INORGANIC CHEMISTRY Laboratory

Chemistry 5192

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Inorganic Synthesis

***Syllabus* - Chem 5192 -** Fall, **2017**

First Day class meets – Friday, September 1 (quant lab, 2 pm, lecture on silicones and phosphoranes, attempt questions before lecture)

First Assignment due - WEDNESDAY, AUGUST 30 (silicone question draft due)

Instructors:

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The course is a rigorous laboratory course even though the lab experiments begin late in the semester when all your other courses are going full speed. BE PREPARED TO WORK DILIGENTLY. Late reports, lack of preparation, etc. will not be tolerated and point penalties apply to all late and haphazard work.

Typically, each experiment takes two lab periods. To prevent decomposition, etc., it is mandatory that these syntheses are completed in this time rather than allowing them to sit from Wednesday to the following Monday. The course will introduce you to many new techniques and to many new concepts that have not been covered in any other course. Much instruction will be through direct interaction with the instructors, but most learning will occur through your individual effort and from digging out information and answers from the literature references at the end of each experiment. This lab course will be completely different than any other one you have had thus far.

You will usually work in teams, with team members changing almost every week. This will require extensive effort on your part to make sure you do your share and that you understand what has been done. You must **ALWAYS** thoroughly study (not just skim) the procedures **BEFORE** you come to lab. It is important that you realize that this is not a "cooking" class and that understanding, "what, why and how" are truly the essence of this course. Indeed, that is why you have final exam questions based on the experiments and on lab techniques, including things done "behind the scenes" such as calibrating the vacuum line and distilling solvents.

YOU MUST BE READY TO DO THE EXPERIMENT - no faking it! I will know! Part of your grade will be based on how ready you are. The references at the end of the experiments will be useful in preparing you for the lab, in writing your report, and in answering the questions pertaining to each experiment. Speaking of questions. To lighten your load at the busy end of the semester, to prepare you for each experiment, and ultimately, to help you write the lab report, we require that you answer the **Questions** for each experiment and **turn them in** the **first half of the semester**. We will have lectures on September 1, 8, 22, and 29 to give you some background that may help you answer the questions, but you will also have to find many answers on your own by doing research, including reading original journal articles. Once you have sincerely exhausted these sources, you can always come by or email us and we will get you going in the right direction. All questions and lab reports must be turned in via Canvas Assignments.

Last but not least ---- please come to see us if you have any questions about this class, the information provided here, the questions, etc.

A calendar has been uploaded separately with the schedule and will be sent to you by email.

Learning Outcomes

By the time you finish this course, you will be able to:

- Measure and manipulate organic and inorganic chemicals under an inert atmosphere
- Purify reaction products using distillation, crystallization, and chromatographic techniques
- Analyze reaction products using advanced instrumental techniques, including multinuclear NMR spectroscopy, gel permeation chromatography, and UV spectroscopy
- Communicate experimental objectives, results, and conclusions through professional reports written in the scientific style. Specifically, you will be able to
  - State and defend a thesis/objective with adequate attention to analysis and evidence
  - Demonstrate an understanding of essay and paragraph development and organization
  - Craft sentences with attention to audience, purpose and tone, as well a sentence variety and diction
  - Demonstrate proper use of grammatically and mechanically correct English

Experiments:

Note that a "code" name for each experiment is given in bold and that some of the most important experimental techniques are listed below the titles.

- *Preparation of Silly Putty: a Cross-linked Silicone* ***"Silly Putty/Silicone"***
- *Synthesis of an Organosilicon Thioether Ligand and Its Complexation to Ag(I)* **"Sulfur-Ag"**
  main group element compounds, transition metal complexes, inert atmosphere and Schlenk techniques, vacuum distillation
- *Synthesis of Well-Defined Polymers by Low-Catalyst-Concentration ATRP and Post-Polymerization Modification to Fluorescent Materials* **"ATRP"**
  gel permeation chromatography, inert atmosphere, polymerization methods, inorganic polymers, polymer properties
- *Synthesis and Characterization of* $(CH_3)_2NPF_4$ **"Phosphorane"**
  vacuum line techniques, e.g. trap-to-trap distillation, vacuum transfers
- *Jacobsen's Asymmetric Olefin Epoxidation* **"Jacobsen's Catalyst"**
  chiral transition metal complex catalysis, thin layer and column chromatography, lanthanide NMR shift reagents
- *Preparation/Spectroscopic Analysis of Semiconductor Colloids* **"Semiconductor"**
  IV-VI and II-VI semiconductors, micelles/microemulsions, UV spectroscopy, cluster compounds, biopolymer, solid state chemistry

Grading

Lab and lab reports	48%
Homework Questions	36%
Final Exam	16%

The Final Exam will consist of questions on lab techniques and concepts related to the experiments.

Laboratory Requirements and Grading

Before we get access to lab space to do the experiments, you will attend FOUR FRIDAY (2 P.M.) lectures (September 1, 8, 22, and 29) and you will answer a set of questions related to each of the six experiments. These will be due (one per week) starting with a draft of your answers to the "silicone" questions on **August 30**, [final set due September 6]. (See calendar sent via email and at the end of this document.) ***These must be submitted electronically via Canvas and all chemical drawings must be done using chemical drawing software.**** In some cases, you can sketch, scan, and cut and paste, e.g., question #11 on Silicone Questions. By answering these questions, you will be ready to begin writing the Introduction Section of the lab report (something to save you time later).

* While you may purchase a program like ChemDraw, often at a greatly discounted student rate, there are numerous free programs like KnowItAll® (http://www.knowitall.com/academic/welcome.asp), Marvin Sketch (for both PC and Mac), ChemSketch (www.acdlabs.com) (only for PC).

The experiments to be done in this course are listed above. While you will work in groups of two or three, each person must keep a notebook and write an **independent** report (i.e. you do not get together to answer the questions, etc.) With the exception of the first lab/report on Silicones, you will have one week to complete each experiment and you must submit your report electronically via Canvas the following **Friday** (one week + 2 days). Comments will be returned to you via Canvas as well. The first lab (silicone/silly putty) will be done by everyone on Wednesday, October 11 in the quant lab. This report will be due **Wednesday, October 18** by midnight, only one week after the lab is completed. It will be the simplest report and will give you the opportunity to learn the many details of writing an ACS journal style report on simpler concepts. It will be graded promptly and returned BEFORE you second report is due so you can correct mistakes you made.

There will be penalty of 5 points for each day that the questions and the report is late, with weekends counting as one day.

Grading for the **lab and report** will be approximately as follows.

I.	Prelab understanding, attendance, technique, yield, and purity	15%
II.	Notebook	10%
III.	Report	75%

Each of these sections is discussed below.

Lab Notebooks

I. **Yield and purity** are self-explanatory. These will be graded on an incremental scale, e.g., no yield, poor yield, good yield. **Technique** will be based on how you handle the equipment and chemicals, your understanding of the procedures, your safety habits, how your follow instructions, and, most importantly, ***how thoroughly you prepare for the lab before you actually arrive***. **Preparation:** The most important thing you can do to enhance your grade, your understanding of the experiment, your successful completion of the work, and everyone's safety is to spend at least an hour preparing for the lab before Monday afternoon. Read the experiment in this booklet, look at the references, and begin to answer the questions at the end of the experiment. Some answers will become clear as you work in lab. Also calculate the amounts of starting materials that you will need (mmoles, grams, mL), look up physical constants that are needed (molecular weight, density, boiling points), and safety hazards for all chemicals and equipment. We will also factor in punctuality, leaving lab for extended periods, ability to work in a group, how well you clean up, etc.

II. **Notebooks.** The following information is what should be included in any research notebook and is, therefore, appropriate for this course. The notebook should be a bound notebook with factory prenumbered pages. **NEVER** remove pages from a notebook. This implies dishonesty (e.g., results that were deleted because they did not agree with the hypothesis). This procedure is mandated for patents too. However, since this is a relatively short course and because you probably have an old organic or quantitative analysis lab notebook, you may use these for this course. All entries should be made directly into the notebook in as legible a form as possible (not on paper towels!). A sample of a research notebook pages is given on the next page and should approximate your notebook. Note that there is plenty of detail, but it is concise. Grammar is not always the best, but that can be corrected in a report. Getting the information into the notebook is more important. (*Please note your lab partner's names in you notebook.)* ***Past Tense!***

- Table of Contents: Leave a few of pages at the beginning of the notebook for this and keep it up to date. List the name of each experiment or give the formula for the compound prepared, e.g. "Synthesis of $(Me_3Si)_2NP(Me)(Ph)$ " and list the page number in a column on the right.
- Begin each new experiment on a new page (right hand side. Reserve the left-hand side of the page for tare weights, calculations, physical constants, etc.) Put a title at the top of the page.
- Draw the structural formulas for reagents and products
- Write the amount of each reagent used in a column under its formula in the equation. This should be done before you come to lab.
- molecular weight (grams/mole)
- number of moles or mmoles

- grams or mL
- other relevant information used to calculate the amounts (density, molarity)
- The actual experimental work should then be written in paragraph form and should be written as you complete the process. For example, "The Grignard reagent was transferred to the addition funnel via syringe and then was added dropwise to the stirred solution at -78 °C over 50 min." All of this should be on the same page and continued to the next page. *Put a date in the margin each time you write the procedures performed on a new day*. In a real research lab, you may be doing several experiments at the same time. EACH experiment should be started on a new page. If more than one page appears to be needed, estimate the number of pages that should be skipped to complete that experiment. If enough pages are not skipped, simply write "continued on page xx" at the bottom of the page. NEVER use a diary format where several experiments are recorded simultaneously. This is a nightmare to decipher when it is time to publish. ***Past Tense!***
- If the procedure or glassware is unfamiliar and/or complicated, include a sketch in your write-up.
- Summarize your results at the end of the experiment. Include yields, boiling or melting points, spectral data etc. Write some comments about whether the experiment worked or what might be done to improve it next time. Don't just quit leaving the reader to wonder if you completed the experiment.
- **All spectra, GPC, TGA, DSC, etc., should be clearly cross-referenced by notebook page number for easy referral** (e.g., ^{1}H NMR #21c is the third proton-NMR spectrum recorded for the experiment that is written-up beginning on page 21 of the notebook.)
- In a real research lab, write up and *date* every experiment you do (on a separate page), even if it is the 50th time you are doing it. Repeated procedures need not get a separate entry in the Table of Contents. Simply add the new page number to the original entry.
- All spectra, GPC, thermal analyses, and any other data acquired on a separate piece of paper must be completely labeled with
    - notebook page and your name
    - formula or, if unidentified, give the reaction
    - relevant purity or how purified
    - relevant reaction conditions
    - solvent (for spectra)
    - conditions used to acquire data (e.g., temperature, flow rate, sweep width)
    - date of data acquisition
- **Scanned Copies of Data:** Please scan (or photograph) all additional data such as IR or NMR spectra and paste this into the report. Scan in BLACK and WHITE to keep images small. Also, use the 300 dpi setting. Only scan your overlays for the semiconductor experiment in color.
- Scan your notebook pages (black and white) and merge these into the Appendix of your Lab Report.

Student's Name

Date 2-25-85 **3**

Subject

Instructor's Name

$(Me_3Si)_2C(H)Cl$ + Mg $\xrightarrow{THF}$ $(Me_3Si)_2C(H)MgCl$

194.5 g/mol — (3.4g)

190 mmol — 272 mmol

37.0g — 24.3g/mol

in ~~136~~ 156 ml THF — in 56 ml THF

should have been 136 ml

THF ~ 190ml | PCl_3 137.3g/mol, d = 1.574 g/ml

16.4 ml

188 mmol

$(Me_3Si)_2\overset{H}{C}-PCl_2$

MW = 261

Grignard was prepared by the literature procedure in a 500 ml flask (Organometallics, 1985, 4, 339.) Difficult to start rxn eventually added more (~½ ml) BrCH₂CH₂Br (+ initial 1½ to 2ml). Also heated the rxn w a mantle during addition of ~ first half of soln. Refluxed 1 hr after addition was complete. Stirred another hour at RT.

A 1000ml 3-necked flask equipped with a paddle (mechanical) stirrer, N₂ inlet and 500ml addition funnel was charged with PCl_3 and THF. Soln was cooled to 0° and Grignard soln was transferred to addition funnel via cannula. Grignard was added dropwise over 1 hr while stirring vigorously. Ice bath removed and mixture was stirred overnite.

Solvent was removed under vac. Inadvertently added THF instead of hexane. Filtered. Removed solvent. Added hexane. Filtered again. Washed several (4 or 5) times with 50ml hexane. Removed solvent. Distilled residue through 10cm column

^{31}P38A B.P. 50-57°/0.01 torr. Yield. 7.5g 1st fraction 50-55°. ^{31}P38A $^{31}P\delta(CH_2Cl_2)$ 225.6. Middle fraction 55-58°/0.01. Yield 32.7g

P38B $^{31}P\delta(COCl_2)$ 227 small but not insignificant; 225. Last fraction. 8.7g (discarded

^{31}P38C $^{31}P\delta(CH_2Cl_2)$ 227 ½ half the 225. ^{1}H of middle cut - ^{1}H38B small doublet at δ1.28 J = 13.18. failed to tape shift of Me₃Si doublet onto spectrum!

Total yield: 40.2g (154 mmol) 81%

Lab Reports

The report is an important part of your grade and should contain each of the following 5 sections, in the format shown. Because an important part of being a functioning chemist in today's world is handling a word processor, you **must**

- prepare reports in MSWord using the specific format described in the next few pages
- ***use a drawing program to draw all structures***.* Learning to use such a program is part of your homework. Equations copied from pdf files, your friends, etc. will not be accepted.
- use subscripts and superscripts
- spell check
- please proofread your report
- insert page numbers in the center of the footer
- submit **ALL REPORTS AND SUPPORTING INFORMATION ELECTRONICALLY via Bb** (Canvas) as a single MS word file for each report**.** You should scan spectra and notebook pages and then paste them into your MS Word document so that you need to submit only one document per report.

The following sections should be included in your lab report. You should use *The ACS Style Guide: Effective Communication of Scientific Information* to make sure you follow the required ACS format for an article in an ACS journal, e.g., *Inorganic Chemistry*. Note that the format for references is different in non-ACS journals. You need to adhere to the ACS format (that includes using US spelling and not British; using superscripts for reference numbers in the text, but listing the references with non-superscript numbers at the end of the manuscript). The *ACS Style Guide* will also be useful for all of your research proposal for the Ph. D. requirements. Please note that you can download chapters of the *ACS Style Guide* if you are on campus or connected to SMU by VPN. The link is http://pubs.acs.org/isbn/9780841239999.

Your report will be graded for comprehensively, including for content, style, **grammar**, and **concise and clear scientific wording**, and format. If it does not conform to this, the report will be returned to you for corrections. If done correctly half of the points deducted for each corrected portion will be returned. This option applies to only one of the six reports.

The following pages provide more detail on content and appearance of the report.

* While you may purchase a program like ChemDraw, often at a greatly discounted student rate, there are numerous free programs like KnowItAll® (http://www.knowitall.com/academic/welcome.asp) and ChemSketch for PC users. ChemDoodle (nominal charge of ca. $20, but works for both PC and Mac and even Linux, and works well when properly used). (Hey if you can afford to buy your own Mac, then you can afford $20. Consider it the cost of a textbook which you don't' have in this class.) Note, that it takes some setup and some time to learn to properly use these programs, but as full-fledged, B.S. Chem major, you MUST be able to properly draw structures.

Title (bold, one size larger font, e.g., 14 instead of 11 or 12, cap all significant words)

Authors full name: In this case list your name first and place an asterisk (*) after it. Then list your lab partners as coauthors.

Department of Chemistry, Southern Methodist University, Dallas, TX 75275

Date report is submitted

Abstract (this is the heading - in bold face, only first letter of the word capitalized)

Write this section LAST so that it accurately summarizes the rest of the report. This is the most important part since it is the executive summary of the purpose or goal, the experiment or method used, and results in a very short, concise format. A short paragraph, or about the length of the rest of this title page, should be sufficient for this class. Read a few journal articles so you fully understand the information that must be in an abstract.

Double space this and the entire report. Margins should be at about 1 inch on all pages. Place page numbers at the center bottom of each page (but not on the first page). All this can be done with the footers in MSWord. AVOID THE USE OF FIRST PERSON. It is very rarely used in scientific writing. In fact, even oral presentation should sparingly use "I" or "we", but sometimes it is useful for emphasis.

Introduction (use this style - bold; start on a new page if the first page is nearly full, or leave at least one skipped line as done here.)

The introduction sets the scene or explains the state of the field in terms of published work. It uses this background information to point out an unanswered question or missing piece of data to set up the purpose or goal of the work reported in this manuscript. You should

be able to find a few other ways that the compound has been made, but then point out why the method(s) need improvement. It is sometimes useful to use chemical reactions or schemes here. More on how to do that in the Results and Discussion Section below. You will need to find a couple useful literature references related to the work. This can be done by reading the reference(s) provided with each experiment. End the section with the specific question to be answered or goal. This is the "what" and the "why" section.

Experimental

I usually prefer to have this very detailed section at the end of a manuscript because it interrupts the flow of the manuscript, but until further notice, put it after the introduction. You will benefit by providing the details early in the report. Note this is NOT a cookbook, so it is written in PAST tense and it is written professionally following strict rules.

The first paragraph lists the source of all reagents and solvents and how they were purified (if relevant). Look at one of my papers as an example. Do not start a sentence with a number. If the name of a compound is the first word in a sentence, (e.g., 2-butanone), reword the sentence. Sometimes something as simple as "Then 2-butanone was added via syringe." The general methods and special precautions are also listed (e.g., "all reactions were done in flame-dried glassware using standard Schlenk techniques to exclude oxygen and moisture..") The second half of the paragraph lists the model numbers of the instruments used and give details of sample preparation. (Sample : NMR spectra were recorded on a SGI/Bruker DRX-400 spectrometer using $CDCl_3$ and D_2O as a solvent. Positive ^{1}H and ^{13}C NMR chemical shifts and ^{31}P, ^{29}Si, ^{13}C NMR shifts are downfield from the external references Me_4Si and H_3PO_4, respectively. Elemental analyses and IR spectra were obtained on a Carlo Erba Strumentazione CHN Elemental Analyzer 1106 and a Nicolet 560 IR spectrometer, respectively.)

The next paragraph gives a very detailed description of how the experiment was done. (This includes amount in grams or mL and in moles usually in parentheses depending on the wording used. See a real journal article). This must be in PAST tense and cannot simply be a copy of the procedure given in lab since the actual procedure from lab will vary slightly. A great

deal more detail on procedures (for example syringing, preparing an NMR sample, freeze-pump-thaw, etc.) should be given since this is a lab report, even though these usual processes are not described in a journal article. List all characterization data in this section. Once again, there is a specific format for doing that. See both the *ACS Style Guide* and a real ACS journal article for format.

Results and Discussion

This is typically the longest section of a journal article. While all four sections (Abstract, Introduction, Experimental, and Results and Discussion) are equally important, the Results and Discussion (R&D) often takes the most time and thought. (By the way, all acronyms must be defined in the text the first time they are used.)

In this section state what was done (not the details but the general method -- see journal articles) (e.g., "An N-silylphosphoranimine was made by oxidation of xyz phosphine using hexachloroethane." If the lab involves a synthesis, indicate how you isolated the product, purity, and yield. Then state how the compound was characterized and discuss how the data confirms that this was indeed your product. Discussion of structure may also be relevant. If there are impurities, what discuss the evidence that indicates what these might be. Discuss problems and source of error (decreased yield, impurities, etc.) If the synthesis/experiment were to be repeated, what should be done differently. Most of the above will be separate paragraphs, though, in some cases, a couple of these topics may be merged. The questions you answered may also indicate what/how to interpret the results and may indicate what topics are important to discuss. Use equations that you have drawn using professional software (see above), tables, schemes, and figures to enhance this section (see *ACS Style Guide* for how to do these). Insert them into the text at the appropriate positions and refer to them in the text by the proper numbers. Please ask the instructor if you have questions.

Conclude with a summary paragraph(s) that relate(s) the work back to the introduction. This will state the relevance of the methods/techniques/instrumentation, the use and relevance

of the materials/compounds prepared, and where/why these are applicable in other fields of chemistry and science or technology.

References *(ACS Format)*

1. Wisian-Neilson, P.; Onan, K. D.; Seyferth, D. *Organometallics* **1988**, *7*, 917 – 921.
2. Wisian-Neilson, P.; Ford, R. R.; Ganapathiappan, S.; Islam. M. S.; Raguveer, K. S.; Schaefer, M. A.; Wang, T. *Phosphorus, Sulfur, and Silicon* **1990**, *51/52*, 165 – 168.

3. Note that in the text, the references numbers are superscripts, but here they are not superscripts, they are number followed by a period (not parentheses), and the text is wrapped with alignment as done here. This is not done with the tab key, so learn (or ask) how this is done.
4. Doing your references incorrectly will result in major deductions for the lab report. It's easier to do them right the first time (and gets you a lot better for your grade.)

Notebook Pages

This is specific to Chem 5192 reports and not journal articles. Please scan your notebook pages, spectra, etc. (in black and white or gray scale for everything except the semiconductor overlays), and cut and paste from the scans into this report. Scan them to pdf since jpeg, tiff, etc. files are MUCH MUCH larger. Your lab report should not be over 5 Mb in size if you do these scans correctly.

SAFETY

The safety of people in the laboratory comes above all other considerations. Each individual working in this lab is responsible for safety in the area under his/her control and shares responsibility for the overall safety of the lab. The following is a **partial** list of some commonly overlooked safety procedures. Please keep them in mind at all times. These procedures are not specific to this course and should be followed in every laboratory in which your work. (Yes, even in your undergraduate research lab!!!!)

1. **THINK!** Plan your work and take into account all possible sources of error or experiment failure.
2. **Wear safety glasses** and lab coats **at all times**, even when just "checking" something simple or when **washing dishes**. Use the pull-down safety shield on the hoods as much as possible.
3. Carry out ALL reactions, column chromatography, precipitations, transfers etc. in a hood.
4. Never heat a closed or partially constricted system or a system that may become closed via plugging of a trap. Each nitrogen line attached to any reaction, distillation, etc., should be vented through a mercury manometer.
5. Wear gloves when handling any chemicals or solvents, even the "harmless" ones. Wash your hands with soap frequently.
6. Keep all exits clear. The safety exit to the lab room, e.g., Room 31, is near the back of the room. This door leads into the departmental instrument room.
7. Keep all aisles clear of cans (including the solvent cans, boxes, stools, chairs, electrical equipment, etc.) Also keep all drawers and cabinet doors closed.
8. Memorize the location of all fire extinguishers. Be sure to keep sand buckets handy (and labeled) so you can always grab them in an emergency. This means you do not put them in or under your hood. Use only sand for all hydride, organometallic, or phosphine fires (virtually all fires in this lab).
9. Keep all solvent bottles and cans in the appropriate cabinet below the hoods when not in use. Do not store any chemicals in the cabinets with vacuum pumps. Store acids in a separate cabinet. Keep all other chemicals in the hood or in designated shelves. Do not leave bottles or cans on the bench tops or on the floor.

10. **Label ALL** flasks and bottles, even things that you intend to use "right away". It is especially important to **date** and **label** everything stored in a refrigerator. The refrigerator should be cleaned and defrosted about every six to nine months to prevent dangerous messes. (We don't have a refrigerator in this course, but you should know this anyway.)

11. Do not pour chemicals down the sink. Disposal containers are available --- chlorinated hydrocarbons, non-chlorinated solvents, solid waste (silica gel), and heavy metal waste. Be careful of what you pour into these containers. The solvent should be relatively clean or we will have a unique and dangerous reaction.

12. Never throw out chemical containers until ALL chemicals have been carefully removed. Remove all labels, clean each container, and make sure all solvents have evaporated before discarding. This includes all solvent cans or bottles, ampules, broken glassware, and reagent bottles. Destroy all traces of organometallics with iso-propyl alcohol and then water.

13. **ALL** traces of spilled mercury must be vacuumed up immediately and placed in a waste container. Do not just sprinkle sulfur over it! We have a special suction flask for this purpose. Be sure this device is completely sealed up before returning it to storage. Never throw any mercury into the sink or trash. Dirty mercury can be cleaned fairly well by filtering through a small pin hole in a filter paper.

14. Dispose of all glass including disposable pipets in the container labeled "Broken Glassware." It must be cleaned prior to disposal.

15. Watch for electrical hazards such as frayed or broken electrical cords or damaged heating jackets; excessively warm motors; ovens, pumps, or other electrical equipment operating near chemicals.

16. **Smoking, Eating, and Drinking** in the laboratory are **PROHIBITED** at all times.

HOUSEKEEPING

It is a standard policy to maintain clean, neat, and orderly labs at all times since these are generally safer and more efficient labs. Some important items are listed below, though these will vary from lab to lab this semester. Listen to your instructors for specific information. The following applies in the Wisian-Neilson lab.

1. **Dishwashing**. Dishwashing requires special diplomacy and courtesy. Be sure you always take care of your own dirty glassware or an equivalent amount of dishes. In this course, each student will be expected to come at least one time this semester on the day after lab (i.e., Tuesday or Thursday) to remove glassware from the KOH bath, etc. A signup sheet will be posted on the first day of lab.

   Glassware should be placed in a KOH bath (recipe below) for at least four hours. After a thorough rinsing with water, the items should be rinsed (**not soaked!**) in an acid bath, rinsed again with water, hung to dry, and finally oven-dried. When something is needed immediately, rinse it with acetone after the usual base and acid bath treatment, and then dry it in the oven. Be sure to save the acetone rinse for use in making dry ice baths. Never put anything away unless it is spotless! Never leave glassware, especially fritted items in the KOH bath for extended periods of time (over 1 day) because the glass gets badly etched, flasks become eggshell thin, and frits become sieves.

   The following procedure for dishwashing should be followed:

   a. remove clean glassware from the oven and put the dry dishes into the correct drawers or cabinets (if locked, please leave them in the oven to avoid conflicts with Chem 1301 or see the TA for a key.)
   b. transfer glassware on the drying rack to the oven,
   c. remove everything from the KOH bath, rinse, etc. (see procedure above). Be sure to carry this process straight through to the drying rack. It is a waste of time to leave things in the acid bath. Don't "dig around" in the base bath to find your glassware, because this causes breakage and cut fingers.

Recipe for KOH Bath:

~2 lbs (~ an 800 mL beaker full) of KOH pellets (reseal the KOH bag, please)

~ 2.5 gallons of iso-propyl alcohol (**not** ethanol)

Carefully dissolve the KOH in a minimum amount of H_2O (ca. 800 mL); then add isopropyl alcohol.

Recipe for Acid Bath:

~ 50 to 100 mL conc HNO_3 in 1 or 2 gallons of water.

The following items should NEVER BE PLACED IN A KOH BATH!!!

-*glass syringes and needles* (Needles corrode in the acid baths, too, so don't put them in that either!)

-*NMR tubes* (in extreme cases you can try filling a tube with KOH and setting the tube in a beaker for a few hours)

-*the washers and O-rings from Teflon stopcocks*

-*thermometers*

-*anything so small it could be lost*

-*anything with lots of grunge*. (Preclean such items with acetone, CH_2Cl_2, appropriate CHEAP solvents, or soap.)

2. **Phosphine and sulfide odors** must be contained by rinsing **ALL** exposed items of glassware (flasks, dispo-pipettes, stoppers, NMR tubes, etc.) in a **dilute bleach** solution **in the hood BEFORE** any further washing is attempted. If it smells like phosphine or thiol in the lab, you are not doing it right or your hood isn't working.
3. Glass **Syringes** should **NEVER** be soaked in KOH or HNO_3 or soap. Instead, clean them with soap and water (brush) and acetone. Remove the needle and the barrel from the syringe **immediately** after use to prevent these pieces from fusing. Also rinse needles immediately to prevent plugging. This is also important for the cannulas. Store clean syringes with barrels inserted to keep them free of dust and store cannulas with corks or septa on the ends to prevent injuries.
4. Clean fritted filters **immediately** after use and **do not** soak them in the KOH or HNO_3 baths. Use an aspirator to pull water and other cleaning solutions through the frit. (Be careful not to mix acetone with HNO_3 in the process since these react violently!).
5. Lab bench tops both in and out of the hoods should be dusted regularly and scrubbed occasionally with soap and water. People working in the same lab must **cooperate** in keeping general use areas, reagent shelves, balances, cabinets, etc. clean and well-organized. This goes also for departmental areas, especially the NMR rooms. Keep such areas free of clutter, excess paper, Kimwipes, etc. This can easily be done in a couple of odd moments.
6. Keep the sink drains clean. Keep mesh over the sink bottom to prevent glassware breakage and loss of stir bars down the drain.
7. Be careful not to spill things on the floor, especially when washing dishes. Everything stains (or dissolves) the tiles and the only way to keep it from complete ruin is to **work carefully!** Wipe up spills of anything on the floor or counters (especially around the KOH baths). Remember that you are not the only/last one to use the lab space.

8. Keep the equipment for each experiment in the designated drawers. Keep all stopcocks fastened in the proper piece of equipment.

EQUIPMENT USAGE AND MAINTENANCE

Please ask for help if you have never used a piece of equipment. In most cases a simple explanation can be provided quickly. In other cases you should consult the manual. The following are some general things you should know. Those that you will definitely need in this course are marked with an asterisk.

1. **Vacuum pumps.** Proper maintenance requires the following:
   - *a. Check the oil levels frequently.
   - b. Change the oil no less than every **four months.**
   - c. Tape or tag the pump with the date of the oil change.
   - d. Turn pump off when not in use (even overnight).
   - *e. Always bleed air into pumps within seconds of shutdown to prevent oil from backing up into vacuum tubing.
2. **Heat Guns.** *Always* run them on cool for a few minutes before turning them off; do not set them down while cooling. Keep the nozzle at least one inch away from the item being heated to prevent back-up pressure.
3. **Teflon Stopcocks.** If a stopcock is leaking, clean it, *very lightly* grease the O-rings, and then heat the glass gently with a heat gun while the stopcock is closed. This will cause the Teflon to soften slightly and seal better. Cool before opening.

*4. **Dewars.** Precool *slowly* by adding small amounts of liquid nitrogen at short intervals. Wait until the boiling subsides before each small addition. Make sure that all glass Dewars are properly wrapped with tape or plastic mesh for safety.

5. **Tesla Coil.** Always unplug when not in use to avoid burning out the capacitors.

*6. **Gas Cylinders and Regulators.**
   - a. All gas cylinders must be secured to a wall or bench with a strong clamp.
   - b. Make sure that all threads are dirt/grease free when making connections.
   - c. Use a wrench to tighten regulator to the cylinder but don't over-tighten.
   - d. **To open**, turn the regulator screw counterclockwise until it is fully released, then open the main cylinder valve cautiously while standing with the cylinder between you and the regulator. Slowly turn the regulator screw to the desired pressure (less than 8-10 lbs for our work).

e. Keep the main cylinder valve closed when not in use and keep the regulator screw in the counterclockwise released position. This is important because the diaphragm can break if this valve is opened (tightened) when the main cylinder valve is opened.

*7. **General Electrical Equipment.** Always unplug electrical equipment when not in use to prevent accidents and burned out motors. Generally, do not leave vacuum pumps, mechanical stir motors, heating mantles, etc. on overnight.

*8. Variacs should be operated at 120 volts not 140 volts and should not be used above a setting of 50. Usually something less than 30 is sufficient, especially near the beginning of an application. It seems that the smaller the heating mantle, the lower the setting.

*9. Oil the lab jacks and clamp screws periodically or when they begin to turn with difficulty.

General Experimental Techniques

1. Use plastic clips or rubber bands to hold together all ground glass joints.
2. Use stopcock grease or Teflon sleeves on all ground glass joints.
3. Clip or wire condenser hoses to glassware
4. **Never** bleed N_2 into vacuum lines with the McLeod gauge open unless there is an outlet in the system to relieve the pressure (i.e. an opened stopcock, vacuum source, open ended mercury manometer)
5. NMR samples:

   ^{1}H: Mix sample to solvent in ca. 30 % v/v solution. $CDCl_3$ is the most common and least expensive solvent.

   ^{31}P and ^{13}C: sample:solvent = 30 – 50 %
6. Always beware of closed systems where pressure can build up. **NEVER** do a reaction in or heat a closed system!!!!!

- Disability Accommodations: Students needing academic accommodations for a disability must first be registered with Disability Accommodations & Success Strategies (DASS) to verify the disability and to establish eligibility for accommodations. Students may call 214-768-1470 or visit http://www.smu.edu/provost/alec/dass to begin the process. Once registered, students should then schedule an appointment with the professor as early in the semester as possible, present a DASS Accommodation Letter, and make appropriate arrangements. Please note that accommodations are not retroactive and require advance notice to implement.

- Religious Observance: Religiously observant students wishing to be absent on holidays that require missing class should notify their professors in writing at the beginning of the semester, and should discuss with them, in advance, acceptable ways of making up any work missed because of the absence. (See University Policy No. 1.9.)

- Excused Absences for University Extracurricular Activities: Students participating in an officially sanctioned, scheduled University extracurricular activity will be given the opportunity to make up class assignments or other graded assignments missed as a result of their participation. It is the responsibility of the student to make arrangements with the instructor prior to any missed scheduled examination or other missed assignment for making up the work. (University Undergraduate Catalog)

August

Sun	Mon	Tue	Wed	Thu	Fri	Sat
		1	2	3	4	5
6	7	8	9	10	11	12
13	14	15	16	17	18	19
20	21 SMU classes begin	22	23	24	25 No lecture at 2 pm start working on silicone questions	26
27	28	29	30 Draft of silicone questions due at midnight	31		

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September

Sun	*Mon*	*Tue*	*Wed*	*Thu*	*Fri*	*Sat*
					1 Lecture on silicones and phosphoranes	**2**
3	**4** Labor Day	**5**	**6** Silicone questions (final version) due (midnight)	**7**	**8** Lecture on sulfur-Ag complex and Jacobsen	**9**
10	**11**	**12**	**13** Phosphorane questions due (midnight)	**14**	**15** Quant exam will run into lecture time – no lecture	**16**
17	**18**	**19**	**20** Sulfur-Ag questions due (midnight)	**21**	**22** Lecture on ATRP	**23**
24	**25**	**26**	**27** Jacobsen questions due (midnight)	**28**	**29** Lecture on semiconductors	**30**

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October

Sun	*Mon*	*Tue*	*Wed*	*Thu*	*Fri*	*Sat*
1	**2**	**3**	**4** ATRP questions due (midnight)	**5**	**6** Quant exam will run into lecture time – no lecture	**7**
8	**9** Fall Break	**10** Fall Break	**11** Semiconductor questions due **AND** Lab week 1 Silicone lab (everyone meet in Quant. lab)	**12**	**13**	**14**
15	**16** Lab week 2	**17**	**18** Lab week 2 **Silicone report due (**(midnight)	**19**	**20**	**21**
22	**23** Lab week 3	**24**	**25** Lab week 3	**26**	**27** **Report 2 due** (midnight)	**28**
29	**30** Lab week 4	**31**				

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November

Sun	Mon	Tue	Wed	Thu	Fri	Sat
			1 Lab week 4	2	3 Last Day to Drop **Report 3 due** (midnight)	4
5	6 Lab week 5	7	8 Lab week 5	9	10 **Report 4 due** (midnight)	11
12	13 Lab week 6	14	15 Lab week 6	16	17 **Report 5 due** (midnight)	18
19	20	21	22	23 Thanksgiving	24	25
26	27	28	29 **Report 6 due** (midnight)	30		

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December

Sun	*Mon*	*Tue*	*Wed*	*Thu*	*Fri*	*Sat*
					1	2
3	4 Last Day of classes	5 Reading Day	6 Reading Days	7 **Final Exam** **3 - 6 P.M.**	8	9
10	11	12	13	14	15	16
17	18	19	20	21	22	23
24	25	26	27	28	29	30
31						

2017