Chemistry 4260

Advanced Integrated Laboratory Techniques

EXPERIMENTS

Spring 2018

Every Student is required to perform experiments #1, #2, #3, and #4. Experiments #3, and #4 are the only ones where students may work in pairs. All other experiments are to be performed individually.

The remaining two required experiments may be chosen from a list that will be provided after the first week. It is however suggested that a greater depth of experience may be attained by selecting two closely related experiments for the final two.

Report Format :

Each experiment will have a different report with different requirements. Most of these procedures include specific report elements that must be included. However, all reports will share these common elements:

Remember that the experiment is a vehicle to get you to think about and work on the questions associated with each experiment. Do not wait until you begin writing the report to start looking at and working on the questions. Begin the questions at the same time or before performing the experiment.

1. **Short** introduction to explain the nature and goal of the experiment. (No more than half a page.)
2. **Brief** but accurate discussion of relevant theory and/or the procedures used to performed the experiment. In the case of a synthetic experiment, outline the synthetic approach utilized. No more than 3 pages and **DO NOT CUT AND PASTE FROM THE HANDOUTS**.
3. Data:
   a. Delineate the procedure/process being performed and the crude data obtained, if any.discuss how each reaction (measurement) is performed and discuss any problems that arose, how points were chosen, how peaks were assigned.
   b. Make note of any important observations about the data or its collection that may be relevant to its accuracy or useability.
4. Results:
   Tabulate any calculated or derived results with descriptive labels.

5. Discussion of Results:
   Describe the meaning, relevance, and implications of the results obtained in the context of the system being investigated.

6. Experimental Notebook Copies: Only for "wet experiments".
    a. the copy pages from your laboratory notebook for the experiment.
    b. **It is imperative that it be clear and legible so that the experiment can be reproduced in the lab by a third person using only these sheets and that all data necessary had been recorded during the execution of the experiment.**
7. Answers to experimental Problems.
    Each experiment has a set of questions and problems that may require some significant computer or pencil on paper or library work to solve.
8. Interpretation of any spectra required or requested as part of the experiment. It should consist of at least a copy of the spectra with structural assignments for the major spectral features shown indicated with a structural drawing.
9. Excel spreadsheet, e-mailed to me. If the experiment requires a spreadsheet a complete copy of the spreadsheet used must be included, via e-mail, so that it can be checked for form, accuracy, and completeness.

Excel spreadsheets:

Some general rules and guidelines for spreadsheets in this course.

Only the following may be entered as a direct entry:

a. Labels
b. Raw experimental data obtained in the laboratory.
c. Constants.

Everything else must be calculated by reference to the above data and constants. Values that are generated by a macro must be labeled as such with a notation as which cells were used by the macro.

Point Break-down: 20 point per report:

Format:	Proper	2 points
	Complete	2 points
	Results	4 points
	Discussion	4 points
	Questions	8 points
	Total	20 pts

!!!!!!!! Important Dates !!!!!!! :

A report is required from each person by each date specified below. The report must be handed to me (Dr. Arney) on or before **5:00 PM** of the date. After the date, 20% per School Day will be deducted from any potential grade. After five days it is a zero.

Feb. 3, Mar. 2, Mar. 23, Apr. 13, Apr. 27, the last before the scheduled final time. NO WORK WILL BE ACCEPTED AFTER THE SCHEDULED FINAL EXAM TIME.

ADVISE:

(1). It is to your advantage to attempt to perform two or more experiments concurrently as most will have significant waiting periods which could be utilized for other experiments and work-ups.
(2). Prior to lab you must obtain an acceptable procedure for the experiment, from the indicated sources, and prepare to perform the experiment by studying and understanding the operations involved and the nature and handling of the materials to be used.
(3). The multi-step synthesis is best started as early as possible and performed concurrently with other experiments.
(4). Each week, a one-hour lecture session will be devoted to covering a technique, problem, or concept. These sessions will generally be associated with that week's dry lab work.
(5). You will not be "***prepped***" for each wet lab and are responsible for having the appropriate procedures and knowing the proper use of equipment. However, potentially hazardous operations will be monitored and **NEW** procedures, such as vacuum distillations will be discussed and demonstrated as necessary.
(6). When stuck, ask questions? Often you will receive assistance in figuring out the problem, other times you may get help figuring out why you are stuck.
(7). The T.A.'s primary function in the laboratory is for safety and to provide the necessary material and equipment. The T.A. is not a source of information on the performance of the experiment and does not have a clue to the question "does this look right?".

Hydrogen Atom and Hydrogen Molecule Cation (Experiment#1):

All students must perform this experiment. Each student will independently set up the Excel spreadsheet, perform the necessary operations on the data, and answer the accompanying questions for the lab. With a little planning, the entire Excel set up for the lab can be performed in one afternoon. However, most require 2 afternoons, one for the hydrogen atom and its questions and one for the hydrogen molecule cation.

Hückel Molecular Orbital Theory (Experiment#2):

All students will perform this experiment. Each student will independently set up the Excel spreadsheets, perform the necessary operations on the data, and answer the accompanying questions for the lab. This experiment requires the use of Gaussian 09W.

Stability Constant for $Ni(Gly)_3$ Experiment (Experiment #3):

All students must perform this experiment. Students will work in pairs for the "wet" portion of this experiment. Each pair will collect their own data, but each student will independently work up the data. With a little planning, the entire wet portion of the experiment can be performed in 1 afternoon. However, most require 2 afternoons; one to prepare the solutions and one to perform the measurements.

Kinetics Experiment (Experiment #4):

All of the students will perform the **Kinetics Experiment**. The experiment requires a significant number of points at different temperatures in order to produce decent results. Students will work in pairs for this experiment. Each pair will perform the experiment in duplicate at a single temperature. Different pairs will use different temperatures. However, **each person must** work up the pair's data separately for the two runs at their temperature. The results (rate constants and temperatures) will be shared with other groups since each person needs at least three temperatures over at least a 10 ^oC range. More temperature points may be used (possibly better results), but at least three are required. That means at least six (6) sets of data, two (2) for each temperature. **I STRONGLY SUGGEST THAT YOU CHECK THAT THE DATA IS REALISTIC WHEN SELECTING POINTS FOR YOUR WORK**

Experiments to be Performed: .

1. **‡Approximate Solution of the Hydrogen atom (H) and Hydrogen Molecule Cation (H_2^+) by Numerical Methods in a Spreadsheet.** The wavefunction for the one electron hydrogen atom is approximated by a linear sum of four Gaussian functions and its coefficients are optimized by solving the Schrodinger equation for the best energy. The techniques learned from the hydrogen atom are then applied to the one electron hydrogen molecule cation. This lab focuses on introducing the numerical matrix methods utilized in *ab-initio* software and becoming familiar with much of the non-obvious elementary aspects of MO work. Application of the derived approximate wavefunction will be compared to the exact wavefunction. The Matrix-LinAlgebra add-in for Excel, allows us to focus on the general methods more clearly.
2. **‡Hückel Molecular Orbital Computational Lab.** Introduction to the theory and application of Hückel MO theory is covered in some illustrative but revealing cases. Provides some introduction to the use of matrix methods and systems of equations. This is a "dry" laboratory focusing on the enhancement of mathematical skills and understanding of theoretical applications.
3. **‡Stability Constants of $Ni(glycinate)_n^{(2-n)+}$**. Handout procedure based that found in ***Angelici***(exp 22). Determination of the stability constants for the complexation of Ni^{2+} ion by glycine as a bidentate ligand. **In addition to the regular report format,** the data and calculations must be neatly and clearly set-up in an MS-Excel Spreadsheet which will be turned in with the report via e-mail attachment.
4. **‡Kinetics Investigation of the Nucleophilic Aromatic Substitution Reaction:** Spectrophotometric measurements of the reaction of piperidine with 2,4-dinitrochlorobenzene will be utilized to determine the rate constant of the reaction at several temperatures and the thermodynamic properties of the transition state will be calculated to gain a better understanding of the rate-determining process.
5. **‡Inorganic Syntheses/Analysis: TBA**
6. **‡Organic Synthesis/Analysis: TBA**
7. **‡Analytical Experiment/Analysis: TBA**
8. **‡Computational Project: TBA**
9. **‡Programming Project: TBA**

Tentative **Schedule for Integrated Lab Activities:**

Date	Topic for Tuesday 1-2:00 PM Overview		
Jan 18	Introduction, Hydrogen atom		
23	H-atom, Hydrogen molecule cation		
25	H-atom, Hydrogen molecule cation		
30	**Hydrogen Molecule Cation**		
Feb 1	**Hydrogen Molecule Cation**		
6	**Hydrogen Molecule Cation**		
8	Huckel Molecular Orbital theory		
13	Huckel Molecular Orbital theory		
15	Huckel Molecular Orbital theory		
20	Nickle Glycinate Stability constants		
22	Nickle Glycinate Stability constants		
27	Nickle Glycinate Stability constants		
Mar 1	Kinetics		
6	Kinetics		
8	Kinetics		
13	Introduction to Least Squares, Statistics, and Matrices		
15	Introduction to Least Squares, Statistics, and Matrices		
20	Introduction to Least Squares, Statistics, and Matrices		
27	TBA according to class needs.		
29	TBA		
Apr 3	TBA		
5	TBA		
10	TBA		
12	TBA		
17	TBA		
19	TBA		
24	TBA		