also need the solid, you should allow it to dry on the filter paper before removing it to be weighed.

Vacuum Filtration

This type of filtration is usually performed when it is primarily the solid that is the desired compound. Take a Buchner funnel (or Hirsch funnel) and a Buchner flask (the "vacuum filtration" flask that has a glass side arm) and attach the thick-walled rubber hoses to the side arm of the flask as well as the aspirator or vacuum.



A vacuum filtration setup. Diagrams made in Chemix

Once the flask is **secured to a ring stand** and attached to the aspirator via the rubber hose, place the funnel (along with the neoprene adaptor) into the neck of the flask. Place an appropriately sized, **pre-weighed** piece of filter paper over the holes in the Buchner funnel; the filter paper in this method should not be folded and should fit the size of the funnel such that all holes are covered. Wet the filter paper with the solvent you are using and turn the aspirator on full (partially turning on the water could cause water from the aspirator to get sucked into the Buchner flask).

While occasionally swirling the vessel with your mixture, slowly pour into the funnel, aiming for the center of the filter paper, and allow the mixture to be filtered by the action of the vacuum. Wash out any solid that remains in the original flask using filtrate, as described in the previous section. When all the material has been filtered, wash the solid (sometimes referred to as the "filter cake") as previously described. Allow the solid to dry on the filter paper, and then find its mass.

Note: Before turning off the aspirator, disconnect the vacuum hose. Otherwise, water will suck back into your flask.

The majority of this chapter has been adapted from the CEM 161/162 manual: Cooper, M. M. *et. al. Cooperative Chemistry for Michigan State General Chemistry Laboratories*, **2019**.

20.

PURIFICATION

Once you have isolated your product, you will need to purify it. There are several ways to purify an organic compound.

Recrystallization

If your product is crystalline (or can be induced to crystallize), recrystallization is the best way to purify the compound.

Crystallization

Crystallization is the process in which a compound becomes crystalline. Often the initial product isolated from a reaction work-up is not crystalline, even though you might assume it would be. The crude product from a reaction often appears as an oil due to the presence of impurities or small amounts of solvent.

The process of crystallization is actually rather unfavorable from a thermodynamic point of view, and there are several things that you can do to enhance the crystallization process:

1. Add a small amount of solvent (a few drops) to the oil and scratch the glass container to provide nuclei on which crystallization can occur. Adding solvent allows the compound to go into solution and then crystallize.
2. Cool the mixture; crystallization will be more likely to occur from a cold mixture than a warm one.
3. If these methods do not yield crystals, remove some of the solvent or seal the container and leave it till next session; some compounds take time to crystallize.

If all your efforts to crystallize the mixture fail, you may have to resort to purifying the compound by chromatography.

Steps for Recrystallization

Recrystallization is a technique for purifying a crystalline solid. The technique involves dissolving the solid in a minimum amount of **hot** solvent. The solution is then allowed to cool, and the crystals of pure material re-precipitate and can be filtered off. In the course of this operation, the impurities stay in solution (in theory) such that the material is now free of most of the impurities.

Single-solvent Recrystallization

The procedure that involves **single-solvent recrystallization** includes the following steps:

- **Find a suitable solvent**. Always use small amounts (around 100 mg or the tip of a spatula) for your trial recrystallizations. A test tube can be used as the container since it can be heated. If you know the structure of your compound, you can deduce the property of polarity and therefore make an educated guess as to which solvents to test. The ideal solvent will be one in which the compound is quite insoluble at room temperatures and soluble at higher temperature. The higher temperature you choose should be lower than the boiling point of the solvent. As a guide, consider a solute to be soluble in a solvent if it dissolves 30 milligrams per milliliter of solvent, or more.[1]
  So, for example, a non-polar organic compound would not be soluble in water at any temperature, and thus water would not be a suitable solvent. On the other hand, a non-polar solvent might dissolve the compound even at low temperatures, and thus would not be a good recrystallization solvent either. The best way to establish the optimal solvent is through trial and error, but you can rule out some solvents with knowledge of how the structure of the chemical determines its properties.

- **Perform the test on a small scale.** Add a small portion of your solvent in a dropwise manner to the test tube and record your observations. If the solid dissolves, try another solvent. When you find a solvent that does not dissolve the solid at room temperature, heat the solvent with a water bath, a steam bath, or a sand bath. **Do not use a flame** unless the solvent is water. Observe and see if the solid dissolves. If it does, cool the solution under running water while shaking the test tube and observe to see if crystals form. If the solid is not soluble even at high temperatures, try another solvent.
- **Rapidly cool the system**. Add the minimum amount of hot solvent to the solid mixture until all dissolves. Cool the solution quickly and look for signs of crystallization. Smaller crystals are formed from

1. Lehman, J. W. Operational Organic Chemistry: A Problem-Solving Approach to the Laboratory Course, 3rd ed. Prentice-Hall: Upper Saddle River, NJ, 1999.

quickly-cooled solutions, which are easier to handle and filter. If you need larger crystals, allow the solution to cool slowly.

- **Seeding and Scratching**. If no crystals have formed after ~5 minutes of cooling, try scratching the side of the test tube with a spatula or glass stir rod. This will provide a rough surface which promotes crystallization. Alternatively, you could introduce a small "seed" crystal of the material on which the chemical can attach to an already-formed crystal and continue the process. Generally, the initial stages of crystallization are thermodynamically unfavorable, but once the process has begun it will proceed without any further help from you.
- **Reduce the amount of solvent.** If no crystals have formed even after "seeding and scratching", try boiling off some of the solvent in the hood and repeating the cooling procedure.
- **Start over with a new solvent**. If still no crystals form, boil off all the solvent in the hood and try another solvent.

The procedure that involves multi-solvent recrystallization is similar.

Binary-solvent Recrystallization

When using **binary–solvent recrystallization**, the procedures include the following steps:

- **Find two suitable solvents.** First solvent A should have a high solubility for the target compound at high temperature. Second solvent B should have a low solubility for the target compound. Both solvents should be miscible.
- **Perform the solubility test on a small scale**. Add a small portion of your solvent in a dropwise manner to the test tube and record your observations. If the solid dissolves, try another solvent. When you find a solvent that does not dissolve the solid at room temperature, heat the solvent with a water bath, a steam bath, or a sand bath. **Do not use a flame** unless the solvent is water. Observe and see if the solid dissolves. If it does, cool the solution under running water while shaking the test tube and observe to see if crystals form. If the solid is not soluble even at high temperatures, try another solvent. Choose the binary-solvent system based on small-scale tests.
- **Recrystallize the solid using binary solvents upon cooling.** Add the minimum amount of hot solvent A to the solid mixture until all dissolves. Slowly add solvent B until crystals form. Cool the solution quickly for more precipitation. Smaller crystals are formed from quickly-cooled solutions, which are easier to handle and filter. If you need larger crystals, allow the solution to cool slowly.
- **Seeding and Scratching**. If no crystals have formed after ~5 minutes of cooling, try scratching the side of the test tube with a spatula or glass stir rod. This will provide a rough surface which promotes crystallization. Alternatively, you could introduce a small "seed" crystal of the material on which the chemical can attach to an already-formed crystal and continue the process. Generally, the initial stages of

crystallization are thermodynamically unfavorable, but once the process has begun it will proceed without any further help from you.

- **Reduce the amount of solvent.** If no crystals have formed even after "seeding and scratching", try boiling off some of the solvent in the hood and repeating the cooling procedure.
- **Start over with a new solvent**. If still no crystals form, boil off all the solvent in the hood and try another solvent.

When you have found a suitable solvent and recrystallized a small batch of solid, **scale up the procedure** using an Erlenmeyer flask as the container and purify the whole sample. Remember, you can always get your material back by boiling off the solvent, but then the material will not be purified. When your material has recrystallized, filter off the solid with vacuum filtration and retain the liquid. It is possible to repeat the procedure and obtain second crop of crystals, but they will usually be less pure than the first crop.

Distillation

Distillation is a method of separating volatile liquid mixtures based on their boiling point differences or separating a volatile liquid from a solid. The liquid may be the product of a reaction or perhaps the solvent for a reaction. If your product is a liquid that boils below ~20°C, you can distill the liquid at atmospheric pressure. For liquids over 150-200°C, you will need to distill the liquid under reduced pressure.

There are two different kinds of distillation: simple distillation and fractional distillation.

Simple Distillation

Simple distillation is typically used when the boiling points of the components in a liquid mixture are significantly different (greater than 100°C difference) and when the goal is to separate the components efficiently without needing high purity. In a simple distillation process, the lower boiler liquid will vaporize in the original flask, pass the side arm, condense under cooling water in the condenser and be collected as "distillate". A thermometer was used to monitor the evaporation process.

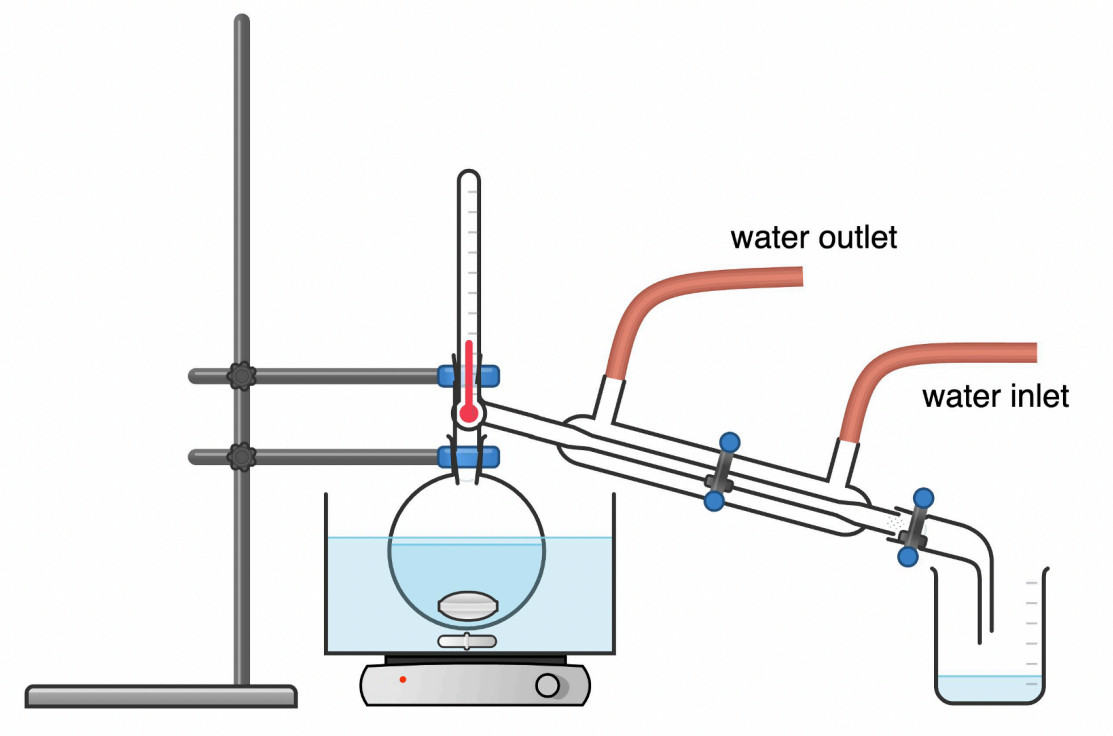


Figure 15. A simple distillation set-up. Created with Chemix

A simple distillation setup typically consists of a round bottom flask containing the liquid mixture to be distilled, a distillation head with a thermometer for temperature monitoring, a condenser to cool and condense the vapor, and a receiving flask to collect the distillate. The boiling flask is heated, and the component with the lower boiling point vaporizes first. The vapor travels through the distillation head and into the condenser, where it is cooled and condenses back into liquid form. The condensed liquid drips into the receiving flask, resulting in the separation of components based on their boiling points. There should be a magnetic stir bar in the water bath and another stir bar in the boiling flask to ensure even heating. All glassware should be fixated with clamps to the ring stand.

Simple distillation is a straightforward process that requires less time and energy. It is usually used when a rough separation is sufficient.

Fractional Distillation

Fractional distillation, on the other hand, is used when the boiling points of the components are closer together (less than 100 °C apart) and when a higher degree of separation and purity is required.

Fractional distillation utilizes fractional column (or "fractionating column") where separation is more thorough and complete. A fractionating column allows for many successive mini distillations within one

column. Each mini-distillation (called a "theoretical plate") represents a complete vaporization and condensation process.

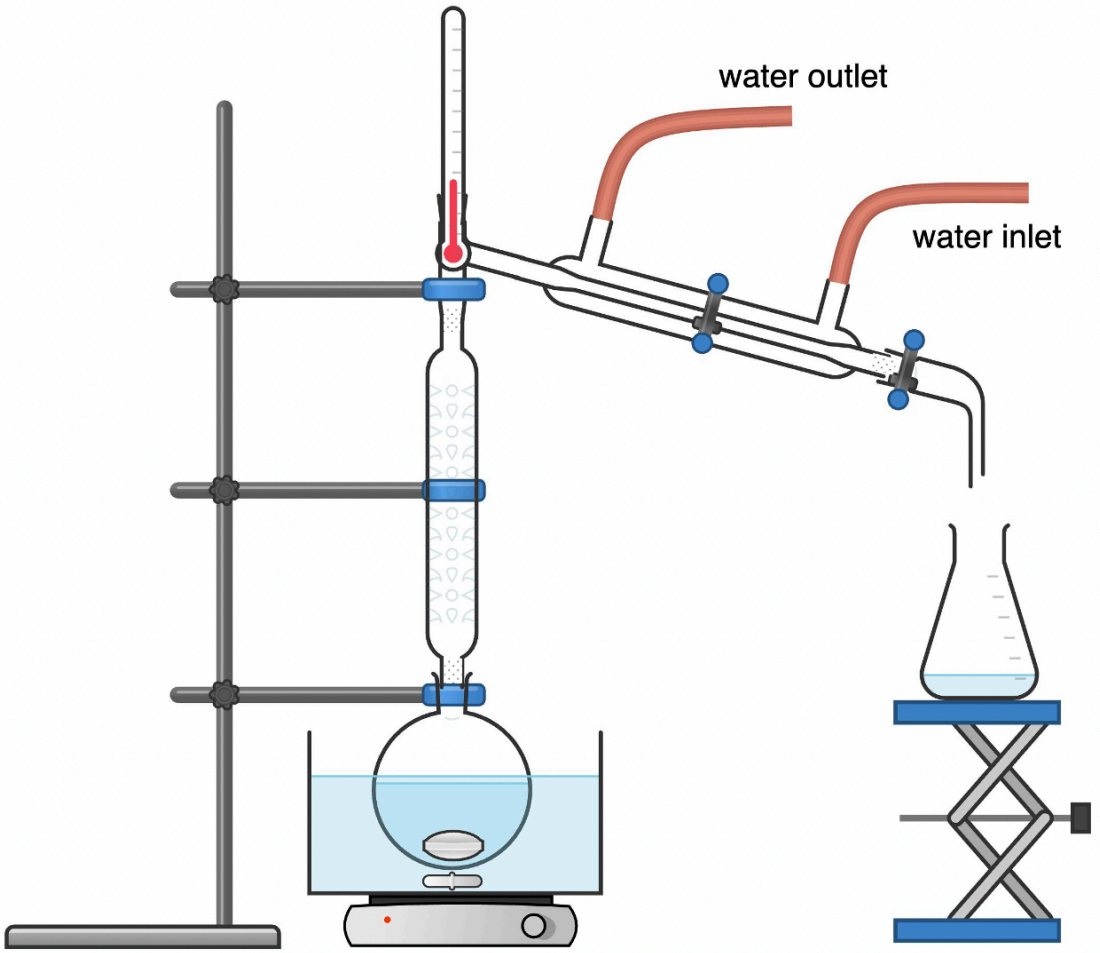


Figure 16. A fractional distillation set-up. Created with Chemix

A fractional distillation setup is similar to a simple distillation setup but includes a fractionating column between the boiling flask and the condenser. The fractionating column contains a series of glass beads or plates that provide a large surface area for vapor-liquid contact. As the vapor rises through the column, it condenses on the surfaces and then re-evaporates, undergoing multiple vaporization-condensation cycles. This process helps to separate components with closer boiling points by allowing for more efficient separation based on differences in vapor pressures.

Fractional distillation is used for liquid mixtures of small boiling point differences and normally takes longer and consumes more energy. From a green chemistry perspective, simple distillation should be considered first for energy-saving purposes when it comes to separating liquid mixtures with large boiling point differences.

Atmospheric Large Scale

Liquids that boil up to 200°C can be distilled at atmospheric pressure. If you are unsure of the boiling point of your material, you can do a trial boiling point determination by heating a small sample of the liquid in a test tube in a sand bath. When the liquid begins to boil, take the temperature of the vapor just above the liquid surface.

1. Set up the apparatus as shown above with the distillation flask placed in a water or sand bath, depending on the temperature needed for distillation. The distillation flask should be no more than half full. **Make sure all the joints are tight**. If you have a loose connection, you will lose your material to the atmosphere, and you might expose everyone to hazardous vapor.
2. Make sure that you have a couple of boiling chips in the liquid **before you begin to heat the flask**. If you begin without boiling chips, the liquid may get superheated and "bump", causing the liquid to spill over into the receiving flask without being distilled. If you forget to add the boiling chips, you will need to cool the liquid before you add the chips, otherwise the liquid will boil up vigorously and potentially boil onto you (leading to severe burns and exposure to irritating chemicals).
3. Place the thermometer head at the level of the adapter leading into the condenser, so that the temperature of the vapor that is being condensed is the one that is being recorded.
4. The condenser should have the water entering and leaving as shown. If the boiling point of the liquid to be distilled is over 100°C, an air condenser (i.e., a condenser with no water flowing through it) should be used.
5. Distill a small amount of liquid into another receiver. Then, when the boiling point of the liquid stabilizes, remove that receiver, and replace with a new clean flask. Distill the liquid until the boiling point starts to rise, or until there is ~1 mL of liquid in the distillation flask. **Never distill to dryness.**
6. Disassemble the apparatus while the glassware is still warm, using caution. If you leave the glassware until it is cool, the joins will likely contract, and you will not be able to get the pieces apart.
7. Put the stopper on the receiving flask containing your newly-purified material.

Reduced Pressure Large Scale

If you have a liquid whose boiling point is over 200°C, it may be difficult to distill for several reasons. It may decompose when heated in air at high temperatures. Alternatively, the apparatus in the lab may not be capable of reaching the temperatures needed.

In this case, you will need to perform the distillation at a reduced pressure which will lower the boiling point of the liquid. To carry out a reduced pressure distillation, the following changes must be made to the distillation setup described above:

1. Set up the apparatus, but instead of boiling chips, place a stir bar in the distillation flask.
2. Turn on the stir plate and then connect the aspirator hose to the vacuum adapter outlet on the receiving flask.
3. Turn the aspirator on fully, and then start to heat the distillation flask.

Continue as you would for an atmospheric macroscale distillation, discarding the first fraction that is distilled; collect the distillate (liquid that has been distilled) that comes over at constant temperature.

Small Scale

For distillations of amounts less than 5 mL, a microscale still should be set up instead of a large-scale distillation.

Column Chromatography

If you isolate an oil from your workup that will not crystallize, it may need to be purified by column chromatography. Chromatography is a powerful technique used to separate non-volatile solids or liquids mixture based on their different affinities with the mobile phase and the stationary phase. This separation is achieved by exploiting differences in the distribution of the components between the two phases, typically due to variations in adsorption and elution properties, for example, **polarity**, size, charge, or other chemical properties.

A column chromatography usually follows five steps, as illustrated in **Figure 17**:

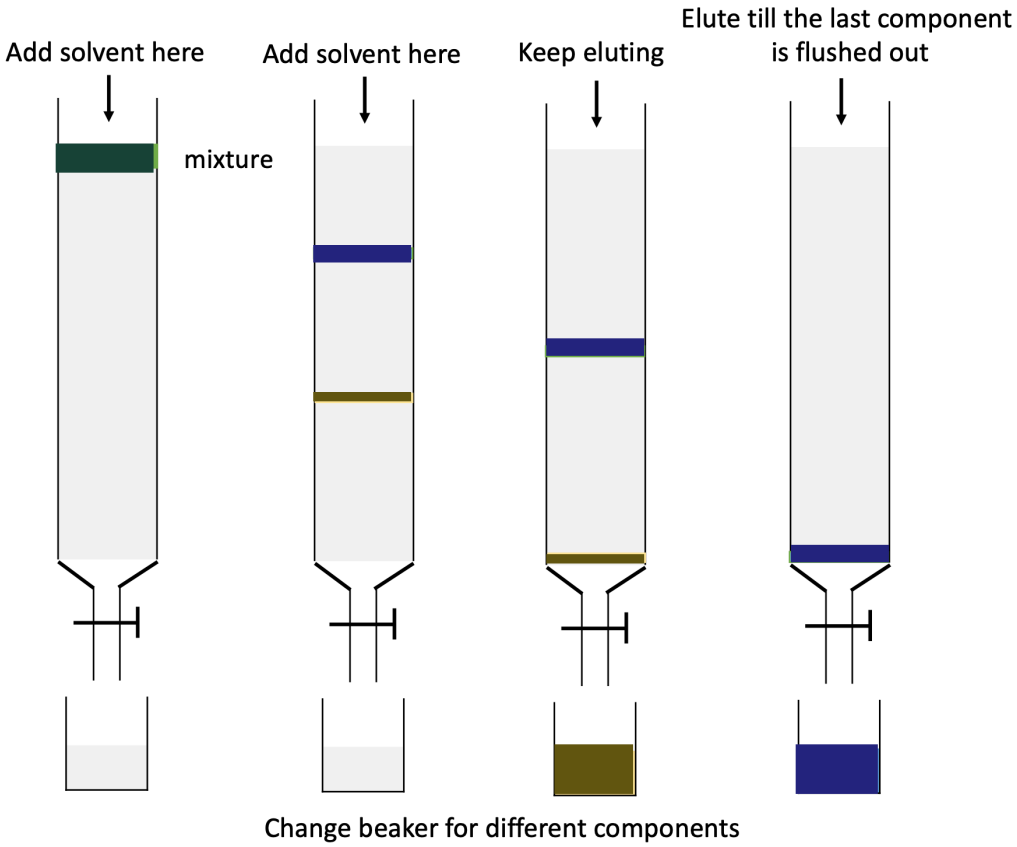


Figure 17. Column Chromatography for purification of a mixture. The figure was created by Mengqi Zhang, CC BY-NC.

1. **Column setup**: A glass column is packed with the stationary phase, which is typically silica gel or alumina. The stationary phase is prepared by wetting it with a small amount of mobile phase (elution solvent) before the separation begins.
2. **Loading the sample**: The mixture to be separated is dissolved in a small amount of the mobile phase and loaded onto the top of the column. The sample is usually loaded using a pipette, and care is taken to ensure that the sample is loaded in a small, concentrated band and not interrupting the packed column.
3. **Elution**: The mobile phase is then passed through the column using a gravity flow or a pump. As the mobile phase flows through the column, the different components of the mixture interact with the stationary phase to varying degrees. Components that interact more strongly with the stationary phase move more slowly through the column, while those that interact less strongly move more quickly. Note that you may not be able to witness the color change or separation as most compounds are colorless. Real-life monitoring is necessary.
4. **Collection of fractions**: As the components elute from the column, they are collected in fractions. Each

fraction represents a different component of the mixture. The fractions can be collected manually by collecting drops or using a fraction collector.

5. **Analysis**: The collected fractions can be analyzed using various techniques such as thin-layer chromatography (TLC), gas chromatography (GC), or mass spectrometry (MS) to identify the separated components.

Note: The column should not run "dry"—keep adding solvents from the top until the separation is complete.

21.

DEALING WITH UNKNOWN COMPOUNDS

It is important when dealing with unknown compounds to proceed in an organized manner. First and foremost, always follow the highest safety precautions. Wear appropriate personal protective equipment (PPE), such as lab coats, gloves, and safety goggles, to protect yourself from potential hazards. Work in a well-ventilated area to minimize exposure to fumes.

If you want specific information concerning the unknown compound, seek guidance from your lab instructor. If you are provided with a list of possible unknown candidates, look up the SDS information of all reagents to familiarize yourself with the properties of the unknown compound, such as its potential hazards and handling requirements. Dispose of the compound properly according to lab regulations.

Think through the process as far as you can with the information that you have available. When you know what tests you would like to do, you should first practice on known compounds so you will know how to perform them properly and what a positive result should look like. Only then should you begin to perform tests on the unknown compound. It is also important to conserve your material, i.e., you should use microscale quantities whenever possible..

Solubility Tests (Qualitative)

A quick **qualitative** test for solubility can give you a fair amount of information about a compound. For example, if your compound dissolves in water, it is probably a polar or ionic compound. In general, the solubility of ionic compounds decreases as the charge on the ions increases. Solubility in acid or base solution can also give some indication of the properties of the compound.

Suggested Liquids	Inference If Soluble
Water	Polar or ionic compound
If not soluble in water, try 1 M NaOH	Probably an organic acid
If not soluble in water, try 1 M HCl	Probably an organic base
Acetone	Probably **not** an ionic compound

Procedure:

1. Take a small amount of the unknown (e.g., the size of a grain of rice) and place it in a test tube.
2. Repeat with as many samples as there are solvents you want to test.
3. Add solvent into the test tube in a dropwise manner and swirl the solution. Observe carefully to see if any of the compound dissolves.
4. If your compound appears noticeably soluble in any of the liquids, you may also wish to do a **quantitative** solubility test.

General Solubility Tests (Quantitative)

A **quantitative solubility test** tells you exactly how much (in mass) of a solid will dissolve in a given volume of solvent.

Procedure:

- Decide how much volume you wish to use (e.g., 1 mL). Weigh out more solute than you think you'll need and put it in an **Erlenmeyer flask**.
- Then, using a **volumetric pipet,** add a measured volume of liquid and heat the solution while stirring until no more solute seems to go into solution. (It is necessary to heat the solution because solids often take long periods of time to dissolve at room temperature.)
- Place a watch glass on top of the Erlenmeyer flask and let the solution cool until any precipitation is complete.
- Filter off the remaining solid, allow it to dry, and weigh it. You will then be able to calculate the mass of solute in solution, and thus be able to calculate the solubility of the compound in grams per liter.

Measuring Solution pH

pH Paper

To get a general idea of the acidity or basicity of a solution, you can use pH paper. In the labs, we have "universal indicator" paper, which changes color along a spectrum of red (most acidic) to blue (most basic). One then matches that color observed to the scale provided on the packaging to get a rough estimate of the pH of the tested solution.

Figure 16. pH test paper. The figure was created by Mengqi Zhang, CC BY-NC.

Electronic pH probes may be available in lab. Please refer to the user manual and calibrate before use.

Other Tools

Spectroscopy tools represent another class of potent analytical instruments for identifying unknown compounds. Further details will be provided in the following chapter.

Note: The majority of this chapter has been adapted from the CEM 161/162 manual: Cooper, M. M. *et. al. Cooperative Chemistry for Michigan State General Chemistry Laboratories*, **2019**.

PART VI

PART 6: SPECTROSCOPY

Spectroscopy can be defined as the study of the interaction of electromagnetic radiation with matter. The energy of radiation depends upon its wavelength. Different wavelengths of radiation have different interactions with matter. For example, absorption of a photon in the ultraviolet and visible region may cause an electron to move between the quantized energy levels of the atom or molecule; these are called **electronic transitions**. Absorption of a photon of UV light may cause an electron to move to a higher energy level. If the electron drops back down to the original level, a photon of the same wavelength will be emitted.

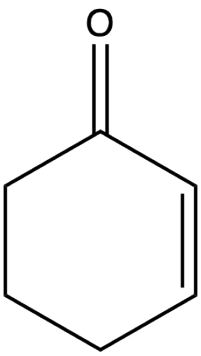
22.

INFRARED (IR) SPECTROSCOPY

Radiation of lower energies causes less energetic transitions between energy levels. For example, infrared radiation is of the same order of energy as the vibrational energy levels of a molecule (which are also quantized); these are called **vibrational transitions.**

Infrared (IR) Spectroscopy

Infrared spectroscopy works similarly to UV-visible spectroscopy (in that it involves absorption of light) but provides different information about the sample. Electromagnetic radiation in the infrared range corresponds to the energy of molecular vibrations. Vibrational energy levels are quantized just as electronic energy levels are. It is possible to measure the absorption of the IR radiation as it causes the molecule to change from one vibrational energy level to another.



A typical IR spectrum appears to be quite complicated, and a great deal of information can be obtained about the structure of the compound under investigation. In organic chemistry, the IR spectrum is very useful because particular functional groups have very specific frequencies at which they absorb. For example, carbonyl groups typically show absorptions in the region between 1800 and 1620 cm^{-1}. The units on a typical IR spectrum are expressed in **wavenumbers (cm^{-1})** which is the reciprocal of the wavelength of radiation absorbed.

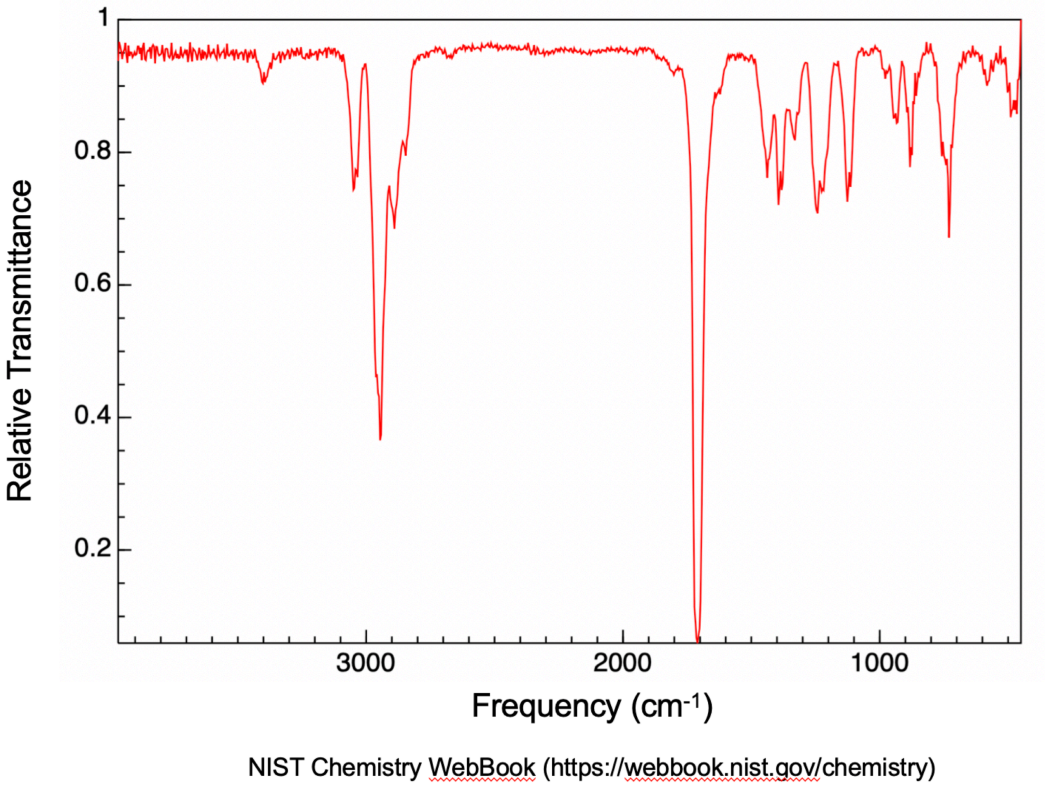


Figure 17. FTIR spectrum of cyclohex-2-en-1-one.

Figure 17 is a transmission IR spectrum of cyclohex-2-en-1-one (NIST WebBook). The most prominent feature is the strong absorption (i.e., little to no transmission) at about 1700 cm^{-1} which corresponds to the carbonyl (C=O) stretching frequency. You may also notice the peaks slightly above 3000 cm^{-1} to be sp2 hybridized carbon to hydrogen (C_{sp2}-H) stretching, while peaks slightly below 3000 cm^{-1} to be sp3 hybridized carbon to hydrogen (C_{sp3}-H) stretching.

The following table lists some common infrared spectroscopy absorptions by frequency regions.

Peak Position (cm^{-1})	Group	Class	Peak Details
3200-3550	O-H stretching	alcohol	strong, broad
3500-3350	N-H stretching	amine	medium
2500-3300	O-H stretching	carboxylic acid	strong, broad
2700-3200	O-H stretching	alcohol	weak, broad
3267-3333	C-H stretching	alkyne	strong, sharp
3000-3100	C_{sp2}-H stretching	alkene	medium
2840-3000	C_{sp3}-H stretching	alkane	medium
2222-2260	C≡N stretching	nitrile	weak
1818-1650	C=O stretching	Carbonyl containing compounds	strong

For a complete list of IR spectroscopy absorptions, see Infrared Spectroscopy Absorption Table.

Collecting an IR Spectrum

The Organic Chemistry Labs at MSU use commercial infrared spectrometers, which may be controlled via a graphical interface on a tablet computer. You should only operate the IR spectrometers after receiving proper training from your TA. However, you may find it helpful to familiarize yourself with the information here so that you can better follow along when it comes time to take IR spectra.

Application Startup and Spectrum Collection

The software has instruction screens built into it. The following instructions provide a detailed walkthrough and additional notes.

1. Sprinkle some ethanol on a Kimwipe. Clean the round panel using Kimwipes and ethanol. Do not sprinkle ethanol directly onto the FTIR instrument.
2. Start **OMNIC Paradigm** software on the instrument screen. Wait till the signal on the top right indicates "Nicolet Summit".
3. Click "**Background**" – wait for preview – click "**Start Background Measurement**". This process may take 30 seconds.
4. Load a SMALL amount of solid sample on the center of the round panel—twist the top detector part down until it is loosely in contact with the solid (do not push it too tight!). If your sample is a liquid, no

need to twist the top detector down.

5. Click "**Measure Sample**". This process may take 30-60 seconds.
6. **Enter** a name "sec 2 gr 2 pj 2 cat 4", for example, for your sample – click "**OK**".
7. Select and unselect from the right graph list to **only** show your spectrum. Eye button enabled: show spectrum. Eye button disabled: hide spectrum.
8. Click "File"→"Create Report"→"Create"→"Print"→Save pdf to your USB drive (inserted next to the screen). Eject the USB drive after the file was saved.
9. Clean bottom panel and top detector with Kimiwipes and ethanol.
10. Perform a simple analysis on the spectrum you collected. Ask your TA for assistance if you have any questions.

In conclusion, FTIR spectroscopy is a powerful analytical technique that provides valuable information about the chemical composition and structure of a wide range of materials. By understanding the principles behind FTIR and mastering the techniques involved, you will be able to analyze unknown samples, identify functional groups, and monitor chemical reactions with confidence.

Part of this chapter has been adapted from the CEM 161/162 manual: Cooper, M. M. *et. al. Cooperative Chemistry for Michigan State General Chemistry Laboratories*, **2019**.

23.

NUCLEAR MAGNETIC RESONANCE (NMR)

As covered in class, one powerful tool for characterizing and confirming an analyte's chemical structure is to take a nuclear magnetic resonance (NMR) spectrum. Unlike UV-Vis (which monitors the behavior of electrons) and infrared spectroscopy (which monitors the vibrational modes of the bonds within a molecule), this form of spectroscopy generates a signal (one or more sharp peaks) as a consequence of the spin of atomic nuclei. Using this characteristic, we can monitor how atomic nuclei of certain atoms (in this course, ^{1}H and ^{13}C) behave in a magnetic field (the spectrometer). For a refresher of the underlying theory and principles of NMR, see the OCLUE textbook (pages 44-56).

Spectrum Collection

The Nanalysis 60e (60 MHz) benchtop NMR spectrometer allows for routine screening of $^{1}H/^{19}F$ spectra with excellent performance. The following instructions provide a detailed walkthrough and additional notes:

NMR Sample Preparation:

- Weigh 0.03 – 0.05 g of solid power in a small beaker and dissolve in ~180 μmL chloroform-D solvent in the fume hood. Low-concentration solution will not yield good ^{1}H NMR spectrum.
- Add the solution to NMR tube with extreme caution. NMR tubes are very fragile! Any insoluble precipitation or suspension cannot be transferred to the NMR tube.
- Here is a reference of how much needs to be added to the tube: between 3-4 fingers would be ideal. It is okay if it goes beyond the four-finger line, but it cannot be lower than the three-finger line.

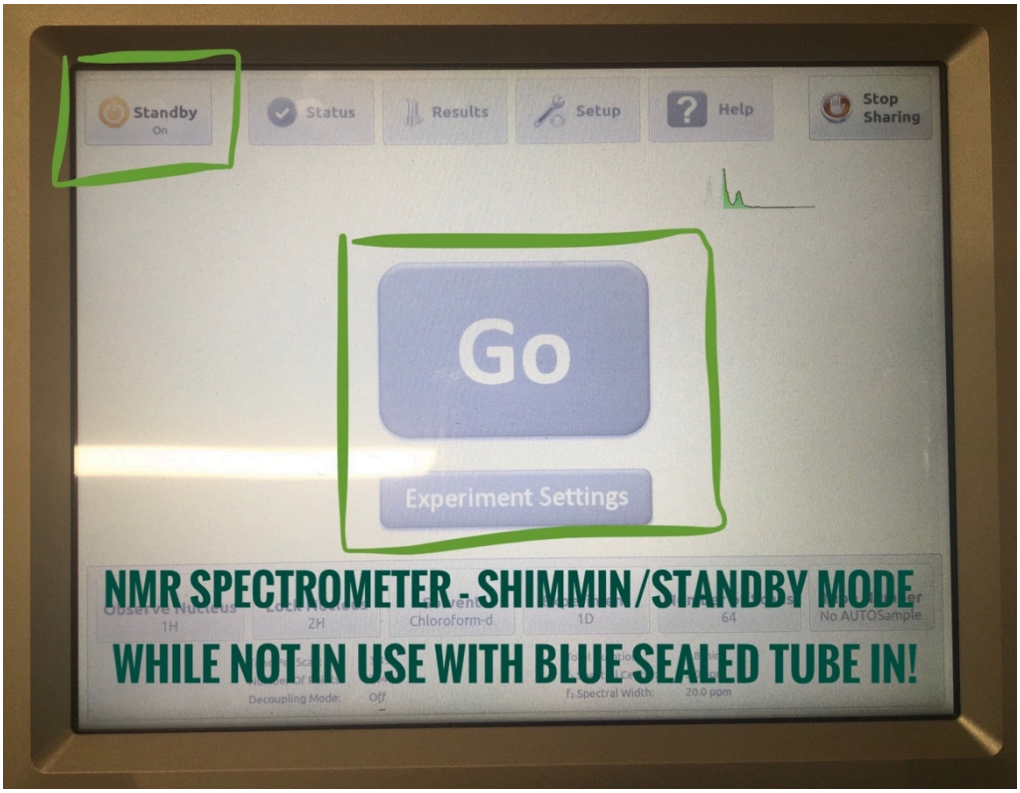
This figure was created by Mengqi Zhang, CC BY-NC.

- Cap the NMR tubes. Chloroform-D is a known carcinogen and health hazard, while being volatile.
- Put NMR tubes in the warmer next to the benchtop NMR for at least 5 minutes before the next step.

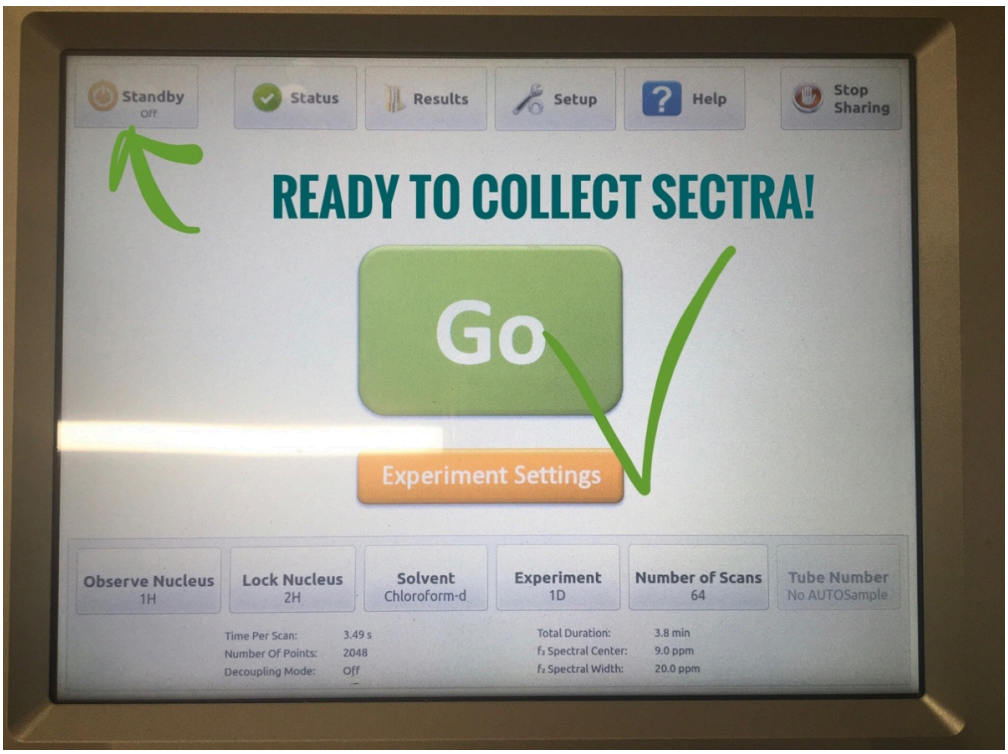
^{1}H NMR Spectrum Collection

Note: this process must be supervised by a TA or course instructor. Report to your TA if you notice anything irregular.

- NMR will be on **Standby mode** as shown below while not running samples. There should be a blue sealed tube in the instrument for standby processing.

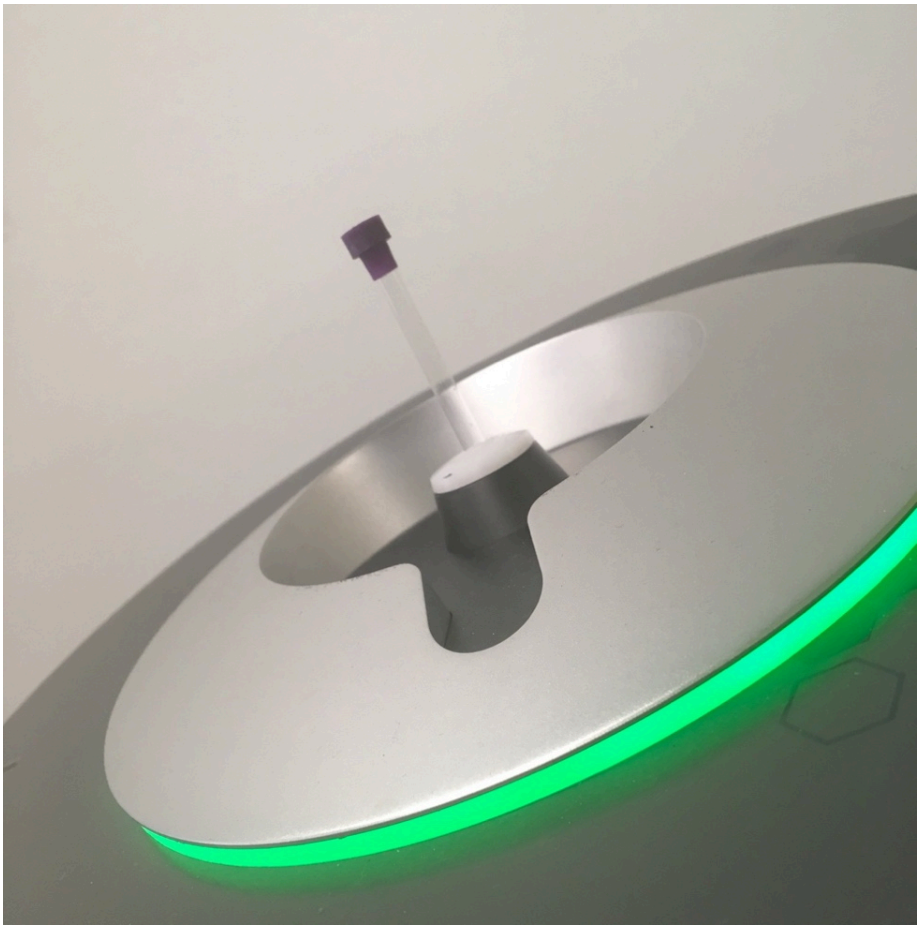


- Click on “Standby” orange button on the top left once. The “Go” button in the middle will turn green and be ready for sample collection!

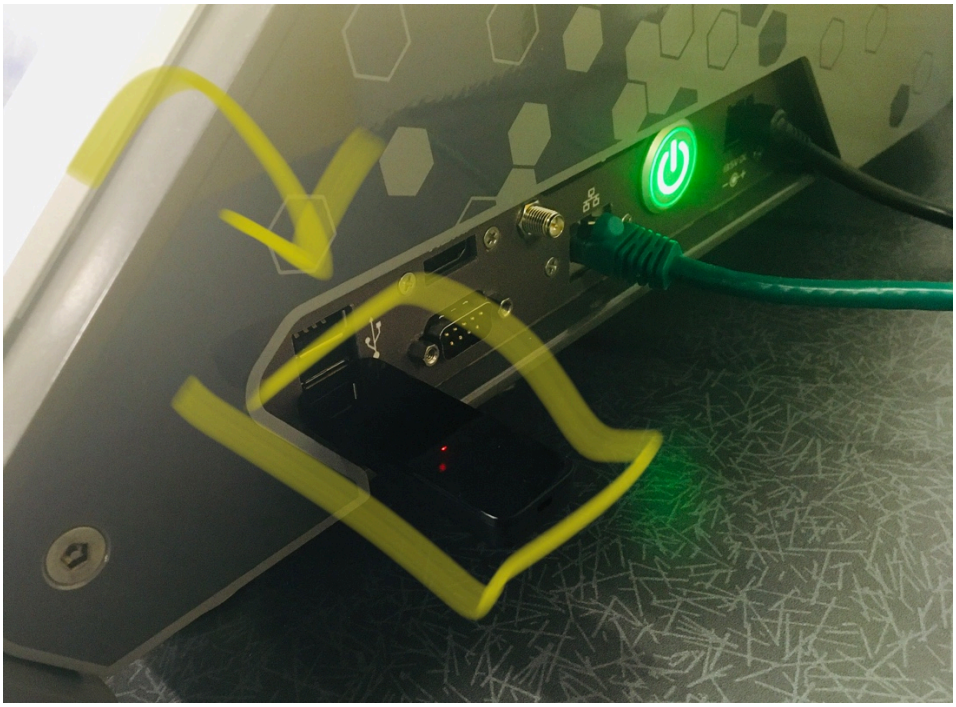


- Replace the blue-top sealed tube in the middle with your capped sample tube.

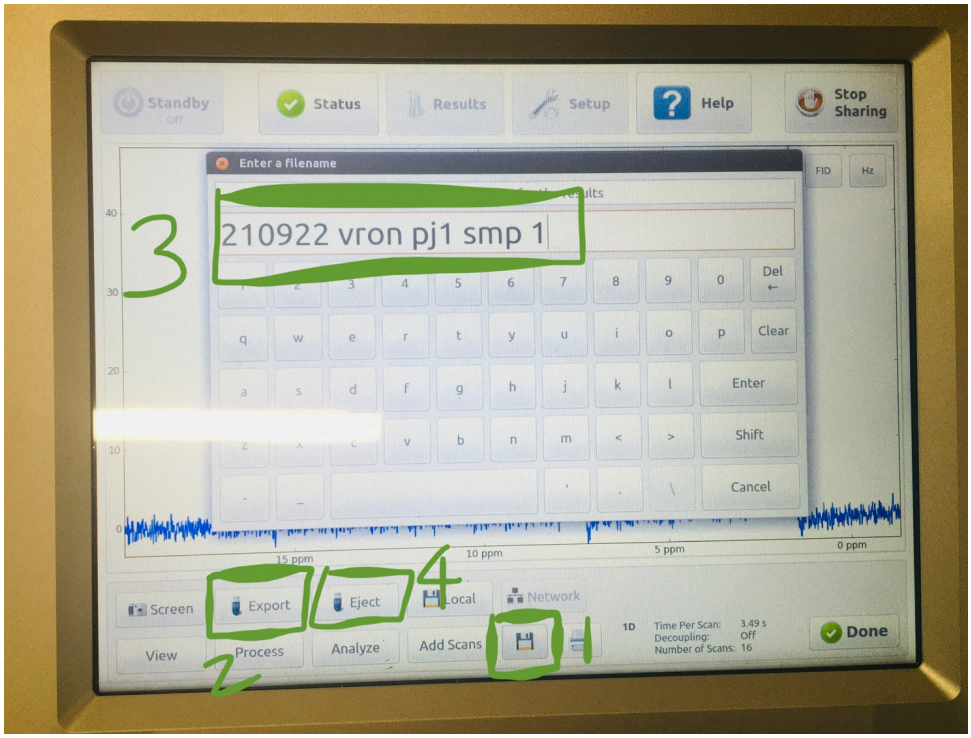
Careful: NMR tubes are pricy & fragile! Remove & insert with caution!



- Click the "Go" button on the screen, and you will see ^{1}H NMR spectrum on the screen. Remember you required the instrument to collect 16 (or 64 scans), so it may take a while (3-5 minutes) to complete! After you see the "Done" button at the right bottom corner, the collection is complete! Don't leave yet. You need to **save** your spectrum for integration next!
- Insert your flash drive (USB drive) in on the right bottom corner.



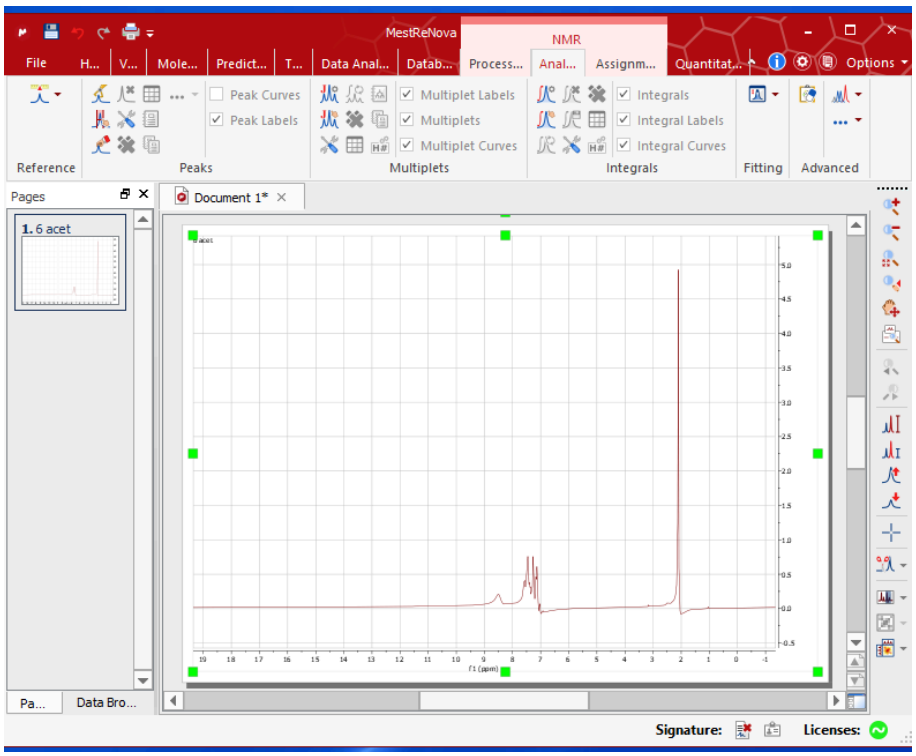
- Click on “save”. Select “Export” on the main screen. Save to a name you recognize. “Enter”, select “yes”. Click “Eject” (the flash drive) after spectrum is “saved”. Allow three seconds between every click to allow responding time for the instrument.



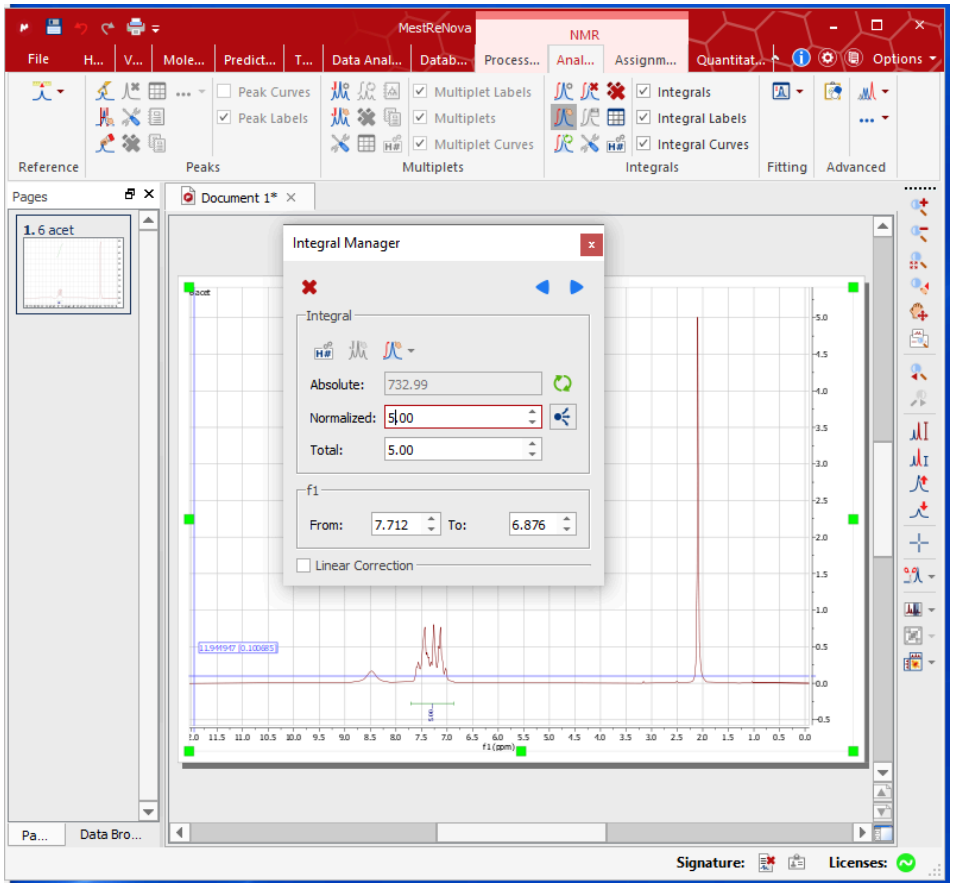
- Swap your sample NMR tube with the blue sealed reference tube in the NMR instrument.
- Click "**Standby**" again. If they asked for "insert blue tube" confirmation, select "yes". The instrument will go back to shimming/standby mode again before you use it next time!

^{1}H NMR Spectrum Integration & Analysis

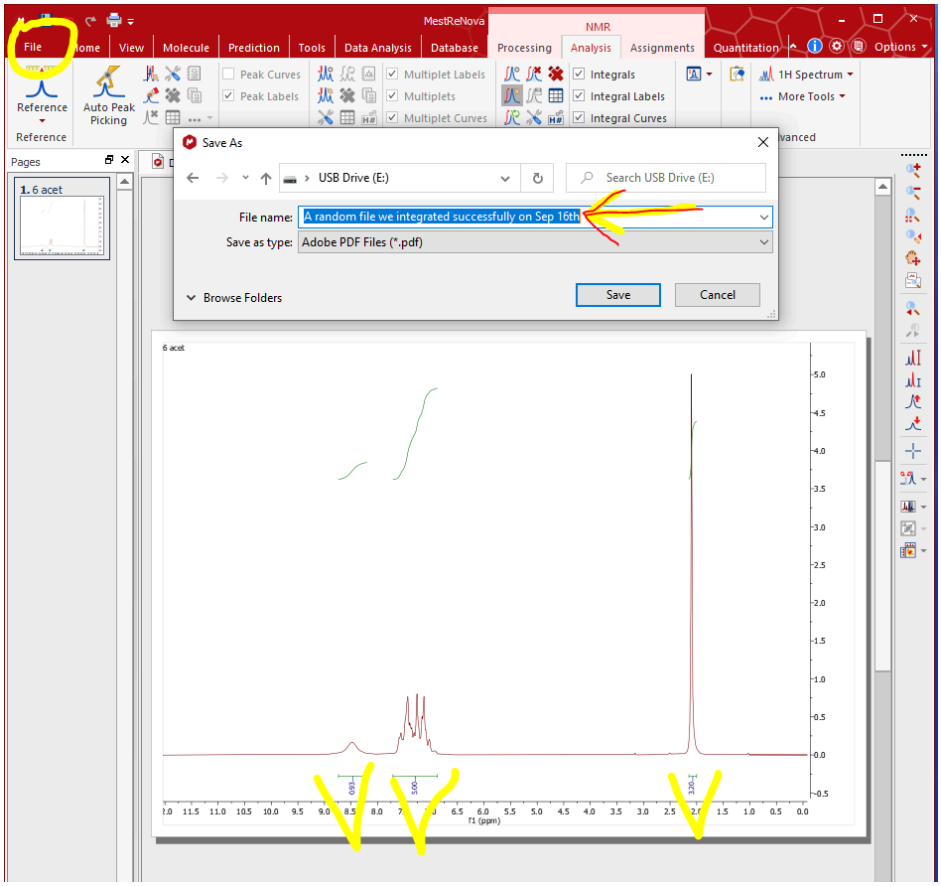
- Insert your flash drive in the computer next to the NMR instrument. Open the **"MestReNova x64"** software on the desktop. Drag the dx file saved in your flash drive onto the opened MestReNova software.



- Right click on the spectrum – **Phase correction – Automatic**.
- Right click on the spectrum – **Baseline – Baseline correction – Yes**.
- Right click – Zoom in – Drag to the area you want to focus on. Recommended range: 13 ppm – 0 ppm.
- [optional] Right click – peak pick – Automatic.
- Right click – **Integration – Manual**. Integrate all dominant peaks with your TA's help. Do not integrate the solvent or impurities' peaks.
- Select a peak range as reference – right click on the peak – edit – Normalize the peak to a predicted number.



- [optional] Right click on the spectrum – Properties – Grid – unselect show horizontal/vertical/frame – OK!
- File – Save As – Name the file and save as “Adobe PDF files” or “JPEG” as file type in your flash drive so that you can analyze on your laptops (Check with your TA if the integrated spectra is correct or not).



Note: The information on the spectrum is essential for identification! Analyze the spectrum collected from the following aspects:

- Integration: number of protons
- Chemical shift: chemical environment the protons are in
- Splitting pattern: "N+1" rule can help you predict the number of neighboring protons (N).

PART VII

PART 7: ASSIGNMENT GUIDES

24.

QUICK GUIDE TO STUDENT ASSIGNMENTS

Assignment	How to Do It	How to Submit It	When It is Due
Planning Scenario and Background Document	• Access on D2L (Content >> project-based laboratories >> projects) • Complete **with team members**	• Download the copy from D2L • Complete the document and turn in to D2L as a team	The day after lab
Laboratory Part 1-3 Document	• Access on D2L (Content >> project-based laboratories >> projects) • Complete **with team members**	• Download the copy from D2L • Complete the document and turn in to D2L as a team	End of lab period
Laboratory Notebook & Citizenship	• Record only what you did and observed in lab in great details • Supplemental files (e.g., graphs) should be saved in Team Drive • This is an **individual** assignment.	Submit a photo of written record or the electronic record you completed in lab on D2L (Assessments >> assignments >> individual notebook and team contribution) individually at the end of the session	End of lab period
Peer & Self Evaluation	• Log in to CATME and complete survey	Completion will be recorded in CATME upon finishing	One week after each project

Assignment	How to Do It	How to Submit It	When It is Due
Informal Lab Report	• Access on D2L (Assessments >> project-based laboratories >> projects) • Fill out in **as a team**	Upload file to D2L as a group (Assessments >> Assignments)	Deadline in syllabus (usually one week after the last lab session for project 1)
Formal Lab Report (sections & final)	• Access on D2L (Assessments >> project-based laboratories >> projects) • Complete the document **as a team**	Upload file to D2L as a group (Assessments >> Assignments)	Deadlines in syllabus (usually one week after the last lab session of project 2)
Oral Presentation	• Prepare **as a team** in slideshow software of choice	• Present during scheduled class time	In class, see syllabus
Poster Presentation	• Prepare **as a team** in desired format	• Present during scheduled class time	In class, see syllabus
Case Study Worksheets	• Log in beSocratic • Fill out **as a team**	• Completion will be recorded upon submission in beSocratic	Completed during the 50-minute "recitation" meeting concurrent with lab enrollment

Assignment	How to Do It	How to Submit It	When It is Due
Case Study Decision Memo	• Access on D2L (Content >> case study) • Complete with team members	• Download the copy from D2L • Complete the document and turn in to D2L as a team	Deadlines on D2L
Case Study Policy Brief	• Type in word processing software of choice	• Download the copy from D2L • Complete the document and turn in to D2L as a team	Deadlines on D2L
Case Study Policy Paper	• Type in word processing software of choice	• Download the copy from D2L • Complete the document and turn in to D2L as a team	Deadlines on D2L

Note: certain assignments requirements/deadlines may subject to changes. All changes will be reflected on D2L.

25.

PLANNING DOCUMENTS

Each session your team will plan the experiments to be conducted the following session. This should be completed as a team with the recorder, ensuring that the information is submitted on D2L. During the planning phase, the communicator needs to summarize the plan and gain approval from your TA as the plan is being developed so they make sure you are not going too far off task or planning to do something dangerous or impossible.

Your TA is **not** there to give you procedures or tell you if you are doing it "right". The main focus of this lab is about using your chemical knowledge and critical thinking skills to solve and report on problems; this laboratory is less focused on having the right answer at the end of the day. It is up to each member of the team to research the problem **before the lab period** and come prepared to work with the team to plan a solution to the project scenario. Each part of a project has guiding questions to help guide your team's thinking. These questions are only part of the information needed in the planning phase, as indicated in the Planning Document rubric.

Time management and properly dividing up work during the planning stage is essential for success in this course. While planning is done as a group, each person is expected to work independently and conduct complete experiments. Each lab period is 2 hours and 50 minutes long. If you work as a single entity and conduct experiments together, that is all of the time you have. But if each member works independently to conduct a well-planned procure, that amount of time is quadrupled allowing your team to accomplish much more in the same amount of physical time! If each team member cannot independently collect data to analyze that feed into the project, then the work is not organized correctly. Note that this does not mean that each person must solve all the project problems independently; it means that each person is making an important contribution to the project goal(s).

Ideally your project plan should have several sections:

- **Responses to planning questions.** Based on the project scenario, you will have to answer questions about relevant background information, what project goals you will focus on, and/or ideas about how to approach the goals. You should cite the references you used in responding to these questions.
- **Equipment and chemical lists**. List all chemicals (and hazards using SDS info) that you will be using. Provide a list of equipment needed outside of the routine equipment in your drawer and include a drawing of the apparatus setup for this project.
- **Equations.** Include both chemical and non-trivial mathematical equations you will be using, if

applicable.

- **Detailed procedure for each person!** Each team member should have their own set of experiments that they can work on independently of the group. Everyone needs to be doing their own small slice of the project that will contribute to the overall project goals. Everyone has a purpose and each person is doing their own experiments. No one is measuring things out for the whole group, cleaning up after the whole group, or just observing. **Everyone does meaningful work!**
- **Include as much detail as possible**. This will hold everyone accountable and provide each member a list of actionable items and techniques so that each person knows what they need to know and do for the next lab period.

Planning documents for the Identification of an Unknown Organic Solid and the Synthesis and Analysis of Painkillers were provided as examples in the Appendix.

Planning Document Template

Title of Lab

Team Leader	Recorder	Communicator	Safety Officer

Planning Questions

Chemicals

Chemical Name	Hazards	Personal Protective Equipment (PPE)

Waste Management

Disposal Method	Material(s) or Chemical(s) That Should Be Disposed By This Method
Sink	
Trash Can	
Waste Carboy	
Other (Specify)	

Equipment

Equations (Reactions, Equations, or Green Metrics)

Project Plan (Procedures)

Write a preliminary plan for your experimental procedure. Indicate what each person in your team will do to solve the problem and what data they will record. **Remember, this is not the team roles**. Break down the work so you use your time effectively and that **each person will end up with a mini lab report for their data and notes** assignment next session.

Sample Planning Document

Investigation of Luminol Reaction

Team Leader	Recorder	Communicator	Safety Officer
Teammate 1	Teammate 2	Teammate 3	Teammate 4

Planning Questions

Responses to planning questions go here**.**

Chemicals

Chemical Name	**Hazards**	**Personal Protective Equipment (PPE)**
Luminol	Not classified as hazardous	Handle with gloves
Bleach (sodium hypochlorite, NaClO)	Hazardous Skin corrosive – Cat. 1 Serious eye damage – Cat. 1	Handle with gloves
Copper sulfate ($CuSO_4$)	Hazardous Acute toxicity, oral – Cat. 4 Skin irritant – Cat. 2 Eye irritant – Cat. 2	Handle with gloves
1 M sodium hydroxide (NaOH)	Hazardous Skin corrosive – Cat. 1 Serious eye damage – Cat. 1	Handle with gloves

Waste Management

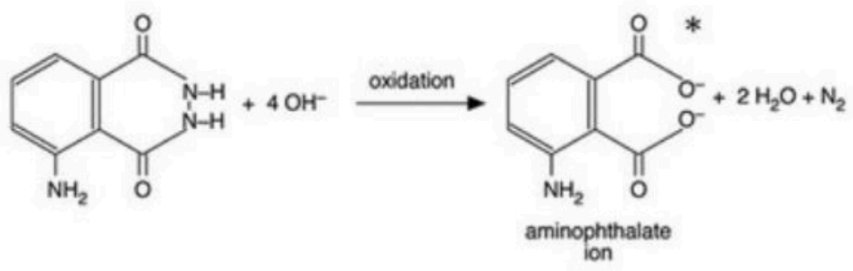
Disposal Method	**Chemical(s) That Should Be Disposed by This Method**
Sink	Excess NaOH (after neutralization)
Trash Can	
Waste Carboy	All reaction mixtures; excess luminol, bleach, and copper sulfate
Other (Specify)	

Equipment

- Small beakers (1 per team member)
- Small test tubes
- 10 mL graduated cylinder (to make solutions)
- Dark container (to better see reaction progress)
- Stopwatch (to time reactions)

Equations

- Luminol reaction (reference link: Chemiluminescence of luminol: a cold light experiment)



Project Plan (Procedures)

Teammate 1

Goal: Determine the effect of bleach on the rate of luminol reaction.

Procedures:

- Dissolve 10 mg of luminol in 2 mL of 1 M NaOH solution in a small test tube.
- In small beaker, mix 0.9 mL of bleach with 9.1 mL of water.
- To start the reaction, add 1 mL of bleach solution to the luminol solution (start timer as soon as mixed).
- Observe reaction in a dark box and time the length of reaction.
- Repeat the above procedure using the following bleach solutions:

Bleach (mL)	Water (mL)
1.8	8.2
3.6	6.4
5.0	5.0
10	0

Data Analysis: Plot length of reaction (time) on y-axis and % bleach on the x-axis. See if there is a trend.

Teammate 2

Goal: Determine the effect of luminol on rate of luminol reaction.

Procedures:

- Dissolve 4 mg of luminol in 2 mL of 1 M NaOH solution in a small test tube.
- In a small beaker, mix 0.9 mL of bleach with 9.1 mL of water.
- To start the reaction, add 1 mL of bleach solution to the luminol solution (start timer as soon as mixed).
- Observe reaction in a dark box and time the length of reaction.
- Repeat the above procedure using 6 mg, 8 mg, 10 mg, and 12 mg of luminol dissolved in 2 mL of 1 M NaOH Solution.

Data Analysis: Plot length of reaction (time) on y-axis and mass of luminol on the x-axis. See if there is a trend.

Teammate 3

Goal: Determine the effect of $CuSO_4$ catalyst on rate of reaction.

Procedures:

- Dissolve 10 mg of luminol in 2 mL of 1 M NaOH solution in a small test tube.
- Add 1 mg of $CuSO_4$ and stir to dissolve.
- In a small beaker, mix 0.9 mL of bleach with 9.1 mL of water.
- To start the reaction, add 1 mL of bleach solution to the luminol solution (start timer as soon as mixed).
- Observe reaction in a dark box and time the length of reaction.
- Repeat the above procedure using 2 mg, 3 mg, 4 mg, and 5 mg of $CuSO_4$.

Data Analysis: Plot length of reaction (time) on y-axis and mass of copper sulfate on the x-axis. See if there is a trend.

Teammate 4

Goal: Assist other group members during their trial and ensure every team member follow safety guidelines.

Data Analysis: Summarize the data everyone collected and propose a solution.

Planning Document Success Guide

This rubric will be embedded into D2L for the lab instructor to evaluate your plan. They will provide you feedback in the comment portion on D2L of any strengths to your plan and any changes that your team needs to make. Planning documents are 10 points total, graded holistically, with an emphasis on the procedures and safety.

Goals for Planning Document		Strengths & Suggestions for Improvement
Planning Questions	Did the team address the planning questions and incorporate the responses into their plans?	
Chemicals	Did the team consider the hazards and risks of all the chemicals needed?	
Waste Management	Did the team designate the waste management plan for all chemicals and materials?	
Equipment	Did the team include an equipment list, especially including items not found in their team drawers?	
Reactions & Equations	Did the team make note of the reaction (if synthesizing) or any equations/green metrics they will need?	
Procedure	• Did the team address this session's goal(s) in their procedures? • Does every team member have their own procedure with an equitable contribution to the goal? • Do the procedures contain specific details of their plans, such as concentrations and approx. amts of chemicals?	

26.

NOTEBOOK AND TEAM CONTRIBUTION

The Laboratory Notebook

You will be required to keep a laboratory notebook for the duration of this course. The notebook is preferably bound, with the pages numbered consecutively for easy reference. The preferred notebook is the carbonless copy type, in which each page is reproduced twice so that both you and your laboratory instructor can keep a copy of your lab notes. Since we are**going green**, you can also choose an electronic notebook.

The notebook is a day-to-day record of your activities in the lab and can only be completed while in lab. It is the place where you will describe experiments **as you do them** and note observations **as you make them**. It is where you will record and analyze your data. Your notebook will be an invaluable tool throughout the semester, as you must have an accurate record of what you did and what you observed in the laboratory when the time comes to write your laboratory reports.

All data, results and weights, etc. should be **recorded directly into your notebook in ink**. Loose pieces of paper might easily get lost. Remember that it is to your benefit to keep a detailed notebook which will make it easier for you to reconstruct the experiment accurately in a report later. Any mistakes should be crossed out but still legible, so that one can see what change was made. Do not erase or cover mistakes with white-out.

The notebook should **not** be a neatly copied reiteration of the laboratory procedure. Rather, you should write what you did and observed. Neatness, spelling, punctuation and grammar are not essential in this notebook. "Perfection" is not expected, but it should be possible for someone else to repeat your work by reading your account, i.e., it should be legible and intelligible. Your data and notes will be shared with your lab partners to help coordinate the team's overall analysis and summary of the work. This can be done by taking a picture on your phone, scanning your notebook pages, or typing up what you did, and then sharing it with your lab partners (e.g., by email or on a shared drive).

Since the lab notebook should be used for recording everything that you do in the laboratory, there is not one particular format that can be used for all situations, so it is important to be flexible. However, there are certain things you should do to make the lab notebook as useful as possible:

- Leave a few pages blank at the beginning of the notebook. As you begin new experiments and projects you can use these pages to prepare a table of contents that can be updated as you go.
- Make sure all the pages are numbered.

- Make sure that each page is signed and dated. Although this may not be so important in an introductory lab, it is certainly good practice, and is required in many research labs.
- Because each team member should have a purpose (as described in the planning document), each person should have a "mini lab report" by the end of the day. This purpose, e.g., "My goal this session is to test for the presence of different cations in unknown #273," should be written at the start of your notebook pages for the day.
- Finally, when you are done experimenting, you should analyze your data and summarize your work for the day.

At the end of each lab period, you will be required to turn in the photo of notebook pages or electronic version of notebook record to D2L. Your instructor will grade your notebook pages based on quality of notebook and attendance/participation. Note that your notebook grade will be assigned a "0" for lab absences.

Team Contribution (Laboratory Citizenship)

In addition to what you have recorded in your laboratory notebook, your instructor will pay attention to how appropriately you comported yourself in the laboratory. Some things they may consider include, but may not be limited to, the following: **timeliness, safety, fulfillment of team role, participation, respectfulness, cleanliness, punctuality, etc**.

Sample Lab Notebook Page

Here is an example of what a lab notebook page might look like. You are **not** limited to one page per lab session; use as many as you need. **Remember to put something behind the corresponding copy page (your notebook may have come with a thick cardstock page) so that your writing does not bleed through to the next blank page!**

Exp. No. 3	Experiment/Subject Esterification – Week 2	Date 5-17-19	100
Name Student McStudentface	Lab Partner Team A	Locker/ Desk No. 201	Course & Section No. 06

PURPOSE To make an ester using 3-nitrobenzoic acid as the starting material.

PROCEDURES

① Dissolved 1.03g of 3-nitro benzoic acid in 25.00mL of ~~e~~ methanol, + added 2 drops of 18M H_2SO_4

② Heated rxn mixture for 30 minutes at ~50°C + then poured rxn mixture into 20.00mL of ~~DI water~~ 0.1 M NaOH

③ Crystals separated by vacuum filtration + left to dry.

OBSERVATIONS / DATA

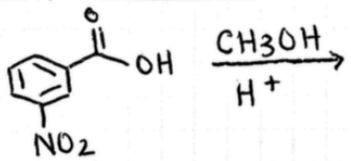
① Rxn mixture got warm + darkened in color

② cream-colored crystals formed as ppt when added to NaOH

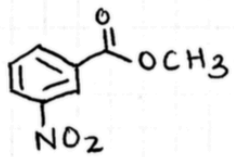
③ mass filter paper = 1.21g
mass crystals + filter paper = 1.71g
→ mass crystals = 1.71g - 1.21g = **0.50g**

ANALYSIS / DISCUSSION

Rxn:



167.12g/mol



181.15g/mol

Theor. yield: $1.03g \times \frac{1\,mol}{167.12g} \times \frac{1\,mol}{1\,mol} \times \frac{181.15g}{1\,mol} = 1.12g$

% yield = $\frac{0.50g}{1.12g} \times 100\% = 45\%$ yield

The product was synthesized with only 45% yield. The low yield may be due to an incomplete rxn (took off heat too soon), or it didn't fully precipitate out.

Signature Student McStudentface	Date 5/17	Witness/TA	Date

THE HAYDEN-McNEIL STUDENT LAB NOTEBOOK

Note: Insert Divider Under Copy Sheet Before Writing

Notebook & Team Contribution Success Guide

Your lab instructor will read your notebook in order to see whether you are keeping up with your work, whether you understand what you are doing, and whether you are recording everything you need. This guide will be uploaded into D2L, and your lab instructor will comment on strengths of your work and contributions to the team as a citizen of the laboratory, as well as offer suggestions for improvement. This assignment is worth 20 points, 10 for notebook record and 10 for team contribution (laboratory citizenship).

Goals for Notebook Page(s)		**Strengths & Suggestions for Improvement**
Record of Individual Lab Work	• Does the notebook page(s) reflect the individual's weekly contribution? • If applicable, does the notebook page(s) acknowledge the contribution(s) of a teammate if it includes results from collaborative laboratory work done?	
Purpose	Is there a clear purpose to the experiments performed by the individual student during the lab session?	
Procedures	Is the procedure clear, reproducible, and reflective of the actual steps taken and amounts used?	
Data and Calculations	• Are the relevant data and observations are neatly presented? • If applicable, are the relevant data analysis or calculations shown?	
Conclusions	Does the individual offer any conclusions as to the results of the experiment?	
Lab Citizenship	Up to 10 points, at the discretion of your instructor, based on performance in lab (e.g., following safety protocol, wearing PPE, cleanliness, teamwork, etc.)	

27.

INFORMAL PROJECT REPORT

The informal project report (IPR) is a short assignment that asks for some conclusion about the project in the form of an evidence-based argument. Students will receive a worksheet designed to encourage critical analysis of their work, scientific communication of findings, and the presentation of a coherent arguments supported by evidence and reasoning.

Evidence-Based Arguments

When writing an evidence-based argument, there are three pieces that are required: **the claim, the evidence, and the reasoning.** Use the guiding questions in each section to help make your argument as complete as possible. Provide all raw data collected in the lab and use them as evidence for analysis and support your claim. Examples are included for each section to help make the description clearer.

Claim: "What Do You Know?"

The claim portion of your argument answers the question, "What do you know?" Depending on the activity, the claim may be given for you, or it could be that you need to make a claim based on your investigation. Claims are almost always declarative statements.

For example, let's consider wave-particle duality of light (the notion that light can behave both as a particle and as a wave). There are two claims here:

- **Claim 1**: Light is a wave.
- **Claim 2**: Light is a particle.

In an evidence-based argument, the claim is the statement you are trying to make **based on data**. Your evidence and reasoning must support your claim to ensure the clarity of your argument.

Evidence: "How Do You Know That?"

The evidence portion of your argument includes the data, results, and/or observations that relate to your claim. Think about what data you need to convince the reader that your claim is valid. It may also be helpful to think about answering the question, "How do I know that the claim I've made is correct?"

As an example, let's look at just one of the claims above: Light is a wave. The evidence you provide might include the what is **observed** in a double-slit **experiment**:

- **Evidence:** When light shines through two slits, a diffraction pattern is visible.

This statement of evidence is not particularly convincing—and thus the argument is incomplete—without the reasoning that links the claim to the evidence.

Reasoning: "Why/How Does Your Evidence Support Your Claim?"

The reasoning portion of your argument is the **most** important part. The reasoning describes why/how your evidence supports your claim using the scientific principles that are at work. This is often the largest part of the argument and is where you are able to demonstrate your knowledge of the system you are exploring.

If we continue with our example, the reasoning explains **how** the wave nature of light is supported by the evidence:

- **Reasoning:** A diffraction pattern is observed when waves that are propagating from each slit meet "in phase" and "out of phase". When waves meet "in phase", constructive interference occurs causing the wave intensity to increase. This is shown by the bright places in the pattern. When waves meet "out of phase", destructive interference occurs. When this happens, the waves cancel each other out producing dark spaces in the pattern. Because we observe a diffraction pattern when light passes through two slits, light must behave like a wave.

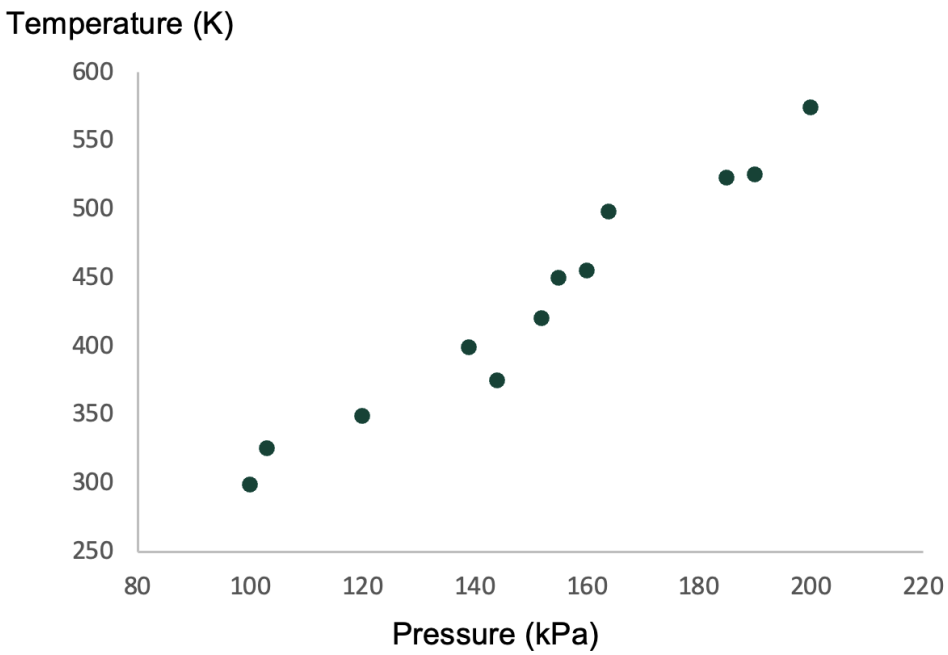
You can see that this explanation is thorough and shows how the observation of a diffraction pattern relates to the claim that light is a wave. It explains why the claim makes sense. The stronger the reasoning is linking your evidence to your claim, the stronger, and more trustworthy, your argument is.

Sample Informal Project Report

Please construct an evidence-centered argument in response to the prompt below.

Your boss, who is not a scientist, wants to know at what temperature to store nitrogen gas. Will storing it at higher temperatures or lower temperatures be safest to store (less likely to implode) compressed gases for the company?

- **What is your claim?**
  It would be safest to store compressed gases at lower temperatures because as the temperature of the gas increases, the pressure also increases.
- **What is the experimental evidence that supports your claim?**
  The data (see below) show that as the temperature (in Kelvin) increases, so does the pressure (in kPa), in a roughly linear manner.



This scatter plot was created by Mengqi Zhang, CC BY-NC.

- **Is your claim supported by established scientific principles (circle one)?**

YES NO

IF YES, explain how your claim and evidence agree with established theory.
IF NO, explain what scientific theory says **should** have happened and suggest what source(s) of error could have contributed to your unexpected results.

- **What is the reasoning that connects the evidence to the claim?**
  The pressure of a gas is the amount of force that a gas exerts on its container. Increasing the temperature of a gas increases the kinetic energy that the gas particles have. With a higher amount of kinetic energy, the gas particles will exert more force when they collide with the sides of the container, and they will also collide with the container more often. This means that particles at higher temperatures will exert more force on the container than particles at lower temperatures.

Informal Project Report Success Guide

A detailed report guideline will be uploaded to D2L, and your lab instructor will evaluate your argumentation along these goals. Your instruction will offer feedback on the strengths of your argument as well as areas to improve. This assignment is worth 60 points.

Here's a general success guide:

Goals for Informal Report		Strengths & Suggestions to Improve
Claim	• Does the claim agree with canonical science understanding? • Is the claim phrased coherently (all parts are in logical agreement)?	
Evidence	• How strongly does the evidence presented support the claim? • Are all relevant pieces of evidence are included?	
Reasoning	• Did the team provide reasoning that fully explains how the claim and evidence are connected? • Did the team demonstrate an understanding of the relevant scientific principle(s)? • For more detailed guidelines, please refer to the information provided on D2L.	

Note: This section was adapted from Erin Duff's work in Cooper, M. M. *et al Cooperative Chemistry for Michigan State University*, **2019**.

28.

FORMAL PROJECT REPORT

What is a Formal Report?

Scientists are investigators who "try out" ideas: they conduct experiments to provide evidence for or against these ideas. They share the results of their experiments in papers and other reports to allow others to learn the results of their investigations. You, too, will conduct experiments, record observations, collect data, and formulate evidence-based conclusions. For one project in this course, you will be asked to write a **formal report** describing the experiment, summarizing your results, and explaining your conclusions or judgments about the meaning of what you observed.

Audience

Professional scientists write for various audiences: themselves, their colleagues, or the public. They must write in a manner appropriate for the chosen audience. In preparing **your** lab report, think of the reader as an educated person who is interested in learning about your experiment; for example, you might imagine that you are trying to describe and explain your work to an organic chemistry student at a different institution who has not done this project. Although you know your reader will be your TA, who knows quite a lot about your experiments, **you** are the expert on what **you** did. It is your responsibility to communicate what you did and what it means efficiently and accurately.

Scientific Style

As you write, pay close attention to the "style" of your work. In scientific papers, facts and interpretation of those facts are what counts; thus, depersonalized writing known as "scientific" or "academic" is most appropriate for your lab report. What are the common elements of this style?

- **Use the third person and passive voice.** Although you are describing what you did, scientific reports should be impersonal. The easiest way to do this is to use the third person and passive voice ("X was done to Y," which contrasts with active voice: "X did Y"). First person ("I/we/me/us") is generally discouraged, and second person ("you") should not be used.

- **Use the past tense.** You are writing about the experiment you have already completed; therefore, everything you describe happened in the past.
- **Be as accurate and specific as possible.** Successful scientific description requires exact detail.
- **Be clear and concise.** Scientific reports are not the place for flowery prose or roundabout descriptions.

Let's look at some examples:

YES/ NO	Example Text	Comments
NO	Student M isolated the product using vacuum filtration.	It does not matter who did it, just that it was done.
NO	Isolate the product using vacuum filtration.	You should tell the reader what you did, not tell the reader to do it (i.e., use declarative sentences, not imperative sentences).
NO	A side-arm flask, Buchner funnel, and filter cone were obtained. Thick-walled tubing was connected to the sidearm flask fitted with the Buchner funnel. Filter paper was placed in the funnel and moistened with a small amount of water. Vacuum was achieved via water aspiration. The reaction mixture was poured into the funnel, thus isolating the solid product from the rest of the solution.	This is **too much** detail. Typically, common techniques do not need to be described in step-by-step detail; you just need to be clear about what technique you used. Unless you modified a standard procedure or did something novel, then you do not need to get into individual steps of how the technique was performed.
NO	The product was isolated.	This is **not enough** detail. There are many ways to isolate a product. What method did you use?
YES	The product was isolated by vacuum filtration.	This gets straight to the point of how you collected a reaction product.

The style used in scientific reports may be quite different from that required for a paper in another field. It is important that you write in a style appropriate for your audience, which may require you to understand and get used to using different writing styles.

General Format

The following is an outline of the fundamentals of writing a lab report. Please read it carefully. There is also a sample laboratory report with comments for you to read. The grading criteria for your lab reports will refer to the following outline.

Lab reports contain the following sections: **Title Page**, **Introduction**, **Experimental Methods, Results**, **Discussion**, **Conclusion**, and **References**. Your lab report must be typed and should include tables and figures (e.g., graphs, molecular structures, etc.) where necessary. The accepted font is Times New Roman, size 12, and the pages must be double-spaced.

Title Page

The first page of the report is the title page. The title should reflect the content and focus of the project described in the report. It should be as short as possible and include essential key words.

Note: Due to MSU Regulations concerning the use of Turnitin.com, **do not put your name on the title page**. The D2L System will manage the file and ensure we know who turned in what paper.

Introduction

The text of the report begins with an introduction. In this section of the paper, **concisely tell the reader what you intended to do and why it was important to do it.** Make sure you clearly communicate to the reader the purpose of the experiment(s) and provide background information required to understand the experiment(s) and the reason it was worth doing. Remember, longer is not necessarily better; indeed, for your report, you will likely need no more than one page to provide a suitable introduction.

You must cite credible sources to support statements you make about the scientific background or basis for your investigation. Citations tell the reader a few things: 1) that you have prepared properly for the experiment by providing yourself with a relevant background from experts in the field or other accepted authorities, and 2) where they can delve more deeply into the subjects you have introduced.

These citations should be numbered consecutively in the text and listed in the References Section. Typical sources for your citations include textbooks, your lab manual, reference books, and internet resources. For details on formatting your citation, see **References** at the end of this section.

Experimental

In this section, you give the reader a detailed account of the actual experiment. Scientific experiments are not considered valid unless they can be repeated by others. In this sense, science has no secrets: scientific theories become established only when the experiments that led to them can be repeated or verified by others besides the original investigators.

Scientists also use lab reports as a means of learning and sharing techniques. While others may not choose to duplicate your exact experiment, they may choose to use your procedure in a similar investigation. Thus, the experimental section of your lab report should be sufficiently detailed such that others could read your lab report and successfully duplicate your experiment.

Results

In this section you summarize the outcome of your experiments for your reader. This section will consist primarily of **data** that you and your group members gathered in the course of the experiment. You must organize the data so that it is easy to read. Numerical data is usually presented in tables. Relationships between sets of data (e.g., intensity vs. wavelength) are usually presented as graphs. Graphs, drawings, and other images are called **figures**.

All tables and figures should be numbered and labeled descriptive titles (e.g., **Figure 1: Infrared Spectrum of Polypropylene**). Any tables or figures should **also** be introduced in the written body of your report, using its corresponding number (e.g., "The infrared spectrum of polypropylene is shown in Figure 1"). Place tables and graphs near where you mention them in the body of your report. This makes it easy for the reader to use and understand the graphs, charts, tables, etc.

Discussion

The discussion section of your report is the most important one for you and your reader. In this section of the report, you interpret the results of your experiment for the reader. You explain **what** the results mean (claim), **how you know** what they mean (evidence), and **why** your claims make sense (reasoning). Here, you will also mention any weaknesses or limitations of the methods you used. This demonstrates not only how successful your experiment was but also how well you understood the experiment. The discussion section can be difficult to write, but you will learn more about your experiment and yourself as an investigator as you write it.

Before you begin writing this section, review your Introduction, Experimental, and Results sections. Look at all these sections, as well as your lab notebook, as you write the Discussion section. In the context of the goals you presented in the Introduction, you should discuss **all** of the results of **all** of the experiments that you presented.

Conclusion

Your overall conclusions about the project have probably already been mentioned elsewhere in your report. You may have predicted some of the outcomes of your experiments in the **Introduction** and discussed them again as an empirical conclusion (meaning that it was derived from your experiments and observations) in the **Discussion** section. In the Conclusion section, you should briefly summarize those major conclusions. In a sense, it is a summary of the **Results** and **Discussion** sections combined. A single paragraph may be enough to clearly summarize the outcome of your investigation.

References

The final section of your report tells the reader where to find any of the sources of information you used in your report. In the body of your report (particularly in the **Introduction** and **Discussion** sections), you will have mentioned other sources of information. Each of these references should be numbered consecutively within the text as superscripts. At the end of your report, include a complete reference list, numbered in the order in which you mentioned each source. The reader can use this list to follow up on any source you mentioned or to do additional reading.

References should follow the American Chemical Society Guidelines for superscript number format, which can be found in the ACS Style Guide.

Here is an example:

> *A similar experiment has been reported by Haight[1] and expanded by Vogel[2].*
>
> **References**
>
> 1. Haight, G.P. *J. Chem. Educ.* **1965**, *42*, 468.
> 2. Vogel, A. I. *A Textbook of Qualitative Inorganic Analysis*, Longman: New York, **1979**, 358.

Format for Typical References

Source Type	General Format	Example
Journal Article	Author (Last Name, Initials). *Journal Title Abbreviation (italics*—see ACS Style Guide page 288 for abbreviations), **Publication Year (bold)**, *Volume Number (italics)*, starting page number—ending page number.	Carmel, J.H.; Herrington, D.G.; Posey, L.A.; Ward, J.S.; Pollock, A.M.; Cooper, M.M. *J. Chem. Ed.*, **2019**, *96*, 423—434.
Book	Author (Last Name, Initials), *Book Title (italics)*, Publisher: City, **Publication Year (bold)**, page number.	Harte, J. *Consider a Spherical Cow*. William Kauffmann, Inc.: Los Altos, CA, **1985**, 83—88.
Web Page	Web Page Title. URL (accessed Mon. Day, Year).	Journal of Chemical Education (ACS Publications). https://pubs.acs.org/journal/jceda8 (accessed Apr. 10, 2019).
Course Lab Manual	Author (Last Name, Initials). *Title of Manual*. Institution Name, City, State. Student laboratory manual, Year.	Cooper, M.M.; Day, E.L.; Zhang, M. *Cooperative Organic Chemistry Student Laboratory Manual*. Michigan State University, East Lansing, MI. Student laboratory manual, 2024.
Your Lab Partner's Work	Author (Last Name, Initials). Institution Name, City, State. Personal communication, Year.	McStudentface, S. Michigan State University, East Lansing, MI. Personal communication, 2019.

Plagiarism and Turnitin

What is Plagiarism?

Examples include direct copying of someone else's work, published or unpublished, and representing it as your own; sentence-by-sentence paraphrasing of someone else's work; changing words here and there in material from another source. If you use a reference or another source, you should cite it and convey the ideas that you are using from the reference in your own words. **In short, make sure that all ideas in your work for this course are expressed in your own words.** It is only when you are able to express scientific ideas using your own words that you truly understand what you are writing about. In addition, **figures (diagrams, graphs, etc.) and tables submitted for an individual grade must be your own work.** Make it clear which ideas are your own and which came from other sources. Whether accidental, blatant, or self-plagiarism, the same standards and penalties

apply. Additional information about plagiarism and MSU's policies on plagiarism can be found on the website of the Office of the Ombudsman.

Anything submitted for an individual grade, even though such assignments will be based on your team's experimental work, must be your own work and not copied from your fellow team members. Do not share your work on the Formal Report assignments with other students or ask other students to see their work on Formal Report assignments because both constitute academic misconduct for these assignments.

Use of Turnitin

The following has been adapted from "Syllabus FAQ," Office of Ombudsman.

Consistent with MSU's efforts to enhance student learning, foster honesty, and maintain integrity in our academic processes, we have chosen to use a tool called Turnitin to compare your papers with multiple sources. The tool will compare each paper you submit to an extensive database of prior publications and papers, providing links to possible matches and a "similarity score." The tool does not determine whether plagiarism has occurred or not. Instead, we will make a complete assessment and judge the originality of your work. All submissions to this course may be checked using this tool.

You should submit papers to Turnitin via D2L **without identifying information included in the paper (e.g., name or student number)**. The D2L system will automatically show this information to us when we view the submission, but the information will not be retained by Turnitin. If you forget and submit your paper with your identifying information on it, your identifying information will be retained in the Turnitin repository. Your submissions will be retained in the Global Turnitin repository.

In choosing to use Turnitin in this class, we have agreed to follow five guidelines:

- We will use Turnitin as part of a balanced approach to encourage academic integrity and foster student success.
- We will openly disclose use of Turnitin in this course on the syllabus and at the time assignments are announced.
- For a given assignment, we will use Turnitin for all papers.
- We will make the final determination of originality and integrity.
- To ensure privacy, we will ask students to remove identification (e.g., names and student numbers) from submissions.

If you have any questions about the use of Turnitin in this course, please bring them to your instructor's attention.

Formal Report Success Guide

Your lab instructor will read, comment on, and evaluate your report. They will comment on how well you communicate necessary background information as well as the processes and results of the experiment itself. Your instructor also observes how carefully you follow the format for lab reports described in this manual. The guide included here is a general guideline and shows how your report will be assessed. Within each section, the instructor will comment on the strengths of your writing and offer suggestions for improvements you can make. Make sure you understand what the goals on the guide mean. Read them carefully and ask your lab instructor about any which puzzle you.

You will be submitting your formal report within one week after the last lab section.

Introduction Guideline

Introduction		
Goals for Introduction Section		**Strengths & Suggestions to Improve**
Introduction of scenario and project goals	Does the introduction summarize the problem to be investigated? Does the introduction highlight the relevant project goals and/or criteria for answering the driving question posed in the scenario?	
Relevant and sufficient background	Does the introduction include—as needed—any relevant background information that sufficiently supports choice of experiments?	
Originality	Are ideas from the project scenario or relevant background information expressed in their own words and organization? (Paraphrased and summarized, not verbatim)	
Reference use	Does the introduction appropriately use and cite outside literature?	

Experimental & Results Success Guide

Experimental		
Goals for Experimental Sections		**Strengths & Suggestions to Improve**
Original description of methods	In the experimental section, are the procedure(s) that the team used described in their own words? (i.e., not just copied from the project scenario)	
Basis in literature	For experimental details that were incorporated based on external reference materials, were these references appropriately cited?	
Clear & reproducible	Is the procedure provided completely clear and detailed? From this section, could a peer replicate the entire experiment?	
Experiment details only	Does not mention any details that belong in other sections (i.e., provides experimental details only).	
Procedure format and organization	Is the section formatted as a narrative (paragraphs)? Is the section logically organized (by experiment)?	

Results		
Goals for Results Section		**Strengths & Suggestions to Improve**
Inclusion of important results	Does the section include all results (positive AND negative)?	
Results only	Is this section focused on presentation of the results? (Discussion, analysis, or interpretation of results in the next section)	
Presentation	Are the data presented in an appropriate, easily readable format? Are all measurements labeled with appropriate units?	
Graphs & data tables	Do all graphs and tables include appropriate labels and titles?	

Discussion & Conclusion Success Guide

Discussion		
Goals for Discussion Section		**Strengths & Suggestions to Improve**
Claim	Does the data presented relate to the claim made?	
Evidence	How strongly does the evidence support the claim? Are all relevant pieces of evidence included?	
Reasoning	Does the Discussion provide scientific reasoning that fully explains how the claim and evidence are connected? Does the reasoning demonstrate an understanding of the relevant scientific principle(s)?	
Relates results to project goals	Does the Discussion of results clearly and logically relate to achievement of project goals?	
Discussion of error and/or limitations of experiments	Does the Discussion identify unexpected or inconsistent results? Does the Discussion describe possible sources of error? If no unexpected results are observed, does the Discussion describe the limitations of experimental approach or methods?	

Conclusion		
Goals for Conclusion Section		**Strengths & Suggestions to Improve**
Project Goals	Are all of the goals for the project addressed? If applicable, do they suggest future work based on their results?	
Limited to summary	Is the conclusion a concise summary with no new data presented?	

References		
Goals for Reference Section		**Strengths & Suggestions to Improve**
Complete source list	Are all the cited sources in the paper listed? Does the list contain at least one source beyond the course materials?	
In-text citations	Overall, are the In-text citations in proper superscript numeric format AND numbered in order of appearance?	
Reference list format	Are the references in ACS style?	

Overall		
Goals for Overall Writing Presentation		**Strengths & Suggestions to Improve**
Clarity and precision	Is the argument presented in the overall report completely clear and precise with adequate detail?	
Units	Are all of the measurements labeled with appropriate units?	
Logical and readable	Are all the sections logical and easy to follow?	
Style	Is the entire report written in scientific style?	
Title	Is the title page correctly formatted?	

How to Submit Your Report

Accepted File Formats

You can use any word processing program you wish: Microsoft Word, Apple Pages, Google Docs, etc. **However, regardless of what program you use, you must submit your file as a PDF (.pdf, preferred) or Word Document (.doc or .docx) format.** Other formats, including Apple Pages (.pages) documents, cannot be read by Turnitin. Therefore, you should be especially careful if you use a program other than Microsoft Word since the default file type is likely not a PDF or Word Document. Most programs can save files as PDFs, which is the preferred file type for Turnitin.

Note: The file must include the .pdf, .doc, or .docx extension in its name.

If you need to manually add the extension to the file name, or you are unsure if the extension is in the file name, consider the steps below:

- **MacOS users**: If the extension is hidden in the file name, you can either add the extension manually or go to "Get Info" (item on the File dropdown menu) and unselect "Hide extension."

Submitting to Turnitin via D2L

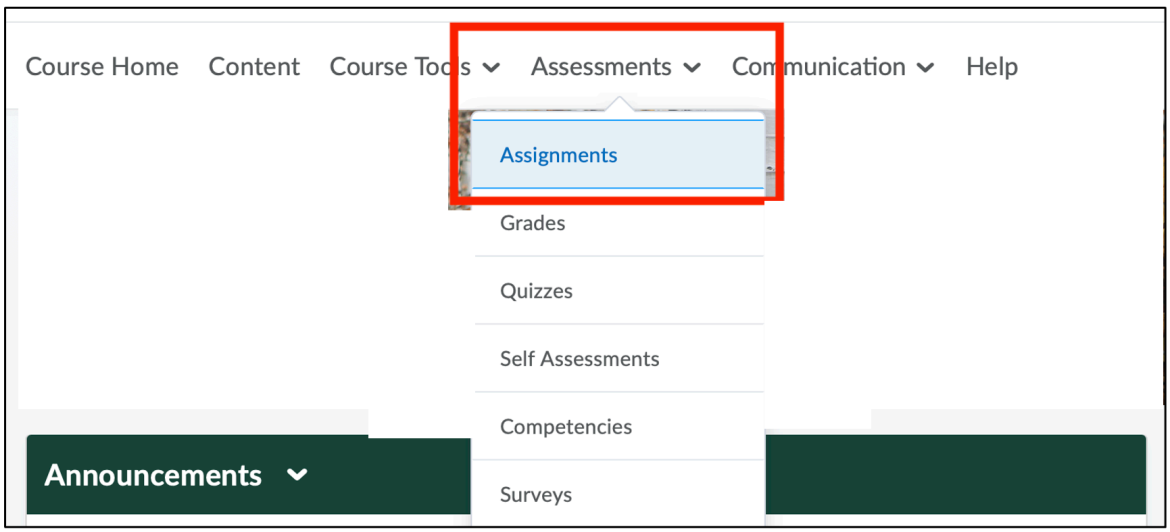
All files are to be turned in on to the course website on D2L. Once you go to the D2L course page, the report can be uploaded by navigating to the Assessments >> Assignments page (see screenshots below).

Note: Be certain you are uploading the correct document! Only the most recent submission will be graded.

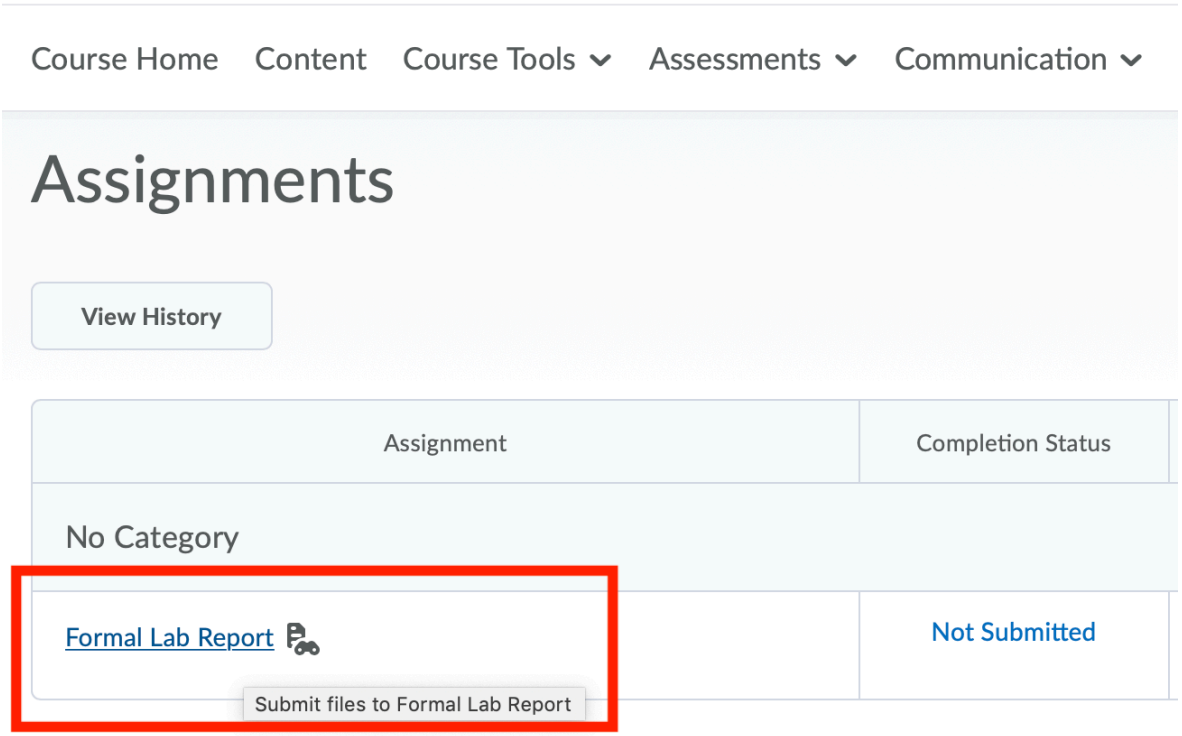
If it is a group document submission, everyone will have access to the file uploaded by one team member. Only one team member needs to turn it in.

Follow the instructions on the next page for document submission on D2L:

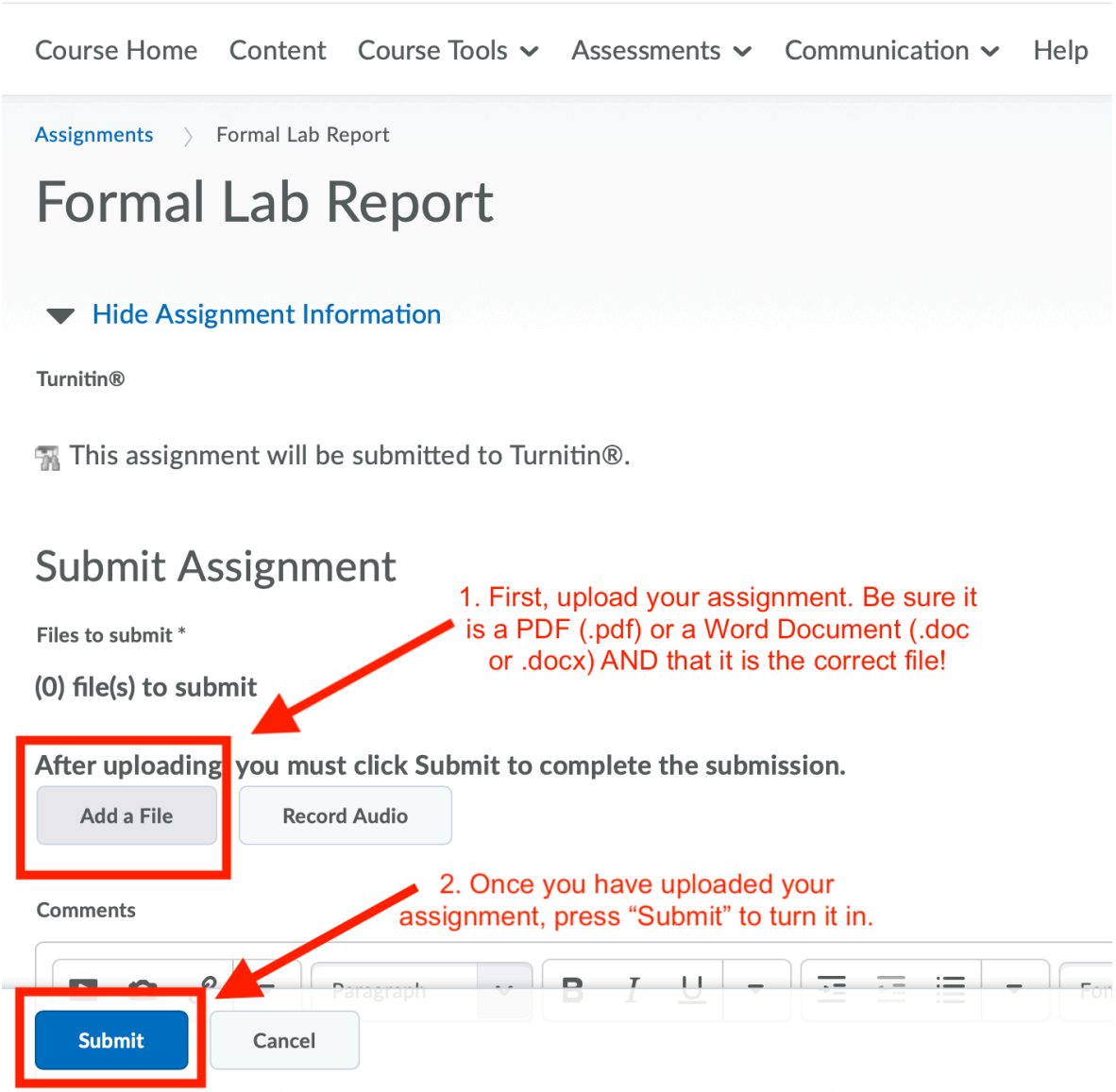
1. Navigate to the Assignments page after logging in to D2L.



2. Navigate to the assignment submission page.



3. Upload your assignment and then click “Submit”.



Sample Formal Project Report

An Investigation into the Structure and Properties of an Unknown Acid

Introduction

The goal of this laboratory project was to investigate the structure and properties of an unknown compound which had been found in an unmarked bottle in a local high school laboratory. The team of investigators was called in when a new high school chemistry teacher noticed the bottle sitting on the back of a shelf. The teacher asked the retired teacher who formerly occupied her classroom what it could be, and they narrowed down the possibilities to nine acids: formic acid, acetic acid, citric acid, lactic acid, phenol, salicylic acid, pyruvic acid, 3-nitrobenzoic acid, and 4-nitrobenzoic acid.

Hazardous waste is regulated by the Environmental Protection Agency (EPA) under the Resource Conservation and Recovery Act[1]. Considering that only one of the possible identities of the unknown acids (citric acid) is considered nonhazardous by EPA and State of Michigan standards[2], it was especially critical that the acid was identified in preparation for disposal. Identifying the acid would enable the teacher to safely discard the unwanted chemical.

Experimental

First, the physical appearance and odor of the unknown compound were observed. The melting point was determined using a Mel-Temp apparatus.

Solubility Tests (Qualitative).

A small amount (about 0.100 g) of the unknown was added to 2 mL of the solvent in a test tube and shaken. The test tube was observed to see if any of the solid had dissolved. The solvents used were toluene, acetone, methanol, water, 0.1 M HC1 and 0.1 M NaOH.

Solubility Tests (Quantitative)

Acetone, Methanol, Water, and HCl: The compound was too soluble in acetone and methanol, and not soluble enough in HCl and water, to test the quantitative solubility.

NaOH: The unknown compound (0.205 g) was placed in a small Erlenmeyer flask and 5.00 mL of 0.1 M NaOH was added. The mixture was stirred and heated slightly for about 15 minutes. A watch glass was placed on top of the flask, and the solution was left to cool for one week. The resulting mixture was filtered, and the solid was dried to constant weight.

Chemical Tests

Aliphatic Alcohols: The ceric nitrate test[3], which indicates the presence of aliphatic alcohols if a color change occurs upon addition of ceric nitrate, was performed by putting ceric nitrate (5 drops) into a porcelain test plate, to which a small amount of the unknown (dissolved in a few drops of acetone) was added.

Aldehydes and Ketones: The 2,4-dinitrophenylhydrazine (DNP) test[4], which indicates the presence of aldehydes or ketones if a precipitate forms upon addition of 2,4-dinitrophenylhydrazine (DNP), was performed by adding a drop of an ethanolic solution of the unknown to a few drops of the DNP reagent in a porcelain test plate.

Aromatic Alcohols: The ferric ion test[5], which indicates the presence of aromatic alcohols if a color change occurs upon addition of Fe^{3+} ion, was performed by adding a drop of an ethanolic solution of the unknown to a few drops of the $FeCl_3$ in a porcelain test plate.

Molecular Weight Determination

A small amount of the unknown acid (about 0.25 g) was dissolved in 200 mL of water upon heating. An acid-base titration was performed with 0.100 M NaOH, using phenolphthalein as an indicator. For thoroughness, the titration procedure was performed three times.

Spectroscopic Analysis

A small amount of the unknown acid on the tip of a spatula added to an NMR tube and dissolved in deuterated acetone in preparation for ^1H NMR analysis. The spectrum was acquired on a Varian 300 MHz NMR spectrometer.

Results

The physical properties, including the appearance, odor, and melting point, of the unknown compound are provided in Table 1.

Table 1: Physical Properties of Unknown Compound.

Appearance of compound	Cream-colored needles
Smell	None
Melting point	135.2 – 138.9 °C

The results of the qualitative solubility tests are provided in Table 2. The color of the resulting solution and the relative solubilities are described.

Table 2: Qualitative Solubility of Unknown Compound.

Solvent	Color of Solution	Solubility
toluene	pale yellow	slightly soluble
acetone	pale yellow	very soluble
methanol	pale yellow	very soluble
water	pale yellow	slightly soluble
dilute HC1	pale yellow	insoluble
dilute NaOH	deep yellow	very soluble

The quantitative solubility of the unknown compound in 0.1 M NaOH is provided in Table 3. The calculation to determine the quantitative solubility from the data is shown in Equation 1.

Table 3. Quantitative Solubility of Unknown Acid in 0.1 M NaOH

Starting Mass	Recovered Mass	Dissolved Mass	Volume NaOH	Solubility
0.205 g	0.095 g	0.110 g	5.00 mL	22 g/L

$$\frac{0.11\,\mathrm{g}}{5.00\,\mathrm{mL}} \times \frac{1000\,\mathrm{mL}}{1\,\mathrm{L}} = 22\,\mathrm{g/L}$$

Equation 1. Calculation of quantitative solubility

The results from the qualitative chemical analyses for the presence of various functional groups (aliphatic alcohols, aldehydes or ketones, and aromatic acids), are provided in Table 4.

Table 4: Chemical Tests for Presence of Functional Groups or Anions.

Test	Observation	Result
Aliphatic alcohols	No color change	negative
Aldehydes or ketones	No precipitate formation	negative
Aromatic alcohols	No color change	negative

The ^{1}H NMR spectrum of the unknown acid is shown in Figure 1. There were four signals in the spectrum: 12.74 ppm (Ha, 1H, broad singlet), 8.70 ppm (Hb, 1H, singlet), 8.54 ppm (He, 2H, multiplet), and 7.79 ppm (Hd, 1H, triplet).

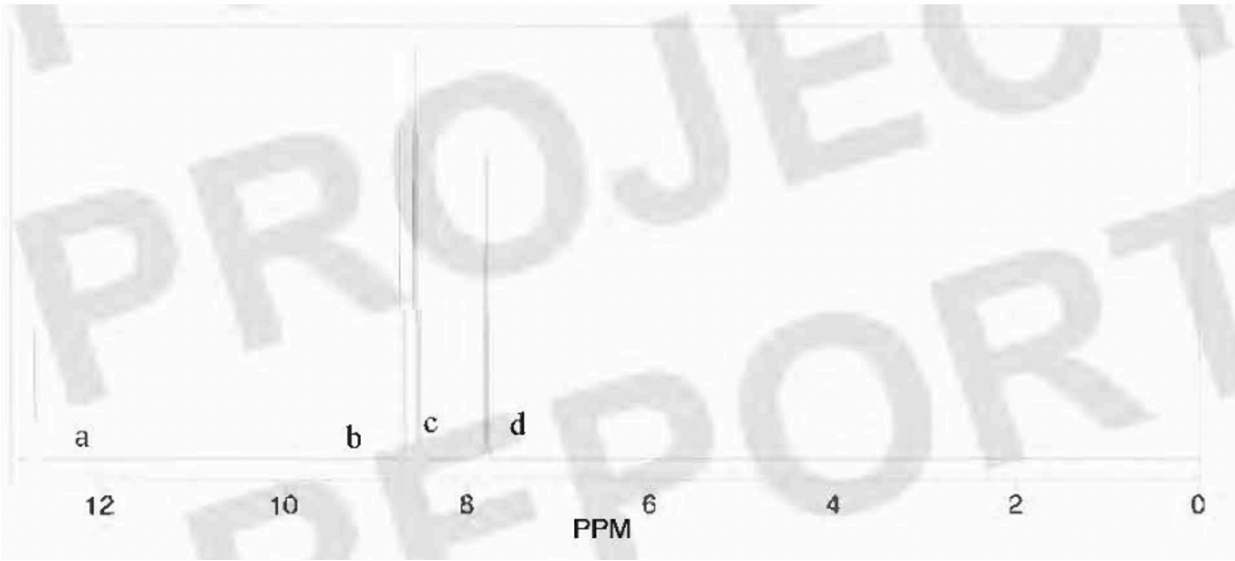


Figure 1. ^{1}H NMR of unknown acid

The data from the titrations, along the resulting molecular weights, are shown in Table 5.

Table 5: Titration Results

Trial	Mass of Unknown Acid	Volume of 0.100 M NaOH added	Molecular Weight
1	0.242 g	14.49 mL	167 g/mol
2	0.251 g	15.12 mL	166 g/mol
3	0.248 g	15.00 mL	165 g/mol

Assuming a monoprotic acid, the results shown in Table 5 were calculated according to the example below:

$$14.49\,\text{mL NaOH} \times \frac{1\,\text{L}}{1000\,\text{mL}} \times \frac{0.100\,\text{M NaOH}}{1\,\text{L NaOH}} = 1.449 \times 10^{-3}\,\text{mol acid}$$

Equation 2. Calculation of moles of unknown acid from titration data

$$\frac{0.251\,\text{g}}{1.449 \times 10^{-3}\,\text{mol}} = 167\,\text{g/mol}$$

Equation 3. Calculation of molecular weight of unknown from titration data

From the three trials, the average molecular weight of the unknown acid was found to be 166 g/mol.

Discussion

Compared to the literature value for the molecular weight of 3-nitrobenzoic acid (167.12 g/mol), the percent error of the experimentally determined molecular weight was 0.6%, as shown in Equation 4:

$$\%\text{error} = \frac{167.12\,\text{g/mol} - 166\,\text{g/mol}}{167\,\text{g/mol}} \times 100\% = 0.6\%$$

Equation 4. Percent error for molecular weight of 3-nitrobenzoic acid

The unknown compound was composed of yellow crystals which had the form of long needles under a magnifying glass. The compound was odorless as indicated in Table 1. Solubility tests indicated that the compound was very soluble in both acetone and methanol, and slightly soluble in water and toluene as indicated in Table 2.

This information indicated that the compound was quite polar in nature since it was not very soluble in toluene which is a nonpolar solvent. The compound was not very soluble in water but was soluble in acetone and methanol, leading to the conclusion was that the compound was a polar molecular compound, supporting the suggestion that this was an organic acid.

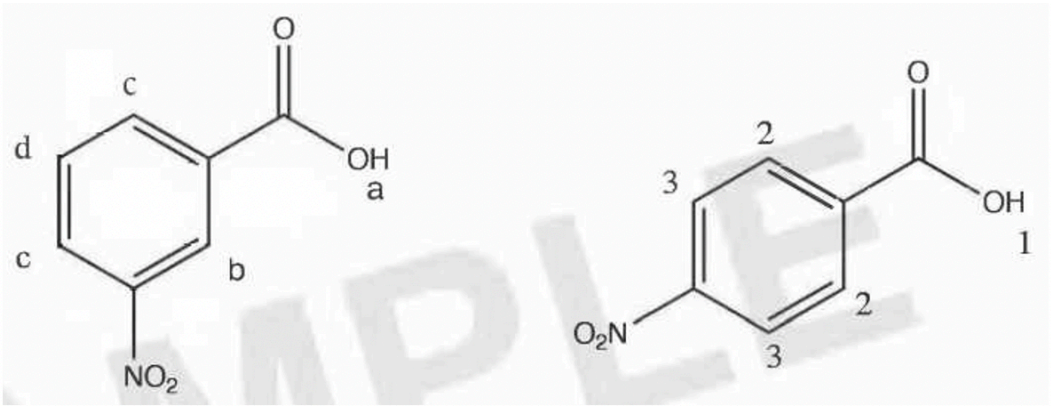
The compound was insoluble in dilute acid (HCl); however, it was soluble in dilute base (NaOH). Solubility in base implies that the compound must have acidic properties. A determination of the quantitative solubility of the compound showed that the solubility was 22g/L in 1.0 M NaOH. The pH of a slurry of the compound was tested and found to be about pH

At first the compound was thought to be insoluble in water, but some must have gone into solution; otherwise the pH of the solution would have been 7. (The pH of pure water was measured with the same pH meter to make sure the meter was functioning properly).

The tests of physical properties gave some insight into the nature of the compound, but further chemical tests were needed to identify the compound. As indicated in table 4, tests for organic functional groups such as aliphatic alcohols, aldehydes and ketones, and phenols were negative. The negative test for aliphatic alcohols ruled out lactic acid and citric acid. The negative test for aldehydes and ketones ruled out pyruvic acid. The negative test for phenols ruled out phenol and salicylic acid. The remaining possibilities were formic acid, acetic acid, and the two nitrobenzoic acids; thus, further analysis was necessary to determine the identity of the acid.

Since the compound was an acid, a titration could provide more information. The acid was assumed to be a monoprotic acid because all polyprotic acids (e.g., citric acid) had been ruled out. The compound was not very soluble in water, but if 0.25 g acid was heated in about 200 mL of water, it would go into solution (at first, the titration was attempted with methanol as a solvent since the acid is much more soluble in this solvent; however, Sabnis et al.[6] indicated that pH titrations are only valid in aqueous solutions). Since all the pH meters were in use, an indicator was used to tell us the endpoint of the titration. An indicator that would change in the pH range of around pH 9-10 was needed, so phenolphthalein was chosen from the chart in the lab manual[7]. As indicated in Table 5, the three titrations led to an average molecular weight for the unknown of 266 g/mol.

This result ruled out formic acid (46 g/mol) and acetic acid (60 g/mol), and it closely matched the molecular weight of nitrobenzoic acid. Because there were two possible nitrobenzoic acids, additional testing was needed to discriminate between them. 3-nitrobenzoic acid and 4-nitrobenzoic acid are isomers; thus, they differ only in the connectivity of the atoms. First, the experimentally determined melting point (135.2 – 138.9 °C) was compared to the literature values for the two remaining possibilities. The melting point of 3-nitrobenzoic acid[8] was 139-141 °C, while that of the 4-nitro isomer[9] was 237-240 °C. Usually, the presence of impurities will lower and broaden the melting point range. From this information, it appeared that our compound was 3-nitrobenzoic acid with some impurities. To confirm this, a ^{1}H NMR spectrum was acquired. As shown in Figure 1, the NMR spectrum revealed four chemically distinct hydrogen atoms. By comparing the structures of 3-nitrobenzoic acid and 4-nitrobenzoic acid (Figure 2), we saw that 3-nitrobenzoic acid has five types of H atoms, while 3-nitrobenzoic acid has only three. At first this was mystifying because the spectrum seemed not to match either compound; however, upon closer inspection, it appeared that the peak at 8.54 ppm was actually two different peaks overlapping, indicating two distinct types of H atoms. Therefore, the spectrum did indeed total five signals, in agreement with what was expected for 3-nitrobenzoic acid. The multiple peaks around 7.79, 8.54, 8.70 ppm (b, c, d) indicates the four aromatic protons on the benzene ring (Hb, He, Hd). The singlet peak at 12.74 ppm (a) refers to the one proton (Ha) on the carboxylic acid group. The splitting pattern follows "N+1" rule, where N refers to the neighboring protons. Integration refers to the number of protons in this specific chemical environment. Due to the presence of conjugation (benzene ring) and the carbonyl group, all protons were deshielded. This explains the large chemical shift.



3-nitrobenzoic acid 4-nitrobenzoic acid

Figure 2. Chemical structures of 3-nitrobenzoic acid (left) and 4-nitrobenzoic acid (right).

Numbers indicate chemically unique H atom locations.

Altogether, the qualitative tests, titration data, and spectroscopic data showed that the identity of the unknown acid was 3-nitrobenzoic acid. To determine how to dispose of the acid safely, the corresponding SDS[8] was retrieved. Using the disposal and toxicity information stated that the compound has an LD50 (oral, rat) of 1950 mg/kg, which indicates that the compound is not very toxic (compared to the sodium cyanide which has an LD50 of 6.4 mg/kg). However, it would be unacceptable to discard it in the trash. Indeed, the SDS states that one seeking to dispose of this compound should "offer surplus and non-recyclable solutions to a licensed disposal company" and "contact a licensed professional waste disposal service to dispose of this material"[8]. Therefore, the research team recommends that the material be placed in a labeled, sealed container until the local professional waste disposal company can dispose of it as an inert organic compound.

Conclusion

The unknown compound was identified as 3-nitrobenzoic acid by a combination of tests of physical properties, such as melting point and solubility, chemical tests, and spectroscopy. The molecular weight was found by titration to be within 0.6% of the actual molecular weight. Since the SDS sheet indicated that the compound was relatively inert and non-toxic, it is recommended that the compound be disposed of by a professional disposal company, but no other special precautions need to be taken.

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This chapter was adapted from Erin Duffy's work in Cooper, M. M. *et al Cooperative Chemistry for Michigan State University*, **2019**.

29.

ORAL PRESENTATIONS

During this course, you will be required to give an oral presentation. This guide is intended to give you some pointers so that your presentations will be more effective.

Giving an oral presentation on a scientific project typically involves presenting a slideshow (e.g., in Microsoft PowerPoint, Apple Keynote, Google Slides, etc.) about your experiment(s). Your slideshow should include slides for each of the major sections that you would include in a formal lab report: **Introduction, Methods, Results, Discussion/Conclusion**. Often, citations for any references that you use, e.g., in the Introduction, are provided as a footnote on the slide containing the information you need to cite.

How Should My Team Present the Slideshow?

Prepare your presentation to take 8-12 minutes, followed by 3-5 minutes for questions from the audience. Each member of your team needs to present some part of the slideshow. You may divide this up as you please, but you should be sure to distribute the amount of content to be presented equally among all team members. One possible way to split up the talk is outlined below:

- **Person 1: Introduction**. Provide the audience with key background information that they need to understand your experiments. Why did your team choose to do the experiments that you will discuss in this talk?
- **Person 2: Methods**. What experiments did you perform? How did you set up the experiments?
- **Person 3: Results**. What happened? How did your initial experiments affect your decisions related to later experiments?
- **Person 4: Discussion.** What did your results mean? What did you learn from those results? Did you include a green chemistry analysis? How could you extend the project further (e.g., what additional experiments would be interesting)?

This distribution of presentation is just a suggestion. Clearly the topics of each person's presentation will change from team to team and project to project.

When planning your talk, imagine that you will be teaching the material you discuss to your audience rather than simply reporting on it. Your points will come across better if you make an effort to explain and reach out to your audience.

What Makes a Good Presentation?

Slide Design

A good presentation has an appropriate balance of text and figures (graphs, diagrams, and/or other graphics). Avoid large blocks of text that you will be tempted to read verbatim; it is often better to write just the key statements that you want your audience to take away from a particular slide. You should still elaborate on those important points but do it verbally—if you prepare and smartly set up your slides (e.g., with the help of animating in things in the order you wish to discuss them), the text you do show should also help remind you of what you wanted to say!

One note about animations: simple animations, such as having text or figures appear as you begin to talk about them, can help guide the audience of where to look on your slide. Be careful with more complicated or excessive animations—that can be distracting to your audience, rather than helpful.

Organization

A well-organized presentation is more likely to be remembered, and the presenter of a well-organized presentation is more likely to be remembered favorably. Even though each member of your team will deliver part of the presentation, you should do your best to make your presentation a cohesive, seamless story. If your listeners can see how your points relate to each other and to your overriding message, those points will carry more meaning.

A Good Attitude

It is okay—even expected—to be nervous presenting in front of your peers, but you should do your best to be engaging. Even if your project is very similar to your classmates' projects, a presentation is always more interesting if the presenter seems interested in what they did and cares about doing a good job.

Being respectful of your peers when they are presenting is also important. Some of your grade for the oral presentation will be based on your interactions with other teams who are presenting their projects. You are expected to pay attention, be polite, and ask questions during the Q&A portion.

Grading

You will be evaluated **as a team** on your slides and **as an individual** on your participation and presentation skills.

CEM 255 Oral Presentation Success Guide

Team & Individual Performance		
Areas to Improve	**Standards for Performance**	**Strengths**
	Organization of Content Is the presentation well-organized with a clear and logical structure? Is the content well-paced and flow smoothly?	
	Sides Presentation Is the key info shown on slides? Does the presentation address goals? Are the slides visually appealing and easy to follow?	
	Presentation Style Does everyone participate in the group presentation? Is the presenter's style engaging, confident and dynamic? Maintaining eye contact and using gestures effectively are recommended.	
	Responses to Questions Does the presenter participate in the Q&A? Does the presenter respond to questions with depth, clarity, and confidence?	

This chapter was adapted from Erin Duffy's work for Cooper, M. M. *et al Cooperative Chemistry for Michigan State University*, **2019**.

30.

POSTER PRESENTATIONS

Scientific posters contain **the same components as the formal lab report** (Introduction, Methods, Results, Discussion, Conclusion, References) but have less text and typically rely heavily on figures (with suitable captions). The text on a poster should highlight key points so that the audience can get the gist of what you did and discovered just by reading the poster, while the finer details and elaboration are provided by the presenter.

How Should I Present My Poster?

A poster presentation is a very social format for presenting scientific projects. You will have many conversations with many people about the work you did. There are essentially two scenarios that play out during a poster presentation:

1. The viewer simply requests, "Tell me about your poster."

In this situation, many poster presenters will have prepared a "canned speech" about 5 minutes in length to guide the viewer through the poster. This speech tells the story of the project, e.g., "We wanted to investigate X (introduction), so we did Y (methods). Here is what happened (results), and this is what each result meant (discussion). Altogether, the results told us Z (conclusion)." Of course, the presenter would replace X, Y, and Z with the details relevant to the project being presented.

2. The viewer asks specific questions about your project.

In this case, you should be prepared to answer any questions relevant to your project.

Possible Formats for Lab Posters

The research poster can be an electronic poster projected on the screen (preferred) or a physical printout that can be pinned to a poster stand or an electronic version projected to the screen. It should be large enough that a small group of viewers can gather in front of your poster to read about what you did and have a conversation with you and your team members about your project.

General Guidelines

- **Text Size**: Your poster text should be readable from a distance of about 3 feet (for titles, about 10 feet), so be sure to size accordingly. Here are some guidelines:
  - Classic Printed Poster Size: 36" x 48"
  - Title: 72-120 pt.
  - Subtitle: 48-80 pt.
  - Section headers: 36-72 pt.
  - Body text: 24-48 pt.
- **Colors**: Like with the size of your text, you should choose colors that make your poster easy to read. For example, you need to provide enough contrast between the text and background; for printed documents, typically you should use a dark text on a light background. Some choose to use light text on a dark background. The visual presentation of text and images of text in the digital resource has a color contrast ratio of at least 4.5:1 for accessibility reasons.
- **Images**: Images do not rely solely on color to convey information.

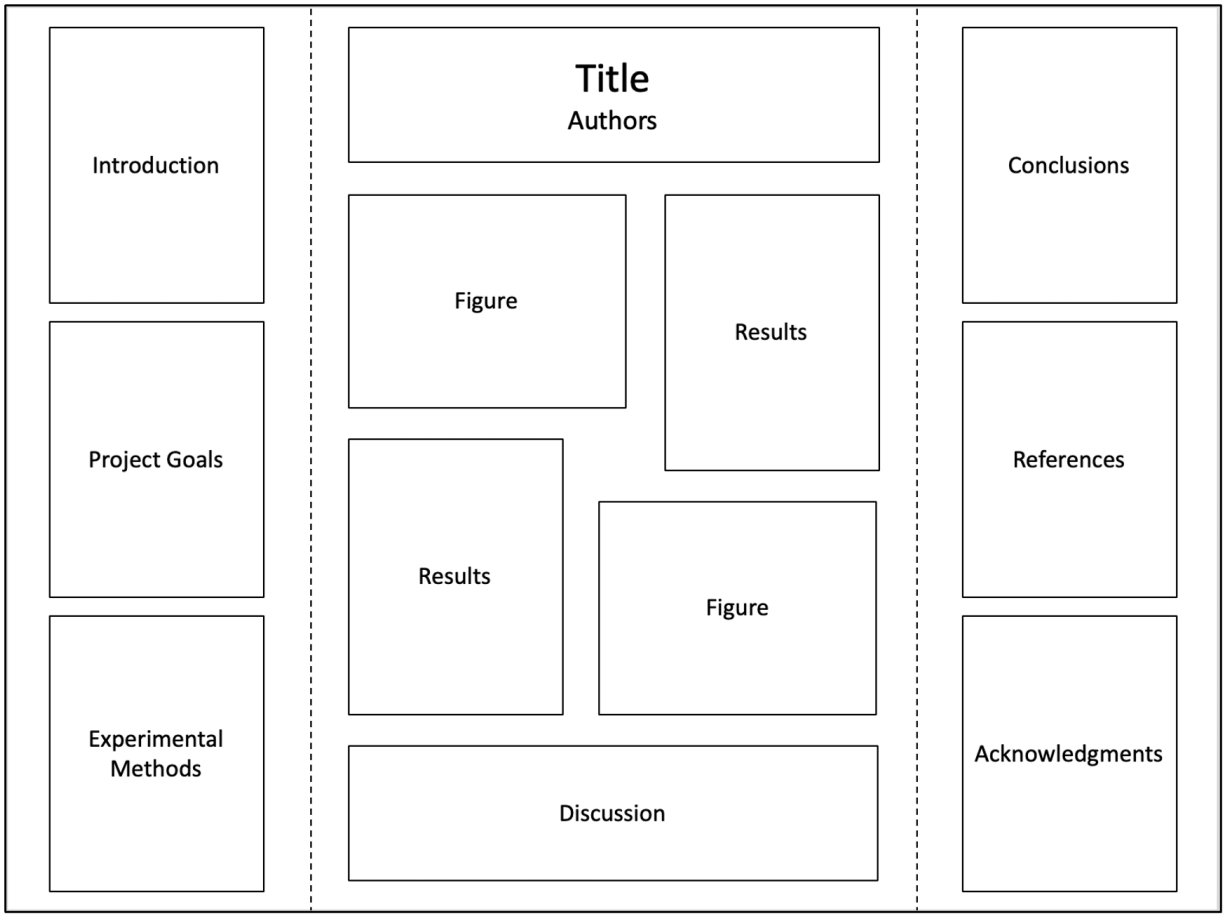
Software to Design Your Poster

Guidelines in this document will be based on **Microsoft PowerPoint**, which is commonly used to make research posters. Other slide presentation programs, such as Apple Keynote or Google Slides, may also work. Alternatively, software for print publications, such as Adobe InDesign, may be used.

Designing a Poster in PowerPoint

- **Slide Size:** The default slide size is not big enough for a poster. To set the slide size, go to **File >> Page Setup** and set the **Width** at 46 inches and the **Height** at 36 inches. This allows for 1-inch margins on all sides. Your **entire poster** should be designed in this single slide.
- **Consider Using a Template**: Research poster templates are available online, such as these free downloads from MSU College of Arts & Letters: Presentation Templates or Colin Purrington: Designing Conference Posters (note that you will have to resize the template to fit our poster bulletin boards; see "Slide Size" above). This may be a good option if your team does not feel comfortable starting from scratch in PowerPoint.

An example poster format is shown below.



Grading

You will be evaluated **as a team** and **as an individual** on your participation in the presentation, knowledge of project background, and understanding of the project.

CEM 255 Poster Presentation Success Guide

Team & Individual Performance		
Areas to Improve	**Standards for Performance**	**Strengths**
	Organization of Content • Is the presentation well-organized with a clear and logical structure? • Is the content well-paced and flow smoothly?	
	Sides Presentation • Is the key info shown on the poster? • Does the poster presentation address goals? • Is the poster visually appealing and easy to follow?	
	Presentation Style • Does everyone participate in the group poster presentation? • Is the presenter's style engaging, confident and dynamic? • Maintaining eye contact and using gestures effectively are recommended.	
	Responses to Questions • Does the presenter participate in the Q&A? • Does the presenter respond to questions with depth, clarity, and confidence?	

This chapter was adapted from Erin Duffy's work for Cooper, M. M. *et al Cooperative Chemistry for Michigan State University*, **2019**.

31.

DECISION MEMO

In addition to the assignments for the laboratory portion of the course, the case studies will focus on forms of scientific communication more common in industry or policy spheres.

The Decision Memo assignment completes Case Study 1: Comparing Synthetic Routes. In this assignment, your team will use your discussion and responses to the case study questions within beSocratic to formulate a short memo addressing the problem, stakeholders, and criteria laid out in the Case Study Scenario.

This assignment is distinct from previous versions in that (A) you will not be carrying out any experiments and (B) the structure of a memo is styled differently.

- While you will use Green Chemistry Tools and Metrics to evaluate the information presented in the case study, you will not be performing any experiments to collect more empirical data than provided.
- The memo is styled to forefront the conclusions and recommendations of your analysis. In the limited time with the attention of policy makers in industry and government, a concise, upfront summary of findings is more effective than the structure of an academic paper. Don't bury the important finding; make it the star of the memo!

Structure of a Decision Memo[1]

This structure reflects the goal of summarizing the findings/decisions and the necessary background and evidence into a single page that is easy to peruse.

Main Point(s) and Decision(s)

These memos get straight to the point and put the most important points, decisions, and/or recommendations up front.

1. Revised from White, P.; Chien, D.; Pomeroy, D. "Policy Memo" MIT Communications Lab

Background

In this section, a concise summary of the necessary background is presented. You should present this information as if your audience has no previous experience with the topic and use this paragraph (two paragraphs max) to bring them up to speed with not only the problem but the chemistry underpinning the problem.

This section should summarize the problem your team has defined from the case study scenario, the key stakeholders in the problem and their needs, and on what criteria an acceptable solution will be evaluated. Your team should concisely describe the general explanatory model of the underlying chemistry that you generated in session 1 of the case study.

Evidence

Your team should compile the evidence you've generated through your reading of any recommended papers and the insights gained through your beSocratic activities. This section should directly address the problem definition from your background section: for each set of stakeholders, what is the relevant data for their needs (from the Scenario at the beginning of the Case Study)? Start with your strongest, most convincing point. Cite the evidence and connect it to the Solution (which synthetic route) you recommend.

This section could be separated into bulleted paragraphs to convey main points for each stakeholder; use figures/tables only when appropriate to support your point. These short paragraphs can be full text, integrate a small table, or be coupled with a figure.

Conclusions and Implementation

Restate your recommendation as a take-away message. Based upon the evidence presented and the problem you've been evaluating solutions for, provide a brief description of how you would recommend the stakeholders proceed in light of your recommendation. Which route should they implement, and are there any specific data points they should monitor as they proceed?

Example from MIT Communication Lab[2]

This policy memo example (PDF) demonstrates the structural organization that puts the main point(s) up front, includes a concise summary of the relevant background information, and supports the main decision and conclusions with a written summary of the evidence. A figure is not always necessary, and if used, limited to one clear, easy-to-read figure.

Decision Memo Template

Names of contributing members: [Insert Group Names Here]

Recommendation: Communicate your decision as to which synthetic route the pharmaceutical should utilize as they prepare for their upcoming EPA audit. **Insert main point(s), decisions, or recommendations here.**

Background: Provide a relevant, concise background that summarizes the problem, stakeholders, and key criteria, **and** briefly explains the key underlying chemistry.

1. Defines the problem,
2. Describes the social and/or environmental context for why the problem matters,
3. Defines the chemistry of the physical system and its components (i.e., the molecules involved in the reactions),
4. Identifies to whom the problem matters (i.e., the stakeholders) and who/what is affected by the problem (e.g., human and/or environmental health), and
5. Specifies the criteria that must be addressed for an acceptable solution to the problem.

Evidence: Summarize key evidence needed to address problem and stakeholders, prioritizing the most relevant evidence. Use one figure or table as needed, but it is not required. Figures or tables should be easy to read from a glance, including defining any abbreviations.

1. Generate a list of important criteria for acceptable solutions to the problem.
2. Gather the data/evidence and scientific information related to the criteria.

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2. White, P.; Chien, D.; Pomeroy, D. "Policy Memo" MIT Communications Lab

3. Analyze the strengths and weaknesses of each solution with respect to the criteria. Explicitly acknowledge and justify any tradeoffs made in choosing an optimal solution (costs and benefits of possible solutions).
4. Provide an evidence-based decision and provide reasoning for your choice using data/evidence and scientific information.

Conclusion & Implementation: End with a summary of the conclusions your team has drawn and briefly describe the next steps for implementing your recommendation. **Bold the final sentence containing the take-home message**.

Decision Memo Success Guide

This guide will be embedded into D2L for the lab instructor to evaluate your memo. They will provide you feedback in the comment portion on D2L of any strengths of your argument. The decision memo is a collaborative effort worth 20 points total, graded holistically.

	Goals for Decision Memo	Strengths & Suggestions for Improvement
Main Point(s) and Decision	• Did the team reference the key problem in a concise fashion? • Did the team synthesize their findings into concise main point(s), decisions, and/or recommendations?	
Background: Problem Definition	Did the team identify the problem, key stakeholders, and the criteria needed to evaluate potential solutions?	
Background: Explanatory Model of Chemistry	Did the team summarize their understanding of the key chemistry underpinning this problem?	
Evidence Summary	• Did the team summarize the evidence to evaluate the potential solutions to the above-defined problem? • Did the team prioritize evidence that supports their conclusions? • Does the evidence address key stakeholder concerns?	
Evidence Figure/Table (Optional)	If included, does the figure or table summarize evidence effectively? Is it easy to interpret at a glance?	
Conclusion & Implementation	Did the team outline a blueprint for implementing their recommendation?	
References	Do they include references in-text?	

32.

POLICY PAPER

This type of scientific communication is typically known as a "white paper", to refer to an official government report and the type of paper it was printed on. These papers are typically regarded as authoritative and informative, while still taking a specific, principled stance on an issue.[1] Policy papers can be commonly written by or sponsored by business and/or industry partners and targeted towards policy makers in government and academia. These types of policy papers are compiled by publishers like C&EN (Chemistry & Engineering News) or CAS.

Structure of a Policy Paper[2]

Unlike the Decision Memo, a Policy Paper starts with the big picture and funnels the reader's attention to the proposed solution. The expected paper length is 2–4 pages, as needed to make your policy proposal. Using the following headings will help your non-technical audience digest your argument.

Executive Summary

Your executive summary should contain a title that states the problem to be solved and your position. For instance, instead of "Teaching Students Green and Sustainable Chemistry", a more descriptive title would be "Engaging Students Green and Sustainable Chemistry: A case study approach to defining sustainability problems and designing green chemistry solutions."

The text of this 1-page summary should concisely summarize the scope, boundaries, and severity of the sustainability problem. It should outline key stakeholders and/or criteria or constraints on the solution design. Finally, it should preview the rest of the paper by (1) stating outright what the proposed solution is and (2)

1. Purdue Online Writing Lab (OWL). "White Paper: Purpose and Audience" https://owl.purdue.edu/owl/subject_specific_writing/professional_technical_writing/white_papers/index.html

2. Purdue Online Writing Lab (OWL). "White Paper: Organization and Other Tips"

predicting what the outcome would be of adopting the proposed solution based on the analysis you conducted in beSocratic.

Problem Definition and Background

Your team should use Case Study 2 Problem Scenario and Background to provide the policymaker with an understanding of the current state of the sustainability problem. What is the general background needed to understand what is going on and how we got here? What is the underlying chemistry of this sustainability problem, and how does it work? Be sure to include a reaction mechanism as appropriate, explaining how and why the reaction occurs. Is the chemical contribution to the problem based on where the chemicals are sourced from (beginning-of-life) or what happens with the post-consumer waste (end-of-life)?

With this general background, use your understanding of the Case Study to define and analyze the problem. Who are the stakeholders? What are the criteria for an acceptable solution? What constraints or issues make this problem challenging to solve?

Summarize the potential solutions your team investigated in the beSocratic activity and your methods for analyzing those solutions. What are the opportunities to tackle this sustainability problem? What would be the challenges, strengths, and implications of each proposed solution?

Recommended Solution

Briefly, use your analysis to provide your recommendation. How would you propose lawmakers move forward with the solution you advocate for? What are the limitations, and how might future scientists and advocates address these concerns?

Conclusion

Summarize your analysis and restate your recommendation.

References

Include a set of references in ACS citation format.

Policy Paper Success Guide

This guide will be embedded into D2L for the lab instructor to evaluate your policy paper. They will provide you feedback in the comment portion on D2L of any strengths of your argument. The policy paper is a collaborative effort worth 20 points total, graded holistically.

	Goals for Policy Paper	**Strengths & Suggestions for Improvement**
Executive Summary	• Did the team reference the key problem in a concise fashion? • Did the team synthesize their findings into concise main point(s), decisions, and/or recommendations?	
Background: Problem Definition	Did the team identify the problem, key stakeholders, and the criteria needed to evaluate potential solutions?	
Background: Explanatory Model of Chemistry	Did the team summarize their understanding of the key chemistry underpinning this problem?	
Summary of Proposed Solutions	• Did the team summarize the evidence to evaluate the potential solutions to the above-defined problem? • Does the evidence presented address key stakeholder concerns?	
Recommended Solution	• Does the team justify their recommendation? • Did the team prioritize evidence that supports their conclusions?	
Conclusion & Implementation	Did the team outline a blueprint for implementing their recommendation?	
References	Do they include references in-text? Do they have a reference list at the end of the paper?	

33.

POLICY BRIEF

Structure of a Policy Brief

The goal of a policy brief is to inform decision-makers about a specific problem or issue and provide evidence-based recommendations for action. Just like the Policy Paper, your Policy Brief will start with defining the problem and funneling the reader's attention to the proposed solution. This Policy Brief will be focused on presenting research findings or policy recommendations on fluorogel remediation method for maximum impact. The expected paper length is 1 – 2 pages, as needed to make your policy recommendation, targeted at policy makers or stakeholders.

Example from MIT lab (PDF)

Policy Brief Template

Names of contributing members: [Insert Group Names Here]

Recommendation: Communicate your decision as to where you would strategically place the fluorogel remediation method for maximum impact to the local Michigan system diagram.

Background: Use the Background section to provide the audience with an understanding of the current state of the PFAS sustainability problem. The Background section can be about 1 paragraph in length and should address the points outlined below:

1. What is the general background needed to understand what is going on and how we got here?
2. What is the underlying chemistry of this sustainability problem, and how does it work?

Defining the Problem: With this general background in mind, use your understanding of PFAS from Case Study 3 to define the problem. The Defining the Problem section should be about 1 paragraph in which you should:

1. Define the problem,

2. Describe why it is a problem,
3. Identify the stakeholders associated with the problem, and
4. Specify the criteria/constraints that must be addressed for an acceptable solution.

Evidence: Present the key evidence used to evaluate the possible solutions to the problem, including any necessary data
your group utilized to design your own proposed solution. In your evidence section, be sure to:

1. Specify the criteria/constraints you used to make your decision,
2. Include the appropriate data that presents a systematic evaluation of solutions,
3. Analyze the strengths and weaknesses of each solution with regards to the criteria/constraints, and
4. Provide an evidence-based decision of your solution to the problem.

Conclusion & Implications: Summarize your analysis by briefly addressing the following:

- Restate the problem you defined for Case Study 3.
- Summarize the potential solutions you investigated in the beSocratic activity.
- What would be the challenges, strengths, and implications of each proposed solution?
- What are areas for future improvement as it relates to the sustainability problem highlighted in Case Study 3?

References: Be sure to include any references you cited in this final section of the Policy Paper. You can use the ACS citation format as outlined here: ACS Style Quick Guide

Policy Brief Success Guide

	Goals for Policy Brief	Strengths & Suggestions for Improvement
Main point	Did the team reference the key problem in a concise fashion? Did the team synthesize their findings into concise main point(s), decisions, and/or recommendations?	
Background: Problem Definition	Did the team identify the problem, key stakeholders, and the criteria needed to evaluate potential solutions?	
Background: Explanatory Model of Chemistry	Did the team summarize their understanding of the key chemistry underpinning this problem?	
Summary of Proposed Solutions	Did the team summarize the evidence to evaluate the potential solutions to the above-defined problem? Does the evidence presented address key stakeholder concerns?	
Recommended Solution	Does the team justify their recommendation? Did the team prioritize evidence that supports their conclusions?	
Conclusion & Implementation	Did the team outline a blueprint for implementing their recommendation?	
References	Do they include references in-text? Do they have a reference list at the end of the paper?	

34.

PEER AND SELF EVALUATIONS

Peer and Self Evaluations

At the end of each project, you will be asked to **evaluate** yourself and your team members on how well each person contributed to the team. These evaluations will be completed using CATME.

Logging in to CATME

Your account should create automatically when your instructor sets up a course in CATME. If you have never used CATME before, then you should get an email with instructions about how to log in.

If you have used CATME previously for another class at MSU, you should login with your institutional email address: **netid@msu.edu**. If you have forgotten your password, click the "Forgot your password?" link underneath the login fields.

Account Dashboard

When you log into your account, you will be taken to a **Summary** page. Any assignments will be listed in a table, such as the one shown in the figure below:

Complete the survey in a timely manner to receive credit. Practice sessions do not count as completing the survey.

PART VIII

APPENDIX

35.

IDENTIFICATION OF AN UNKNOWN ORGANIC SOLID–PLANNING SCENARIO AND BACKGROUND

Scenario

In your new job as a chemical safety aide for MSU's Office of Environmental Health and Safety (EHS), your team encounters an organic research laboratory that has recently "found" an unlabeled jar of a powdered mixture in the back of one of the chemical hoods. The lab manager has narrowed down the possibilities of what the organic powder might be, as listed below:

Possible Compounds

acetanilide	benzoic acid	succinimide	adipic acid	benzilic acid
oxindole	benzamide	n-phenylsuccinimide	salicylic acid	cinnamic acid

Your supervisor tasks your team with testing the powder, purifying it and identifying it. Since your team is responsible for safely disposing of the solid, you will also need to determine as many of its physical and chemical properties as possible to help prevent potentially dangerous interactions with the other waste in the facility.

Before you can identify the unknown, you will need to perform a recrystallization to ensure the purity of the sample. In order to perform the recrystallization, your team will need to perform solubility test in order to select the optimal solvent for your unknown.

Tasks for successfully completing this project

1. Perform solubility test of your unknown at high temperature and at low temperature. Choose the best solvent and binary solvent system for recrystallization.
2. Perform a single-solvent recrystallization and a binary-solvent recrystallization using solubility test results.
3. Use two qualitative and two quantitative identification techniques to identify of the unknown.

4. Share data with your group members and report the identity of your unknown to your TA; assess the validity of their claims based on the evidence from the identification methods.

You will be weighing out **three grams** of the compound; use it wisely! You and your TA will not be told the identity of your compound, nor will you be given any other information about it.

Safety notes:

- Be sure to consult the MSDS/SDS for any compound that you might work with.
- Wear safety goggles, gloves, and appropriate clothing at all times in the laboratory.
- Dispose of waste in the labeled containers. **Do not** pour any waste down the drain unless you check with your instructor first.
- Use great care when transferring solutions of acids and bases.
- If you spill a strong acid or base on your clothes or skin, rinse with large amounts of water immediately and ask one of your team members to tell your TA.
- All of the unknown compounds that you will work with in this project are Generally Recognized as Safe, but normal safety precautions should be observed.

Techniques you may need to learn or review

Consider reviewing the following sections in the Lab Manual:

- Measuring solids and liquids
- Purification via recrystallization
- Solubility test at different temperatures
- Measuring solution pH
- Melting point determination
- FTIR spectroscopy
- 1H NMR spectroscopy

Project Checklist

Use these questions as a guide to complete required tasks and lab documents. This planning scenario and background document should be uploaded to D2L for grading.

Optimization of solvent system

- **Solubility Test**: Five greener solvents are provided below. Your team must test the solubility for the unknown in all pure solvents at different temperatures and summarize the result in the solubility chart next week.

	Water	**Ethanol**	**Ethyl Acetate**	**Toluene**	**Heptane**
Low temperature					
High temperature					

Note: you do not need to fill out this table. It is only a reference table to help you prepare for next week's experiment. Think: how do you plan to collect the data?

- For each solvent that you will test, indicate whether the unknown is soluble when heated, when cooled and at room temperature. How can you control the temperature? What will occur if the temperature is too high?
- Based on the results of the example table below, which is the best solvent for recrystallization? What makes it a good solvent?

Example	**Solvent A**	**Solvent B**	**Solvent C**
Ice bath	High solubility	Medium solubility	Low solubility
Room temperature	Medium solubility	Medium solubility	Medium solubility
Warm water bath	Low solubility	Medium solubility	High solubility

- You need to write a procedure for finding the solubility of your compound. What information will you need to determine this? What factors affect solubility?
- Some organic solvent you used are highly flammable, for example, heptane. Be careful! How would you warm it up?

Recrystallization

- For single solvent recrystallization, each team must test at least one pure solvent for the unknown. For multi-solvent recrystallization, each team must test at least one binary mixture of solvents. Remember the two solvents you chose for multi-solvent recrystallization should be miscible. Indicate which solvents or solvent systems your team will try.
    - For single-solvent recrystallization, what factors did you consider in this selection? How will you manipulate these to induce crystallization?
    - To guide your thinking, consider organizing information on each solvent's general polarity and boiling point, or any relevant properties of the selected solvents. Take the boiling point of each solvent into consideration.
    - For multi-solvent recrystallization, what factors did you consider in this selection? How will you manipulate these to induce crystallization?
    - What are the general steps for a single-solvent recrystallization and a binary-solvent recrystallization?
- Why should we be concerned about purity of the unknown before we attempt to identify it? How would an impurity affect the results of your identification techniques?

Identification of Unknown

- You will choose four techniques to analyze the unknown; justify those selections and describe how you will use the information from each technique to identify your unknown. Are they qualitative or quantitative techniques or both? Why?
- Based on the four chosen identification techniques, hypothesize how impurities could affect the results of these techniques. Be detailed.

Chemicals (add rows as needed)

Chemical Name	**Hazard(s)**	**Accidental Release Measures**

Equipment

Generate a detailed list of equipment you need, hand-draw the apparatus set-up for solubility test and recrystallization.

Project Plan (Procedures)

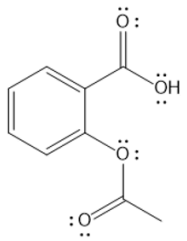
Write a preliminary plan for your experimental procedure for each week. Indicate what each person in your group will do to solve the problem, and what data they will record. Break down the work so you use your time effectively and that *each person will end up with a mini lab report for their data and notes* assignment next week. Be detailed.

36.

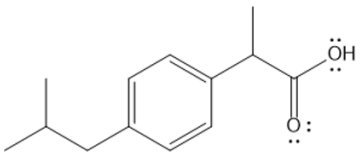
SYNTHESIS AND ANALYSIS OF PAINKILLERS–PLANNING SCENARIO AND BACKGROUND

Scenario

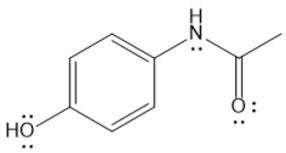
Your team has been employed by a drug development division of a start-up company that has decided to enter the arena of manufacturing over the counter (OTC) painkillers (also known as analgesics). Common ingredients for analgesics may include:



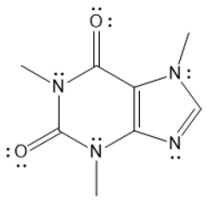
Aspirin



Ibuprofen



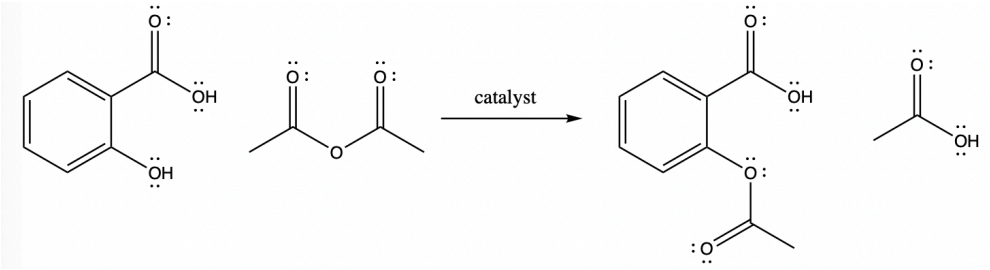
Acetaminophen



Caffeine

Lewis structures of aspirin, Ibuprofen, Acetaminophen and Caffeine

The first and most commonly produced pain reliever was aspirin, and it is frequently featured in various formulations of analgesics. Although it is quite easy to synthesize, pharma start-ups like the one you're employed at must balance the demand from consumers and hospitals, regulatory pressures, and profit margins to justify their investment.



Reaction scheme of aspirin synthesis.

The start-up company has tasked your team with looking for a low-cost green alternative to satisfy EPA requirements and shareholders. Your team is going to explore ways to synthesize aspirin (as shown below) in order to achieve the highest yield, the greenest synthesis, and the most economical synthesis.

After your team has optimized the synthesis of aspirin to satisfy the conditions above, your second task is a consultation with Ingham County Health Department. Patients have been reporting to the emergency rooms in the greater mid-Michigan area after taking a generic OTC analgesic. Many analgesics have more than one active ingredient (or component). Each team member will be given a sample of the generic analgesic and asked to analyze it. You will need to report what the active ingredients are in the binary-mixture sample (part 3). These results will be used to trace the production issues.

Tasks for successfully completing this project

1. Determine which catalyst is greenest and most economical to synthesize aspirin in high yield and provide confirmation of the purity.
2. Use three identification techniques to prove the isolated product from the synthesis is the intended product.
3. Compare the four different routes from a green chemistry perspective: efficiency, safety, waste prevention, cost, and greenness.
4. Analyze the sample of generic OTC analgesic using thin layer chromatography (TLC) to identify the active ingredients in the binary mixture.

Special materials available for this project

In addition to the necessary reagents, salicylic acid and acetic anhydride, your team will test the following catalysts:

Catalysts

Phosphoric acid	Sulfuric acid	Sodium acetate	γ-valerolactone

For your consultation with the Ingham County Health Department, a preliminary screening has indicated that the following components are the most likely active ingredients in the generic:

Potential Components in the Generic OTC Analgesic

Aspirin	Acetaminophen	Acetanilide	Caffeine

Safety notes:

- Be sure to consult the MSDS/SDS for any compound that you work with.
- Wear safety goggles, gloves, and appropriate clothing at all times in the laboratory.
- Dispose of waste in the labeled containers. **Do not** pour any waste down the drain unless you check with your instructor first.
- Use great care when transferring solutions of acids and bases.
- If you spill a strong acid or base on your clothes or skin, rinse with large amounts of water immediately and ask one of your team members to tell your TA.
- Salicylic acid is toxic and an irritant. Avoid contact with skin, eyes, and clothing. Avoid breathing the dust.
- Acetic anhydride is toxic, corrosive, and a lachrymator (causes tears). Wear gloves and use in the hood. Avoid contact.
- Sulfuric acid is corrosive and causes burns. Avoid contact.
- The aqueous filtrate from the crystallization step can be diluted with water and poured down the sink.

Techniques you may need to learn or review

Consider reviewing the following sections in the Lab Manual:

- Purification via recrystallization
- Isolation of crude product via vacuum filtration
- Determination of purity
- Separation of components via Thin Layer Chromatography (TLC)
- Identification techniques
- Spectroscopy

Concepts you may need to learn or review

The following sections from the OCLUE (CEM 251/252) textbook may be helpful:

- Chapter 7: Nucleophilic Attack at the Carbonyl Carbon, especially the Esterification section (pages 135-136).
- Chapter 2: Spectroscopy, especially IR (pages 34-39) and 1H-NMR (pages 46-49)

Additional Background

Your literature search has yielded this general procedure for the esterification reaction.

Esterification

- Catalyst: Phosphoric acid, sulfuric acid, or γ-valerolactone

Place 1.0 g salicylic acid and a stir bar in a small beaker, add 2 mL of acetic anhydride (with care in the hood) carefully while stirring the mixture. Add 5 drops of your chosen catalyst. After about 10 minutes place the beaker in a hot water bath so that any remaining solid dissolves and the reaction goes to completion. Pour the resulting solution onto about 20 mL of ice water, wait for crystallization, vacuum filter and dry.

- Catalyst: sodium acetate

Add 1g salicylic acid, 1.00 mL acetic anhydride, 0.3 g sodium acetate and a stir bar into an Erlenmeyer flask or a beaker (with watch glass on top). Stir the reaction at 55 °C for 50 minutes. Add 20 mL ice water into the reaction mixture. Perform vacuum filtration and dry the solid.

Note: Do not copy and paste the procedures above directly onto your notebook. Use your own words (or drawing).

Using Green Metrics and Tools

In your course D2L page, there are links to Green Metrics and Tools. Use appropriate green chemistry metrics or principles to compare the efficiency, safety hazard, waste prevention, costs, and impact of different synthetic routes.

Project Checklist

Use these questions (1) as a guide to determine if you have completed all of the required tasks and (2) to help you to be able to develop a good report.

Part 1: Synthesis of aspirin

- Draw the structures of the catalysts and label the functional groups. For each catalyst, is this molecule acidic, basic, or neutral?
- In your group, design an experimental protocol that will allow you to investigate which catalyst for the aspirin synthesis that (1) will produce the highest yield, (2) is the most economical (price reference is provided as a document), and (3) provides the greenest synthesis. Hint: refer to SDS information of all catalysts used.
- There are many ways to monitor the progress or completion. Propose a theoretical method.
    - o Esterification reaction is exothermic. Student L plan to use a rise in temperature to monitor reaction progress. The rate of temperature change can be used to determine the fastest reaction. Would this work? Why or why not?
- How will you control the temperature (55 °C) for the esterification reaction using sodium acetate as catalyst?
- How will you prepare ice water for recrystallization? Remember: the water you added in your solution must be DI water. Ice from ice machine is tap water.
- How will you isolate your product after the reaction? How can addition of ice water assist with precipitation/crystallization? Will the aspirin product be a solid or liquid?
- Which green metrics will you evaluate the various catalysts on? What information will you need to record in order to calculate these?

Part 2: Recrystallization and purity testing of aspirin

- Draw the structure of aspirin. Label out the polar bonds.
- What solvent (solvent mixture) do you think it might be soluble in? Consult the green solvent guide in the lab manual to make your selections.

- According to your online literature research, which solvent is most suitable for recrystallization of aspirin?
- Describe **three** methods that you will use to prove that the product you isolated from the reaction is indeed the product you expected and not, for example, starting material.
- What data do you need to collect for the following green chemistry comparison? Note that you will need to use at least 4 green chemistry metrics/principles to evaluate the fourth synthesis.
- Calculate the percent yield and atom economy for each reaction. What data do you need to collect for calculation of limiting reagent and percent yield? How will atom economy change using different catalysts?
- Perform an economic analysis using the Cost Analysis Tool and the EcoScale rubric on each method. (Cost of each reagent was in a file on D2L Project 2 folder.)
- Which green metrics will you evaluate the waste created in each route? How would you estimate the waste created in each route?
- What recommendation would you make to the start-up company and justify that recommendation using the three metrics.

Part 3: Analysis of multicomponent analgesics

You will be given a sample which is a binary mixture of the analgesic from the Ingham County Health Department. You also have the pure standards of the potential components from the scenario (acetaminophen, caffeine, acetanilide) and aspirin product that was purified in week 2. Perform a thin-layer chromatography (TLC) experiment to identify the unknown binary mixture.

- Thin Layer Chromatography (TLC) techniques is widely used in organic synthesis for monitoring reaction completion and purity test. How does it work?
- What is R_f value and how can you calculate it?
- Using knowledge in polarity, which of the four standard reagents will have the highest R_f value and the lowest Rf value?
- Using the standards of the pure analgesics provided, how will you identify what your mixture contains?

Chemicals (add rows as needed)

Chemical Name	Hazard(s)	Accidental Release Measures

Equipment

Generate a detailed list of equipment you need, hand-draw the apparatus set-up for aspirin synthesis and TLC.

Project Plan (Procedures)

Write a preliminary plan for your experimental procedure for each week. Indicate what each person in your group will do to solve the problem, and what data they will record. Break down the work so you use your time effectively and that *each person will end up with a mini lab report for their data and notes* assignment next week. Be detailed.

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