McNeil Group Handbook

Policies, Procedures, and Guidelines

Last Updated: April 2020

http://mcneilgroup.chem.lsa.umich.edu/

Lab Safety

General

- Make sure you complete the UM-OSHA Comprehensive Laboratory Safety Training course and read the Chemistry Department Safety Manual before initiating research. Upload a copy of your completion certificate to the group server prior to starting lab work.

- Make sure you read and sign all lab-relevant SOPs (binder in kitchen).

- Make sure that you complete the safety checklist (next page) and send a signed copy (PDF) to Prof. McNeil before beginning research. Upload a copy onto the group server as well.

- Notify the group safety officers AND Anne immediately if you have been injured or spilled a toxic, caustic, or flammable compound.

- Lab coats and safety glasses must be worn when doing work, either at your bench, hood, or sink OR if you are talking/standing next to someone who is working at the bench, hood, or sink. If you see someone working without a lab coat and/or safety glasses, please remind them of the appropriate personal protective equipment needed for working in the lab. If the problem persists, please notify Anne.

- Open-toed shoes and shorts/skirts (without leggings) cannot be worn in the lab.

- Headphones (even single-ear wearing) are not allowed in the lab.

- Notify group members when you are leaving for the night. This habit helps to ensure that nobody is working alone!

- For additional safety resources, please consult the “safety information” section of the group website.

Guidelines for a Safe Working Area

Bench and Hood Area

- Your workspace should not be cluttered. You must be able to place a new vial/flask/beaker on your bench. All reagent and solution bottles must be clearly labeled without use of chemical abbreviations.

- Nothing can be hanging off the edge of benches or shelves. Flammable solvents and reagents cannot be located within 18" of the ceiling.

- No objects can be within the back 4" of your hood unless it is on a shelf. Check that all water, air and N_2 lines are secured with copper wire. Ensure all tubing and power cords are free of defects.

- All chemical waste must be clearly labeled with no chemical abbreviations and dated. Chemical waste should be capped when not in use. Glass containers containing chemical waste within 4' of a drain must be in a secondary container.

Instrument Room and Shared Space

- If you are responsible for an instrument that generates chemical waste, ensure that the waste bottle is properly labeled, dated, placed in a secondary container and has an appropriate cap.

- Claim any chemicals left in the balance area and return them to the proper place.

Emergencies

Call the Department of Public Safety by dialing **911** from a campus phone or **734-763-1131** from a cell phone. Call **Chris Peters** (Departmental Lab Safety) at **734-763-4527** or **chrpeter@umich.edu** or **Tracy Stevenson** (Departmental Lab Safety) at **734-764-7316** or **steventi@umich.edu**.

Lab Safety Checklist

Please complete the following checklist with the group safety officer, and return a signed, scanned copy as a pdf file to Anne before beginning to work in the lab. Also upload a copy to the group server.

- ❑ I have received safety glasses and a lab coat and agree to wear them at all times when in lab. I am aware of acceptable clothing to wear in lab.

- ❑ I am aware of the location and operation of the safety shower, eye wash, fire extinguisher, blast shield, and fire alarm in ALL of the group laboratory rooms.

- ❑ I am aware of the University of Michigan chemical hygiene plan and the group-specific hygiene plan, and have read and signed all SOPs and I have become familiarized with their contents.

- ❑ I have completed the OSHA safety training.

- ❑ I am aware of emergency phone numbers and department contact numbers in the event of an accident, chemical spill, or other emergency.

- ❑ I know how to access MSDS (material safety data sheets), and I will refer to these if I have any questions about the safe handling of any reagent.

- ❑ I have access to OneNote lab notebook and I am aware of the lab protocols for keeping complete and accurate records of my research. I have shared this notebook with Anne & all group members.

- ❑ I will conduct my research with honesty and integrity and will not intentionally fabricate or misrepresent any scientific data.

- ❑ I have viewed the group job list and will consult the appropriate person before using any equipment for which I have not yet received proper training.

- ❑ I will maintain a safe and clean work environment, will properly label and dispose of hazardous materials, and will safely store and handle all chemical reagents.

- ❑ I will be properly trained (i.e., read the SOP and talk to an experienced user) before handling any new (to you) compounds.

- ❑ I will seek advice from experienced group members about all new procedures, and I will consult with Anne if any procedure poses a potential safety concern.

- ❑ I will consult with Anne on any issue that poses a safety concern. If I see an unsafe operation being conducted, I will ask the coworker to correct the problem, and I will consult with Prof. McNeil if the problem persists or is repeated.

- ❑ I agree to all of the above items.

Name (printed)	Name (signed)	Date

Your Health & Well-Being

(Adapted with permission from Jen Heemstra)

Philosophy

We are all here to grow as scientists and people by pursuing ambitious research goals. However, this goal should not come at the cost of your health and well-being. Your mental and physical health are by far the most important consideration in all that you do while in our lab. Moreover, success should not come at the cost of maintaining your interests/hobbies or healthy relationships in your life. You are more likely to be successful if you take care of yourself and give time to the things outside of work that matter to you. Below are some general guidelines but every situation is unique, and Anne is always open to discussion on this topic, so don't hesitate to ask.

Mental and physical health concerns

If you are not feeling well, either physically or mentally, take the time off you need to seek out help and take care of yourself. If you have an acute situation that requires help, take the day (or a few days) off with no questions asked. If you are going to be out for more than 3 days or miss a group meeting, just give Anne a heads up so that she knows you are okay – no need to give details if you don't want to, it is sufficient to email and say that you have a "personal health emergency." If you need to take more substantial amounts of time off, you can work with Anne and the department to facilitate this absence. Being an undergraduate, grad student, or postdoc is stressful. We all care about you and are here to support you – just let us know how we can help.

- For help contact: Counseling and Psychological Services (CAPS) at (734)764-8312 or the University Health Service (UHS) at (734)764-8320. For confidential support related to sexaul assault call (734)936-3333.

Personal emergencies

Chances are that a life situation will arise during your time in our lab. In these situations, the top priority is taking care of yourself and dealing with the situation. If possible, communicate with Anne to let her know that you are dealing with something and approximately how much time you will need off. You can share as much or as little detail as you feel comfortable with. These situations are inherently stressful, so also make sure you are taking care of yourself and getting help if needed.

Work-life integration

I want you to push your limits to explore what you are capable of. The key is to know when to give yourself a break or some time off. Similar to playing sports, you advance by pushing out of your comfort zone, but if you push too hard you end up injured and stuck on the sidelines. Managing your motivation and work habits while integrating interests and commitments outside of work is a key self-leadership skill that will serve you well throughout your career, and now is a great time to build that skill. You can get useful tips and advice on this from Anne, your labmates, and other resources (books, podcasts, etc).

Group Policies

Group Collaborations

Collaborations are a vital part of the scientific enterprise. In the McNeil Group, members are encouraged to participate in both external collaborations (within and/or outside of the Michigan department) as well as internal collaborations (group members working together on related projects). There are many benefits to these collaborations. Oftentimes, the project's timeline can be expedited when multiple people are working together. In addition, your collaborators can give research projects added areas of expertise and new directions.

When you agree to engage in a collaborative project, you are committing to providing the collaborator with the highest possible quality of materials. The group policy is that with each sample you must provide (as a PDF file) the notebook page that corresponds to the exact procedure used to make the sample, a copy of the ^{1}H NMR spectrum of that sample, the yield and estimated purity, and any other relevant characterization data (e.g., the GPC for polymers or elemental analysis for Ni complexes). It is imperative that all lab members conform to the above criteria to ensure a productive collaboration.

Group Meetings

Research group meetings occur every week. (See the group website for the schedule.)

Each member of the presenting subgroup has 10–15 min to present something that they want to discuss. Students should think carefully about how to use this time. Do you want us to help you troubleshoot an experiment? Do you need help with planning what experiments to do next? Do you want a second opinion on your data analysis and interpretation? Do you think your project should switch directions? Or do you just want to update us on your latest findings? Then the student should craft a short (but polished) slide presentation with all necessary information to accomplish their goal. A few general guidelines: All experiments should be accompanied by the full experimental procedure with a chemdraw scheme and literature references. All data should be processed as if publishing it, (do not post raw data), and the analysis should be clearly depicted using ChemDraw and Illustrator/Photoshop.

In addition, each week 2-3 students will have an individual meeting with Anne. Once or twice a year, the group will go off-site for a retreat where everyone will give a formal research presentation.

Public Presentations of Research

All forms of public research presentations, whether conference talks, posters, or published papers are a reflection of the entire research group and *therefore must be approved by Anne 2 days in advance of their presentation.*

Rotation Students

Rotation students should plan to work *at least* 20 hrs per week in the lab. At the end of the semester, rotation students will present a 30 min formal presentation on their work to the group.

Undergraduate Researchers

All undergraduates are expected to work 4 h/credit-hour and can only perform research if a graduate student or post-doc is also in the lab. The undergraduates are expected to attend and participate in all group meetings.

Expectations and Vacation Time

Your success in graduate school is not correlated with the number of hr/wk, but how well you use the time you have (approx. 5 years). Just physically being in the lab is not enough, you need to focus your time and effort when in the lab on experiments (and readings) that move the project forward and your learning.

Each student/post-doc is allotted 21 days of vacation per calendar year (Jan-Dec). This does not include UM holidays (e.g., Thanksgiving day). To keep track of everyone's dates, there is shared Google calendar. You should write your initials on the date(s) you will be taking off AND what vacation day that is for you (e.g., 10/21). Note that sick days do NOT count as a vacation day.

Data Storage: Group Server

After joining the group you should set up some space on the group server to store all data, publications, presentations, and candidacy- and thesis-related documents.

How to Write a Conference Abstract

The following advice was excerpted (and edited) from "How to write a scientific abstract in six easy steps."

1. **Introduction. In one sentence, what's the topic?** Phrase it in a way that your reader will understand. The readers are others in your field, so they know the background work, but want to know specifically what topic your paper covers.

2. **State the problem you tackle**. What's the key research question? Again, in one sentence. Remember, your first sentence introduced the overall topic, so now you can build on that, and focus on one key question within that topic. If you can't summarize your presentation in one key question, then you don't yet understand what you're trying to present. Keep working at this step until you have a single, concise (and understandable) question.

3. **Summarize (in one sentence) why nobody else has adequately answered the research question yet**. The trick is *not* to try and cover all the various ways in which people have tried and failed; the trick is to explain that there's this one particular approach that nobody else tried yet (hint: it's the thing that your research does). But here you're phrasing it in such a way that it's clear it's a gap in the literature. So use a phrase such as "previous work has failed to address…".

4. **Explain, in one sentence, how you tackled the research question.** What's your big new idea?

5. **In one sentence, how did you go about doing the research that follows from your big idea**. Did you run experiments? This is likely to be the longest sentence, but don't overdo it – we're still looking for a sentence that you could read aloud without having to stop for breath. Remember, the word 'abstract' means a summary of the main ideas with most of the detail left out.

6. **As a single sentence, what's the key impact of your research?** Here we're not looking for the outcome of an experiment. We're looking for a summary of the implications. What's it all mean? Why should other people care? What can they do with your research?

Sample Abstract

(Note: Anne submitted this actual abstract in 2009 for a CERMACS invited talk. It follows quite closely to the format described on the previous page. Use this sample as a guide. Numbers were added to indicate which "how to" is relevant.)

(1) Conjugated polymers are the principal components in emerging technologies due to their beneficial properties, including the ability to synthetically tune the optical and electronic character, their low materials cost, and their facile deposition using solution processing. (2) Recent studies have shown that the nanoscale organization in polymer films may be as important as the π-conjugation in terms of device performance. (3) Surprisingly, there have been few studies aimed at controlling these phase separation processes, in part due to the limited number of polymer architectures available. (4) Recent reports of controlled chain-growth synthesis of π-conjugated polymers using nickel and palladium catalysts have revitalized the search for new materials with specific thermodynamic and optoelectronic properties. (5) This talk will highlight our efforts towards a mechanistic understanding of these chain-growth polymerizations, their utilization in the syntheses of new π-conjugated copolymers with unique microstructures, and (6, sort of) the implications for their use in emerging technologies.

The Paper Writing Process

Stage 1

You should start drafting a manuscript in anticipation of the Triannual Research Updates, which occur around group cleanups (*vide infra* for full details on these meetings). Before drafting any manuscript, however, you should compose an outline. Schedule a meeting with Anne to outline the tentative paper. Come prepared with a comprehensive outline.

Stage 2

Download the appropriate template and begin writing. You have (at most) four rounds of revisions, where Anne provides comments on your writing either on paper or electronically. Use your judgement, then, about when the first draft is polished enough to send along. (Be sure to have a senior/experienced student read your first draft and provide comments before sending to Anne.) After four rounds of revision, Anne will "take over" and finish it up. The goal is for you to improve your writing each time so that your next manuscript's first draft starts at the level where you left off. During this time, you should also be compiling the necessary supporting information file. Please see the guidelines in this manual and use other SI files from the group as reference. Again, use your peers for feedback before sending the first drafts to Anne.

Stage 3

At this point, Anne is polishing the text and figures, and may request additional figures or data (i.e., experiments). The most useful thing you as an author can do is help with identifying appropriate references and performing exhaustive and comprehensive searches. In addition, it is at this point that the SI file should be sent out for group edit.

Stage 4

The manuscript will go through a group edit, usually with Anne incorporating the final changes and getting ready for submission. The SI file should be finalized and ready for submission. Anne will draft the cover letter and ask you for suggested reviewers.

Stage 5

Most papers these days go through at least 1 round of revision. For your first paper, Anne will handle the revisions while sharing the information and process with you. If this is not your first paper, you will have a chance to draft the response to reviewers and revision cover letter.

How to Write a Good Paper
– Titles –

The following information was drawn from a group exercise that took place in May 2019. We took a recent issue of ACS Macro Lett and individually voted on whether we liked each title, commenting on what we liked or didn't like about them. Then we came to a consensus on what makes a good paper title, and what to avoid.

Good Titles…

- we can understand exactly what was done
- are short (~10 words)
- contain action words (e.g., predicting, tuning, etc.)

Bad Titles…

- contain unnecessary words (e.g., synthesis, properties, design, toward, use in, etc.)
- are too long (>15 words)
- hard to tell what is new or interesting
- have too little information
- use unfamiliar acronyms
- force fit catchy terms (e.g., "keep xx on track")

Examples of Good Titles…

- Influence of Counterion Structure on Conductivity of Polymerized Ionic Liquids
- Predicting Monomers for Use in Aqueous Ring-Opening Metathesis Polymerization-Induced Self-Assembly

– Abstracts/TOC Graphics –

The following information was drawn from a group exercise that took place in May 2019. We took the most read articles on ACS Macro Lett and individually voted on whether we liked each abstract/TOC combination, commenting on what we liked or didn't like about them. Then we came to a consensus on what makes a good paper abstract, and what to avoid.

Good Abstracts…

- contain three sections: impact/importance, methods, key results/conclusions
- are self-contained, the conclusions address/fill in the gap mentioned earlier
- follow the 6-sentence abstract guidelines, with slight expansion if needed

Bad Abstracts…

- contain raw experimental data
- contain a lot of technical jargon and acronyms/abbreviations
- use strong words that are a matter of opinion (excellent, versatile, synergy)
- have too much of one thing (e.g., introduction)

Good TOC Graphics…

- present a single take-home message in cartoon format

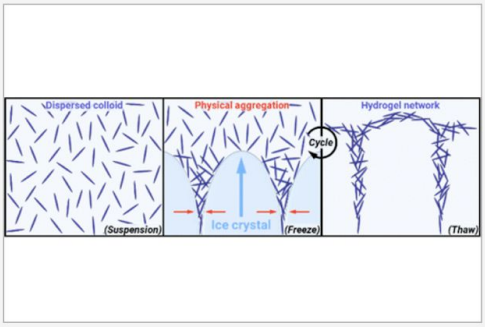
Bad TOC Graphics…

- overly complex with multiple take-home messages
- use actual data plots that require one to take the time to interpret
- contain neon green or other offensive colors
- are too technical (e.g., photos of polymers before/after stretching with ruler)

Example of a good Abstract & TOC Graphics… (link)

Abstract

Gels are attractive for applications in drug delivery, tissue engineering, and 3D printing. Here, physical colloidal gels were prepared by freeze–thaw (FT) cycling of cellulose nanocrystal (CNC) suspensions. The aggregation of CNCs was driven by the physical confinement of CNCs between growing ice crystal domains. FT cycling was employed to form larger aggregates of CNCs without changing the surface chemistry or ionic strength of the suspensions. Gelation of CNC suspensions by FT cycling was demonstrated in water and other polar solvents. The mechanical and structural properties of the gels were investigated using rheometry, electron microscopy, X-ray diffraction, and dynamic light scattering. We found that the rheology could be tuned by varying the freezing time, the number of FT cycles, and concentration of CNCs in suspension.



– Introductions –

The following information was drawn from two group exercises that took place in June 2019. We took a recent issue of Macromolecules (full papers) and ACS Macro Letters (communications) and individually voted on whether we liked each introduction, commenting on what we liked or didn't like about them. We also analyzed the "sections" that were common amongst most introductions and their order. Then we came to a consensus on what makes a good introduction, and what to avoid.

Good Introductions…

- have these basic sections
    - a big picture background (4-5 sentences) ending with a grand challenge (or gap) in the field
    - a narrower picture background (5-10 sentences) that focuses the reader on what work/approaches have been done by us and others to address this grand challenge
    - a clear "gap" sentence that focuses the reader on what this paper is trying to address
    - a brief description of what this paper did (e.g., what system you studied, 3–4 sentences) along with an explanation of why you chose this system (3–4 sentences)
    - then a brief summary of 1-2 main results/take-home message (keep it simple and avoid data, 2-4 sentences)
    - a one-sentence implications statement about how this work will impact others
- contain 1 simplified figure/scheme
- clearly explain all key concepts that are necessary to understand the work/context without getting into the nitty gritty details

Bad Introductions…

- have too long of any of the above sections
- are missing one or more of the above sections
- include equations and undefined concepts (e.g., epitaxy)
- have too many gap statements
- go back and forth between sections

Example of a Good Introduction (full paper)… (link)

Example of a Good Introduction (communication)… (link)

– Results & Discussions –

The following information was drawn from a group exercise that took place in July 2019. We took five recent papers (both communications & full articles) from different journals and individually voted on whether we liked each "results & discussion" section, commenting on what we liked or didn't like about them. We also analyzed the language/phrases that were common among most "results & discussions." Then we came to a consensus on what makes a good "results & discussion" and what to avoid.

Good Results & Discussions…

- Tell a story from start to finish, bringing the reader through the project with multiple guideposts that recap what was learned and inform on what is coming next.
- Consistent paragraph structures:
    - Rationale for why the upcoming experiments were done (1–3 sentences).
    - Description of key results (3–5 sentences).
    - Rationalization of the results (1–3 sentences)
    - Implications of the results. What does this mean for your work and the field? (1–3 sentences)
- Abbreviations with meaning - for example NBE for norbornene as opposed to M1.
- Figures should tell the story without text. Use of color, cartoons, and schemes to help guide data interpretation were all highly valued. See here for a really good example.
- Transitional phrases were really helpful. "We hypothesized" "We anticipated" "To understand" "When then rationalized that"
- Used sub-section headings to help guide the reader on how the parts connect together.

Bad Introductions…

- Have disparate sections that seem unrelated without transitional phrases or sub-section headings between them.
- Descriptions of results without a clear understanding of why the experiments were done.
- Descriptions of results without their implications.
- Contain too many abbreviations and acronyms. It breaks up the flow of sentences and makes the reader work harder to understand/comprehend the experiments.
- Talk extensively about figures located in the SI.

Example of a Good Results & Discussion… (link)

– Conclusions –

The following information was drawn from a group exercise that took place in June 2019. We took a recent issue of ACS Macro Lett and individually voted on whether we liked each conclusion, commenting on what we liked or didn't like about them. We also looked for comment sections/structures among the ones we liked. Then we came to a consensus on what makes a good conclusion, and what to avoid.

Good Conclusions…

- have these basic sections
    - a brief summary of what they did (1-2 sentences)
    - a brief summary of the key results (note: for full articles, we definitely liked having this section, whereas we were more split on whether communications needed to have a results summary)
    - an explicit discussion of the implications of the work (1-3 sentences)
- shorter sentences with simple words/terms were best
- define all terms (even if defined in the paper b/c some people read the conclusion after the abstract)

Bad Conclusions…

- re-hash their discussion of the key results
- contain too many useful words (e.g., versatile, excellent, etc)
- contain numerical data/results
- rely on acronyms
- reference figures or SI

ChemDraw Guidelines

After opening ChemDraw, go to **File** and then **Apply Document Settings from** and choose **ACS Document 1996**. Proceed to draw your structures and reaction schemes. (There are a lot of great tutorials here and here on the web for learning and mastering ChemDraw. There is also a comprehensive 300+ page user guide and list of shortcuts)

Pay attention to the little details, like centering text over arrows, aligning and distributing the structures in a reaction, using the same sized arrows in a single scheme and paper. Use a circle as a scaffold to create a mechanistic cycle and guide arrow placement, etc. Color can be a really useful tool. Iron is a good color for the main structures, then use midnight and cayenne to highlight parts of structures. (Note: These colors refer to choices on Macs, which you should be using for all paper figures.) Avoid greens and yellows. Do not switch between CH3 and Me in a figure or set of figures. Make sure square planar complexes are actually square planar and that the substituents are aligned (vertically/horizontally) on any metal catalyst.

When you are ready to "move" your structures into another document, first select all. Then go to **Object** and choose **Object Settings**. Change the "bold width" to 0.03 and the "line width" to 0.015. Then save your file as both a .cdx and .png. Do not do any re-scaling or sizing in ChemDraw.

Open the png file in Adobe Photoshop. Then go to **Image** and choose **Image Size**. Use a calculator to determine the new width and height (in inches or cm) by multiplying the original values by some %. The smallest you should re-size a ChemDraw image is 65% (usually what I prefer for papers and grants). Most importantly, once you pick a re-size value, stick with it for all images within a single paper. Note that for single column figures, the max size is 3.3 cm and for double-column figures, the max size is 6.5 cm.

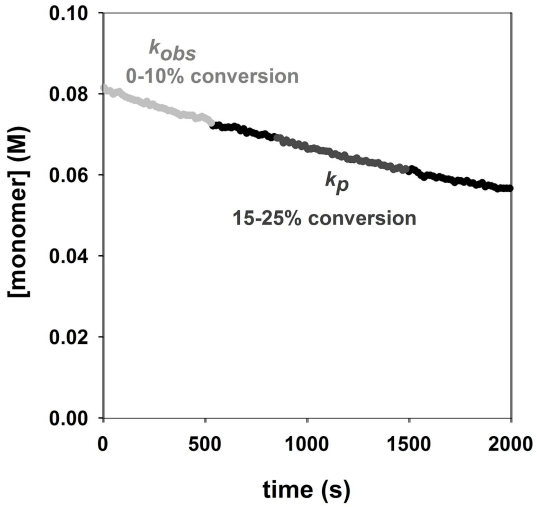
In some cases, you may want to enhance your image in Adobe Illustrator. Open and modify the original png file (before re-sizing). Illustrator enables you to add more diverse shapes (1D, 2D, 3D)/colors and you can even map your chemical structures onto 3D images.

Plot/Figure/Equation/Scheme/Chart/Table Guidelines

Plots

All plots should be created in SigmaPlot. The plot size should be square; I recommend setting the plot dimensions to 3.0 x 3.0 inches. Almost all plot axes should start at 0,0 unless it really doesn't make sense to do so (e.g., with retention volume in GPC). The axis labels should be simple, and contain the units in parentheses. Also, do not capitalize the axis labels. The data on the plot should be clear/readable and labeled so that the take-home message is easily interpreted. Please do not use "legends" or figure captions. Do not give the plot a title either. The plot itself should be self-explanatory. Use color sparingly; use grayscale and dashed lines as a starting point.

Please follow the follow guidelines for plot formatting:
Plot dimensions: 3.0 x 3.0
Axis and Tick Sizes: 3 pt (only use major ticks)
Axis Font: 16 Arial Bold



Figures

First and foremost, figures have processed data (plots, spectra, etc). Sometimes chemical structures and/or reactions can be added to supplement the data, but a figure must have some data! In general, stick to one column figures (width 3.3 cm) unless there is a really strong justification for using the two column width (6.5 cm). Note that two plots can fit side-by-side into a single column figure without appearing too small. Figure captions should be concise and contain essential experimental information!

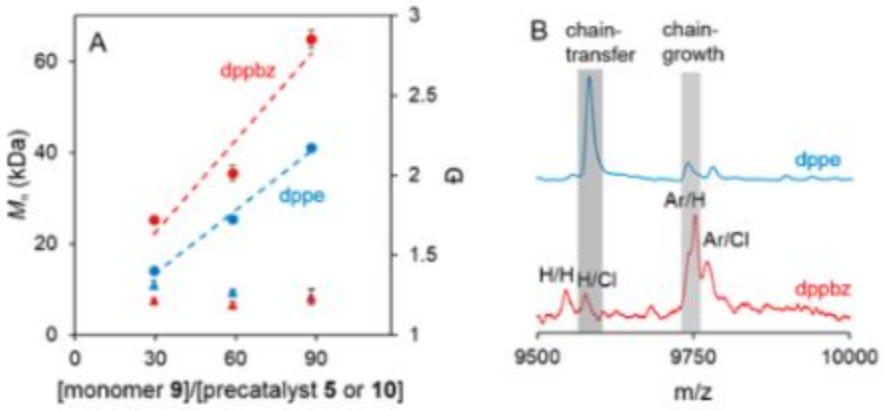
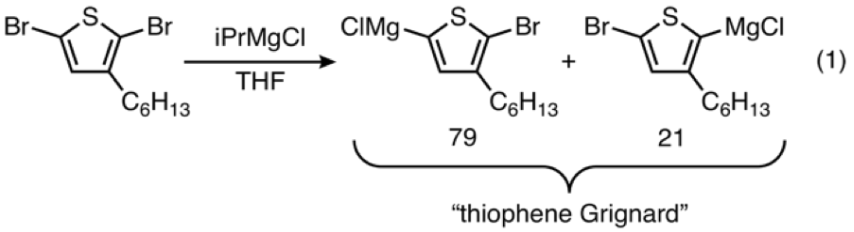


Figure 3. (A) Plot of M_n and dispersity ($Đ$) of PTz-OR versus the monomer/catalyst ratio using either precatalyst **5** (blue) or precatalyst **10** (red) and monomer **9**. (B) MALDI-TOF-MS analysis of PTz-OR obtained via either precatalyst **5** or **10** and monomer **9**.

Equations

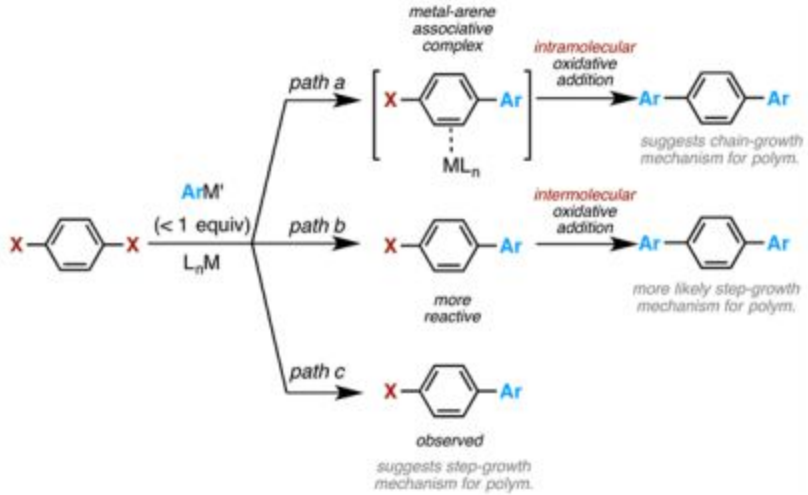
Equations usually contain either mathematical relationships or a single chemical transformation. Number them sequentially in the manuscript (hint: place the # in the chemdraw version to keep the sizing the same). Equations do not contain titles or captions.



Schemes

A scheme is a series of equations that make more sense when grouped together. A good rule of thumb is that if it has more than one reaction arrow, it is likely better represented as a scheme. Schemes require titles which are generally placed above the chem draw (though be sure to check the journal requirements). The title should briefly describe the main conclusions of the scheme.

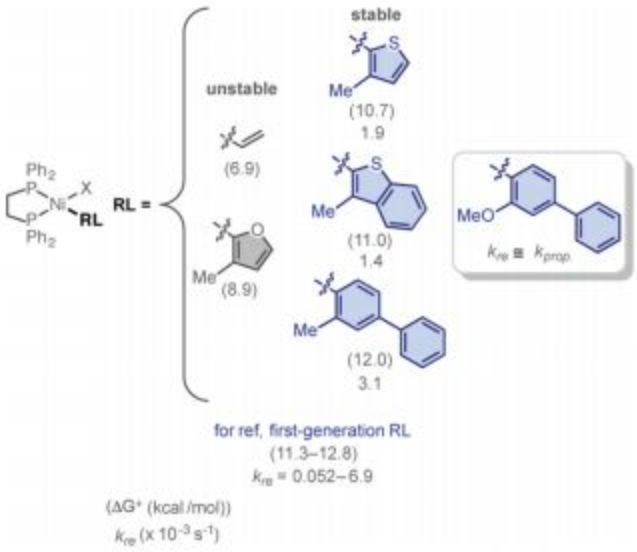
Scheme 1. Difunctionalized Products Can Be Obtained via Two Different Pathways



Charts

A chart is a collection of structures, sometimes with data associated with them. Chart titles are generally placed above the data and briefly describe the major findings.

Chart 2. Second-Generation Reactive Ligands[73]



Tables

I generally avoid tables unless absolutely necessary. Tables are most useful when you run a series of reactions with varying conditions or substrates. Before making a table, consider whether a chart might be a better method of presenting the data.

Table 1 Results of the competition experiments[a]

	$P_{intra} : P_{inter}$			
Equiv. of 3[b]	**1a**	**1b**	**1c**	**1d**
1	95 : 5	65 : 35	97 : 3	98 : 2
2	91 : 9	55 : 45	94 : 6	96 : 4
10	69 : 31	28 : 72	78 : 22	87 : 13
50	40 : 60	13 : 87	49 : 51	71 : 29
100	32 : 68	11 : 89	40 : 60	64 : 36

[a] The reactions were run in THF at rt for 2 h ([Ni] = 0.02 M; [2] = 0.016 M). The reported ratios reflect the averages of three runs, with standard deviations ranging from 0.06–2%. [b] Relative to 2.

Supporting Information Guidelines

Philosophy

This document is an incredibly important document and one that should replicate the results you obtained as depicted exactly in your lab notebook. Every section of SI should be associated with a searchable experiment number from your notebook and data files. Original electronic copies of the ^{1}H and ^{13}C NMR spectra, as well as the HRMS results, elemental results, rate profiles, GPC data, etc should be uploaded to the group server. Your SI must conform to the above criteria or you will be asked to re-run the experiment again prior to submission.

General Guidelines

Open the SI group template and follow the instructions/guidelines.

- For the table of contents: You should list both the section titles as well as their starting page #. The section titles should be informative but not too lengthy. The order of sections should follow the order in which the data appears in the paper.

- The first section is "materials and supplies" and should list the source of reagents and compounds, whether and how they were purified before use.

  Sample Materials Section 1:

  I. Materials

  iPrMgCl (2M in THF) was purchased in 100 mL quantities from Aldrich. Bis(cyclooctadiene)nickel ($Ni(cod)_2$) and 1,2-bis(diphenylphosphino)ethane (dppe) were purchased from Strem. All other reagent grade materials and solvents were purchased from Aldrich, Acros, EMD, or Fisher and used without further purification unless otherwise noted. THF was dried and deoxygenated using an Innovative Technology (IT) solvent purification system composed of activated alumina, copper catalyst, and molecular sieves. *N*-Bromosuccinimide (NBS) was recrystallized from hot water and dried over P_2O_5. Flash chromatography was performed on SiliCycle silica gel (40–63 μm) and thin layer chromatography was performed on Merck TLC plates pre-coated with silica gel 60 F254. Compounds **S2**,[1] and **2b**–**2f**[2] were prepared from modified literature procedures.

  Sample Materials Section 2:

I. Materials

All reagent grade materials and solvents were purchased from Sigma Aldrich, Acros Organics, or TCI. The paint thinner used was Klean-Strip paint thinner made with mineral spirits. Paints used were as follows: black oil-based paint: Rust-Oleum Professional, V7579 Gloss Black, High performance enamel; pink latex-based paint: Valspar Satin Berry Twist 530832, Spring 2014; white oil-based paint: Rust-Oleum, 7792 Gloss White, Protective Enamel. All alkyl amines and carbon disulfide were distilled prior to use. Methanol was dried over activated molecular sieves under N_2 overnight. All other compounds were used without further purification unless otherwise noted. Compounds **S1-S3**,[1] **1a–h**,[2,3] **2a–b**,[4] and **3**[5] were prepared from modified literature procedures. Throughout this document H_2O refers to deionized H_2O, unless otherwise noted.

- The second section is the "general experimental" and should give specific information about the types of equipment used, and if appropriate, how the data was analyzed.

Sample General Experimental Section:

II. General Experimental

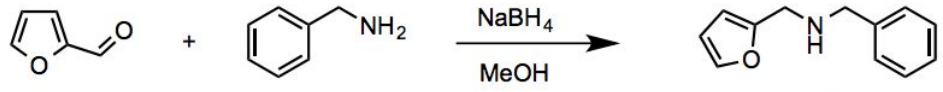
NMR Spectroscopy – ^1H and ^{13}C NMR spectra for all compounds were acquired in d_6-DMSO or D_2O on a Varian vnmr 700 operating at 700 and 176 MHz, or a Varian Inova 500 operating at 500 and 126 MHz. The chemical shift data are reported in units of δ (ppm) relative to tetramethylsilane and referenced by residual protic solvent. An asterisk was used to indicate residual H_2O in all spectra while double bars are used to indicate peaks that have been truncated. The abbreviations s, d, t, at, dd, q, and m were used to signify singlet, doublet, triplet, apparent triplet, doublet of doublets, quartet, and multiplet, respectively.

High Resolution Mass Spectrometry (HRMS) – HRMS data were obtained on a Micromass AutoSpec Ultima Magnetic Sector mass spectrometer via electrospray ionization in negative ion mode.

UV-vis Spectroscopy – UV-vis spectra were taken on a Perkin-Elmer Lambda 850 UV-visible spectrometer. Calibration curves were measured at the λ_{max} for each compound. All experiments were run in triplicate at rt.

- The third section is dedicated to "syntheses" of all materials generated during the course of the work. It should be self-contained, meaning that if you had to make it for this paper because it was not commercially available, then its synthesis should appear here...even if we (or someone else) previously published a synthetic procedure for it. Undoubtedly, you did it a little differently, and the SI should represent your individual work and should match the referenced notebook page exactly! In addition, you should list either the elemental analysis results OR high res mass spec results which support the identity of the compound.

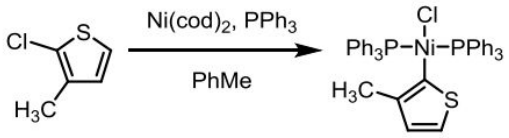
Sample Organic Experimental Procedure:



S1

***N*-Benzyl-1-(furan-2-yl)methanamine (S1).** 2-Furaldehyde (300 µL, 3.63 mmol) and benzylamine (360 µL, 3.30 mmol) were combined in dry MeOH (9 mL) and stirred under N_2 for 18 h. The solution was then treated with $NaBH_4$ (279 mg, 7.38 mmol) in small portions, and stirred under N_2. After ~1 h, no starting material was visible by TLC. The reaction was carefully quenched with H_2O (10 mL). MeOH was removed via rotary evaporation, and the aqueous residue extracted with EtOAc (3 x 10 mL). The combined organic layers were dried over $MgSO_4$, filtered, and the solvent removed via rotary evaporation. The resulting oil was purified by flash column chromatography, eluting with 14% to 20% EtOAc in hexanes to give a clear oil (518 mg, 84%). HRMS (ESI): Calcd for $C_{12}H_{14}NO^+$, 188.1070. Found, 188.1066.

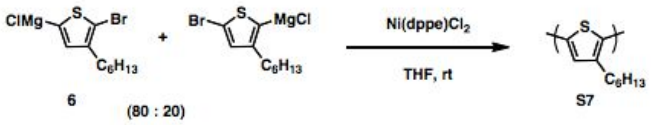
Sample Organometallics Experimental Procedure:



S1

[Bis(triphenylphosphine)](3-methylthiophene)nickel(II) chloride (S1). A 20 mL vial was equipped with a stir bar in the glovebox. Sequentially, $Ni(cod)_2$ (139 mg, 0.506 mmol, 1.00 equiv), PPh_3 (262 mg, 1.00 mmol, 1.98 equiv), toluene (4 mL), and 2-chloro-3-methylthiophene (82 µL, 0.75 mmol, 1.5 equiv) were added. The solution was stirred at rt for 30 min and turned from dark red homogeneous solution to orange heterogeneous mixture. The reaction was removed from the glovebox. Addition of hexanes (30 mL) led to an orange precipitate. The solid was filtered and washed with hexanes (20 mL) and cold MeOH (5 mL). The resulting solid was recrystallized from 1/3 (v/v) THF/hexanes (approx. 20 mL), to give 299 mg of **S1** as an orange solid (84% yield). Elemental analysis: Calcd for $C_{41}H_{35}ClNiP_2S$, C, 68.79; H, 4.93; Found C, 68.49; H, 4.88.

Sample Polymerization Experimental Procedure:



6 (80 : 20) S7

S7. In the glovebox an oven-dried 20 mL vial was equipped with a stir bar and charged with **6** (1.0 mL, 0.20 mmol, 1.0 equiv) and THF (3.5 mL). The pre-initiated catalyst solution (0.50 mL, 0.0013 mmol, 0.0063 equiv) was added. After 1 h the reaction was quenched with HCl (5 mL, 5 M) then extracted with $CHCl_3$ (3 x 5 mL). The combined organic layers were washed with water (2 x 5 mL) and brine (1 x 5 mL) and concentrated in vacuo. The resulting solid was washed with methanol to give 30 mg of **S7** as a dark purple solid (quant.).

- The fourth section is dedicated to the "characterization" (e.g., ^{1}H, ^{13}C, ^{19}F NMR spectra) of the compounds found in the synthesis section. The figures should all have the same x-axis scaling (e.g., 0–8 ppm for all ^{1}H NMR spectra). Be sure to check the journal if any particular guidelines are required for submission. I recommend making a template of just the axes and then pasting the spectra (without axes) into the template for consistency. Each figure should also have chemical structure and structure number. If the peak splitting is hard to see, then insets should be created as well. The figure caption should list the peak positions to 2 decimals (both ^{1}H and ^{13}C), coupling constants (italicize the J), and the number of protons (based on the integration).

  Sample ^{1}H and ^{13}C NMR spectra:

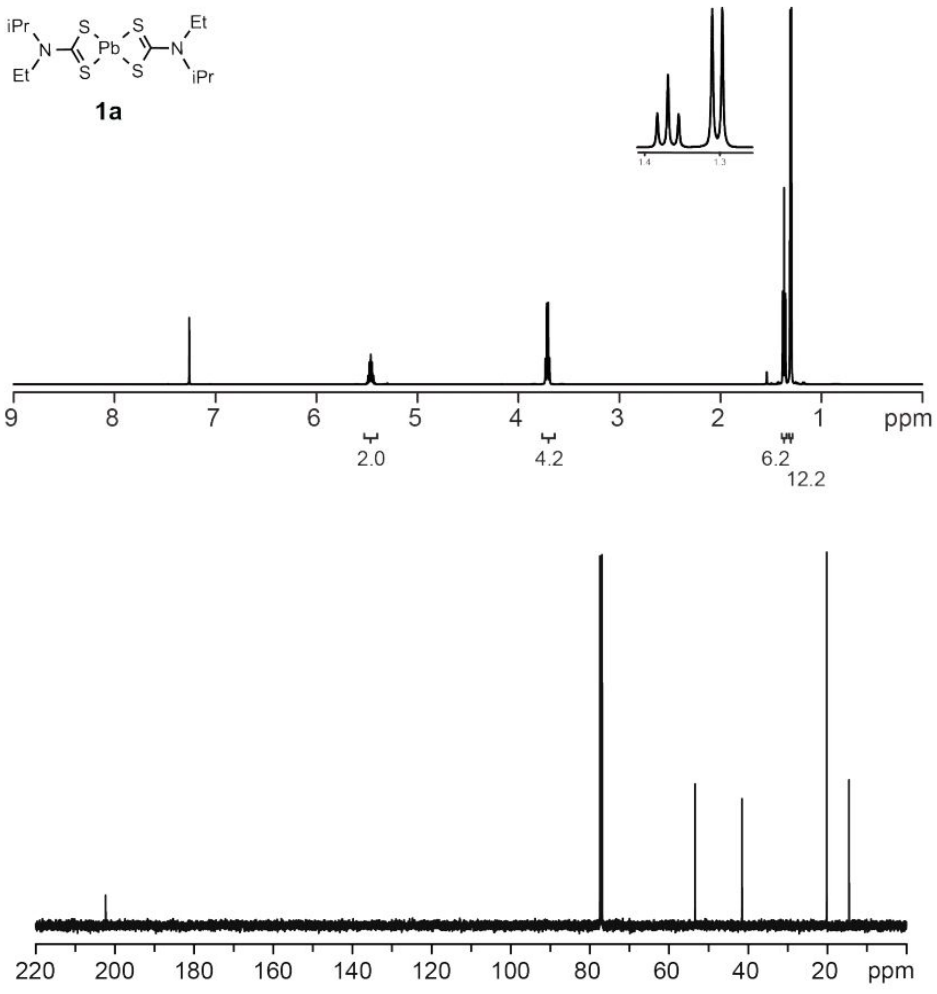


Figure S4. ^{1}H and ^{13}C NMR spectra of **1a**. ^{1}H NMR (500 MHz, $CDCl_3$) δ 5.46 (sept, *J* = 7 Hz, 2H), 3.71 (q, *J* = 7 Hz, 4H), 1.37 (t, *J* = 7 Hz, 6H), 1.30 (d, *J* = 7 Hz, 12H). ^{13}C NMR (126 MHz, $CDCl_3$): δ 202.32, 53.41, 41.56, 20.13, 14.50.

- From here, the list of sections should follow the order in which the data appears in the manuscript.

Common General Formatting Mistakes/Reminders

1. Margins need to be justified.
2. A regular hyphen (-) is used to separate elements of a compound word (i.e. “water-soluble”).
3. The en-dash (–) ([option][-] on a Mac) is used for things like named reactions (“Diels–Alder”), number ranges (“1–13”), bonds (“C–O bond formation”), or negative temperatures (i.e. –35 °C, not -35 °C)
4. Degree signs (°) should be written using [option 8 on a Mac] not using a superscript o.
5. There should be a space between the numbers of a temperature and the degree sign (i.e. “55 °C”, not “55° C”).
6. Abbreviations: h not hours; min not minutes; d not days; s not seconds.
7. Check all elemental formulas and mass spec formulas for errors/typos.
8. Using fewer words is preferred (e.g. “synthesizing not the synthesis of”).
9. Use proper and consistent nomenclature for compounds.
10. HRMS [M + H] versus [M + H^+]. Make sure that your molecular formula corresponds to the peak that you’re reporting. For example, if a proton (H^+), sodium cation (Na^+), etc. is present in your peak, it should also be in your molecular formula.
11. Compounds, schemes, and tables should be numbered sequentially.
12. Amounts of reagents, solvents, etc. are set aside parenthetically. For example, “The solution was washed with brine (3 x 50 mL),” not “The solution was washed three times with 50 mL of brine.”
13. “*That*” and “*which*” are not interchangeable! If removing the words that follow would change the meaning of the sentence, use “that”. Otherwise, “which” is fine.
14. “*Et al.*” and “*and coworkers*” are not interchangeable. Smith, Leone, and McNeil* would be “*Smith et al.*” or “*McNeil and coworkers.*”

Slack Guidelines

Slack

Slack is a cloud-based app that we use for all group communication. All students should join our Slack team “McNeil Group” when you start. Channels should be used to send messages to large groups of people. For example, the “conjugated polymers” channel sends a message to everyone in that subgroup, whereas messages posted to the “random” channel go to everyone. Slack can also be used to send direct (private) messages to anyone in the group, including Anne. You can also start “conversations” with several people at any time by creating a direct message to more than one person. Slack should be used for all file sharing as well, you can upload/download, share via Google drive, etc. One really nice feature of Slack is that both the files and messages are text searchable.

Searching and Reading the Literature

Searching the past literature

Reaxys

Excellent resource when searching for specific chemical reactions & conditions.

SciFinder Scholar

This program is web-based and used whenever you want to find out how to do a specific synthetic transformation. For more information, refer to "Information Retrieval: SciFinder and SciFinder Scholar" by Damon D. Ridley, Wiley, 2002.

ISI Web of Science

There are several very useful functions of this website.

A. Cited References Search

Use this search to find all articles that cite a key paper. This search is especially useful because you can also find articles that cite the paper that cited your key paper!

B. General Topic Search

Use the "and" command to link two concepts and use "*" to expand. For example, if you want to search for fluorescent polymers, but also want to include any search where "fluorescence" is also used. Your search command would read "fluor* and polymer."

C. Author Search

Use this search to find all articles published by an author. You can then refine this search using keywords, dates, or document type, etc.

Note that both search engines allow you to refine your results by narrowing the list and by analyzing the list. For example, if you are searching for a starting material, you can refine your SciFinder result list by "commercial availability." Or if you are searching for articles with a name like John Smith, you can refine by first analyzing the "institution type" and then only select those articles by John Smith at University of Michigan. Other useful tools include Wikipedia, Google, Google Scholar, "Comprehensive Organic Transformations" by Larock, and eROS (encyclopedia of reagents for organic synthesis.)

Google

This search engine can provide a useful starting point for any literature search.

Reading the Current Literature

Keeping up with the current literature is essential to becoming an independent and successful scientist. You should dedicate several hours per week to browsing the latest issues of key journals (to gain breadth) and reading the important articles in your field (to gain depth). My advice is to sign-up for receiving the latest articles either via email or RSS feeds. Personally, I use Feedly to aggregate all my RSS feeds and link directly to the articles. I then download and move the PDF into Mendeley (if warranted) later.

Google Scholar

Google Scholar alerts to keep abreast of papers in your field.

More Suggested Reading Material

“Alternative Careers in Science: Leaving the Ivory Tower” by Cynthia Robbins-Roth

“A PhD is Not Enough: A Guide to Survival in Science” by Peter J. Feibelman

“Tomorrow's Professor: Preparing for Careers in Science and Engineering” by Richard M. Reis

“The Academic Job Search Handbook” by Mary Morris Heiberger

“At the Bench: A Laboratory Navigator” by Kathy Barker

Lab Notebook (OneNote) Guidelines

General

- Every experiment you do MUST be accompanied by an entry in your lab notebook.

- Every experiment you report in SI MUST be accompanied by an entry in your lab notebook.

- Only use OneNote and ask Anne to initiate your notebook within the group one.

- If you repeat a procedure, record it as a new experiment and refer to the experiment for the procedure. However you MUST record the quantities of starting materials and yields for products in the new experiment, even if it is identical. If anything else is different, workup conditions, chromatography conditions, etc, record these changes as well.

- Make sure to include the time at which you start the experiment as well as when you finish it. If you work on the experiment over multiple days, record a new date for each entry.

- Record every action and observation. The more detail you provide, the easier it will be for you (or someone else) to learn from and reproduce your work. Use past tense to describe the experiments (The product was purified by column chromatography.)

- When you are determining the mass of product obtained, record the mass based on the accuracy of the balance.

- If the NMR spectrum is impure and you have to re-purify, report a new yield that corresponds to the purified product. For the supporting information, you should report the yield that matches the final, pure product, not a previous impure fraction.

- Indicate in your notebook the spectra name and date acquired for all data relating to that experiment.

- Record the source of each reagent (lab notebook page or commercial supplier), the date opened and the batch number (if purchased).

- Record the actual quantities added to the accuracy of the instrument you used to measure it. For example, if you intended to add 10 g and you weighed out 10.230 g – record the 10.230 g. If you prefer, you can have two columns in your notebook – one listing the amount you plan to add and one listing the amount you actually add. But you MUST have the amount you actually added, as well as calculations that match the moles, equivalents, etc of the other reagents.

- If you decide that for one reason or another you don't think the data obtained is valid, note this in your notebook and state your reasoning. For example, "There was significant quenching of the monomer during the polymerization as evidenced by the IR signal corresponding to this by-product."

- Read pages 92-98 in the "At the Bench" laboratory manual located in our group library.

- Save all spectra and original data labeled with an identifier that corresponds to the page number in your notebook. Indicate in your notebook the spectra name and date acquired. Be prepared to show this data to me OR a reviewer when asked.

- Keep your electronic notebook up to date! At the end of each week try to make sure all data files have been uploaded and the notebook entries have been documented. Consistent notebook-keeping is an essential part of being a scientist!

- **We should be able to find this data after you have graduated. You must upload all your supporting data files into the electronic notebook**.

Summary

Ultimately, your electronic lab notebook, collections of spectra and other data, and the supporting information documents are the ***most important*** things you will prepare as a scientist during your PhD. You should take pride in these items and spend the time to prepare a solid foundation to support your claims. This documentation can and will be used to verify that indeed these experiments were run and that the results

are as you claim. If there are errors in these records, then it can call into question deeper issues ranging from carelessness, negligence, to scientific fraud. These can result in serious consequences, including getting expelled from the PhD program. **Do not take these issues lightly!**

Collecting and Archiving Scientific Data

Uncertainty in Measurements – Significant Figures

All measurements have some level of uncertainty. Significant figures include all the digits you are certain about PLUS one additional uncertain digit. See attached discussion. Note that our balances are uncertain in the *last* digit you can read.

Counting Significant Figures

Leading zeros are not significant. 0.008 has one significant figure
Captive zeros are significant. 1.02 has three significant figures
Trailing zeros are significant. 40.00 has four significant figures

Rules for Rounding

In a series of calculations, carry the extra digits through to the final result and then round. If the digit to be removed is less than 5, the preceding digit does not change. (1.33 = 1.3) If the digit to be removed is greater than 5, the preceding digit goes up by one. (1.36 = 1.4) Only look at the first number to the right of the significant figure. (4.348 = 4.3) If the digit is 5, then round to the closest even number. (1.35 = 1.4 and 1.65 = 1.6)

Calculations and Significant Figures

For multiplication and division, the answer should have the same number of significant figures as the least precise measurement. (4.56 x 1.4 = 6.4)
For addition or subtraction, the answer should have the same number of decimal places as the least precise measurement. (12.1 + 18.0 + 1.103 = 31.1)

Labeling and Storing Compounds

- Label all vials with a structure AND the notebook page number. Use a small white label and attach to the vial with reinforced clear tape. Sharpie's do not last the test of time!

- Store all synthesized materials in small disposable vials with screwcaps. Do not store any compound in an expensive flask with ground glass joints!

- Indicate the amount of material inside the vial on the screwcap (e.g., 52 mg).

- Store all synthesized compounds either near your bench or in your assigned shared refrigerator. Do not store your compounds on the group chemical cabinets or in the group refrigerator.

Advice for Running Reactions

- For a great reference on synthesis and lab techniques, check out the "Not Voodoo" website by Alison Frontier (University of Rochester) at http://chem.chem.rochester.edu/~nvd/. See also, "The Laboratory Companion" by Gary S. Coyne and John S. Wiley and "Advanced Practical Organic Chemistry" by J. Leonard, B. Lygo, and G. Procter. See also, senior members of our research group.

- Start with pure reagents and chemicals. See "Purification of Laboratory Chemicals" by Armarego and Chai for detailed information on how to purify common reagents and solvents. Garbage In = Garbage Out!

- Run reactions on a small scale the first time (~100 mg or less!). After you have worked out the reaction conditions and purification procedure you can scale up. Do not scale up a reaction more than 3-fold of your previous successful attempt.

- Monitor your reactions by TLC (see below). You can supplement TLC with GC analysis, crude NMR spectra, and IR. None of these techniques are a substitute for TLC.

- Always work up reactions immediately upon completion.

- Take the time to identify by-products the first time through a synthesis.

TLC and Flash Chromatography

Adapted from Prof. David B. Collum's "Mother Liquor Lecture" Cornell

TLC

1. Monitor all reactions before adding the last reagent, during the reaction, and after the quench. If something goes wrong, you will not know when that happens unless you are monitoring the reaction!

2. Always co-spot the reaction mixture with the starting materials to aid identification.

3. If two spots are extremely close, then spot them twice. If a Z pattern develops, they are NOT the same material.

4. Visualization: (1) By sight! (2) UV (3) Stains (see next page).

5. For amines, add 0.5% Et_3N to the solvent chamber. For acids, add 0.5% HOAc.

6. Draw the TLC plate in your notebook and mark with observations.

Flash Chromatography

Still, W. C.; Kahn, M.; Mitra, A. "Rapid Chromatographic Technique for Preparative Separations with Moderate Resolution." *J. Org. Chem.* **1978**, *43*, 2923-2925.

1. Pick a favorite solvent pair (e.g., EtOAc/hexanes) and get to know it in all ratios.

2. Pack column under positive pressure.

3. For many samples, it is convenient to first add silica gel (about 1 scoop per 250 mg) and concentrate to form a coated powder. Add this solid to the pre-packed column and then add the sand.

4. Elute at 2"/min. Change to more polar solvents over time if needed.

5. Monitor the progress of the column by TLC in real-time!

6. Use half TLC plate to spot 5 test tubes, run TLC and then flip over and use the other half for another 5 test tubes.

7. For small-scale reactions, a pipette can serve as a column.

8. OR - Use the automated chromatography system in the lab to run simple columns much faster! (Please note that all new group members must master the manual column before using the Biotage.)

TLC Stains

(Adapted from Prof. Justin DuBois, Stanford University)

Anisaldehyde

Add 2.5 mL of AcOH and 6.5 mL of p-Anisaldehyde to 300 mL of ice cold 95% EtOH (or EtOAc, not denatured EtOH). Cautiously add 8.5 mL of concentrated H_2SO_4 dropwise over 60 minutes. Store unused portions in freezer.

CAM (Ceric Ammonium Molybdate)

4 g	Cerium sulfate (complex with H2SO4)
100 g	Ammonium molybdate tetrahydrate
900 mL	deionized H_2O
100 mL	conc. H_2SO_4

Potassium Permanganate

2 g	$KMnO_4$
20 g	K_2CO_3
5 mL	5% NaOH

Dissolve KMnO4 in 300 mL water.
Add K_2CO_3 followed by NaOH.

Iodine

Add 500 mg I_2 to 20 g silica gel in amber jar.

Ninhydrin

3 g	Ninhydrin
30 mL	glacial acetic acid
970 mL	n-butanol

Stir until dissolved. Store in a brown bottle in the freezer.

PMA (Phosphomolybdic acid)

100 g	PMA
1273 mL	95 % ethanol

Stir until dissolved. Store in freezer.

Cold Bath Temperature Chart

System	°C	System	°C
ethylene glycol/CO_2	-15	Carbitol acetate/CO_2	-67
CCl_4/CO_2	-23	EtOH/CO_2	-72
3-heptanone/CO_2	-38	acetone/CO_2	-77
CH_3CN/CO_2	-42	SO_2/CO_2	-82
cyclohexanone/CO_2	-46	isopropanol/N_2	-95
diethyl carbitol/CO_2	-52	Et_2O/CO_2	-100
$CHCl_3$/CO_2	-61		