

## Chemistry 475 Laboratory

### Introduction

The CHEM 475 laboratory is structured to resemble what you might experience in an industrial research laboratory as a B. S. chemist or as a Ph. D. candidate at a major research university. The individual exercises are designed to expose the student to some of the techniques and methods of inorganic chemistry. It is, however, not an exhaustive survey of the field.

### Laboratory Charges

Each laboratory group will start with \$500 of virtual funds to spend on analyses or other services that are not available in the inorganic laboratory. This is a recharge system and it is similar to that used by many research universities and corporations to spread the cost of shared instruments and facilities among the users. The services subject to recharge, their recharge rate and other information about the services are shown in Table 1. Consultation of faculty members, except the instructor, may be recharged at whatever rate that the faculty member feels is appropriate. Before you can use any of these services you must first complete a form describing the sample and what experiment is to be done, which I will sign. Note that there is a special form for the X-ray diffractometer. The completed form is to then be taken to the faculty member in charge of that instrument (X-ray diffractometer, AA, HPLC and electrochemistry) or given to me (all other services). Turnaround time may be anywhere from two days (NMR) to weeks (full X-ray structure).

Service	Recharge Rate	Notes
NMR spectrum <sup>a</sup>	\$10	if run by the NMR staff
	\$5	if run personally
Elemental analysis <sup>b</sup>	\$30	C, H, N only
Mass spectroscopy <sup>b</sup>	\$30	full spectrum (selected compounds only)
	\$10	parent ion's mass only
X-ray structure <sup>c, d</sup>	\$60	full structure
	\$20	lattice constants only
HPLC <sup>d, e</sup>	\$30	
Atomic absorption <sup>d, e</sup>	\$30	
Electrochemistry <sup>d, e</sup>	\$20	

**Table 1.** Services subject to recharge. Notes: a) special experiments (variable temperature, nuclei other than <sup>1</sup>H and <sup>13</sup>C) will incur an additional charge, b) requires 20 mg of sample and proof of purity, c) requires good quality single crystal, d) set up cost, you must run your own samples, e) faculty consultation charged separately.

Each individual laboratory group is responsible for tracking how much it has spent during the course of the semester. It is expected that you will make an accounting of how the money was spent in each of your progress reports. You may ask for additional funds in the first progress report, but you must include a justification.

This recharge system is not very complicated and is not intended to be perfect. However, intentional attempts to defraud or otherwise cheat the system will result in immediate forfeiture of all

remaining and future funds, which will make it very unlikely that you will pass the laboratory portion of the course.

At most research universities each individual researcher must pay for their chemicals and glassware, while in most industrial laboratories these items are covered by the company. You will not be charged for chemicals or breakage under our recharge system. However, you must still pay for broken, lost or otherwise abused glassware and equipment with real money per the Chemistry Discipline's breakage policy.

### **Laboratory Exercises**

By the end of the semester you must have completed seven exercises from the following groups. Some of the Group 1 exercises are continued in Group 2, and these are marked with an '\*'. You may wish to choose your Group 1 exercises with this in mind. All experiments are subject to reagent availability, the demands of scheduling and the instructor's whim.

In all exercises where the product is a known compound (all Group 1 and some Group 2 experiments) "characterized" means that at a minimum you will obtain the IR spectrum, the UV-Vis spectrum (with molar absorptivities), the  $^1\text{H}$  and  $^{13}\text{C}$  NMR spectra (if diamagnetic) or magnetic moment (if paramagnetic), the mass spectrum and the elemental analysis. Melting points should be determined only for compounds whose melting points are reported to be less than 200 °C.

You will need to perform all of the above methods of characterization in the exercises where the products are not known (most of Group 2). Be aware that the elemental analyses and mass spectroscopy data for these compounds may not be available. It is highly recommended that the X-ray crystal structure and the cyclic voltammogram of all new compounds be determined. In these exercises you will also need to establish the purity of your product.

#### Group 1. Choose three.

*Preparation of (Tetra-(4-methylphenyl)porphyrinato)copper(II)* A derivative of the porphyrin cofactor and its copper complex will be prepared and characterized.

*Tin Chemistry* Two tin compounds, one organometallic and the other a coordination compound, will be prepared and characterized. The mass spectrum of the organometallic compound is available, and must be interpreted as part of the characterization.

*Synthesis and Characterization of a Cobalt-Containing Oxygen Carrier* The compound Co(salen), which served as a model compound for *in vivo* O<sub>2</sub> binding, will be prepared. Its uptake of O<sub>2</sub> will be measured.

*Preparation of Quadruply-Bonded Metal Compounds* Two molybdenum compounds containing a Mo-Mo quadruple bond will be prepared and characterized. A phosphine derivative will be prepared, if time permits.

*Synthesis of Aluminum Acetylacetonate* The compound Al(acac)<sub>3</sub> will be prepared and characterized. The mass spectrum of this compound is available and must be interpreted as part of the characterization.

### Group 1. (continued)

*\*Template Synthesis of a Macrocyclic Ligand* A macrocyclic ligand will be prepared using  $\text{Ni}^{2+}$  as a template. The  $\text{Ni}^{2+}$  will be removed to give the free ligand, which will then be used to prepare the  $\text{Cu}^{2+}$  complex. All compounds will be characterized.

*\*Synthesis of Dipyridineiodine(I) nitrate* A compound where iodine is in a positive oxidation state and where there are no oxygen or other halogens present.

*Synthesis of ( $\eta^6$ -1, 3, 5-methylbenzene)tricarbonylmolybdenum(0)* A compound where an aromatic ring behaves as a ligand. This experiment requires a relatively detailed analysis of the IR spectrum of the product.

### Group 2. Choose four.

*Preparation and Characterization of (Tetra-(4-tert-butylphenyl)porphyrinato)and its Complexes with  $\text{Pb}^{2+}$  or  $\text{Sb}^{3+}$*  Similar to the  $\text{Cu}^{2+}$  experiment in Group 1, but with a different aldehyde and a different metal.

*Synthesis and Characterization of the Complexes formed between a p-Block Metal and Goedken's Macrocycle* Use the macrocyclic ligand prepared with the  $\text{Ni}^{2+}$  template, and remove the nickel as in the Group 1 exercise, but instead of preparing the  $\text{Cu}^{2+}$  complex use a p-block metal ion or metalloid ( $\text{Pb}^{2+}$ ,  $\text{Sb}^{3+}$ , etc.).

*Preparation of a Soluble SALEN Derivative and Its Coordination Chemistry* The synthesis of the ligand is similar to the Group 1 exercise, but a different aldehyde will be used. You have the choice of making a complex of this ligand with either manganese, a p-block metal or a metalloid.

*Synthesis and Characterization of a Cobalt Compound* Prepare and identify a pair of unknown cobalt-containing compounds. Elemental analysis and limited mass spectroscopy data are available.

*Correlation of  $10 Dq$  and  $E^0$  in Chromium(III) Coordination Complexes* A series of  $\text{Cr}^{3+}$  compounds will be prepared and the electrochemical behavior characterized by cyclic voltammetry.

*Synthesis and Characterization of the Complex formed by the Reaction of TPA with  $\text{SbI}_3$*  The ligand tris(2-pyridylmethyl)amine (TPA) will be synthesized and its reaction with  $\text{SbI}_3$  investigated.

*Synthesis and Characterization of the Complex formed by the Reaction of TPA with  $[\text{py}_2\text{I}]\text{NO}_3$*  The reaction between the product of the Group 1 experiment and TPA will be attempted.

*Effect of R Group Variation on the Properties of Cobaloximes* The dimethylglyoxime complex of cobalt has been used as a model of Vitamin B12 (cobalamin) in which there is a very interesting carbon-cobalt bond. A series of three compounds with different R groups bound to the Co will be prepared and characterized. Any differences in their physical properties will be correlated to the nature of the R group.

## **Grading Policies**

All parts of the laboratory will be graded holistically on the following a scale of 0 (no evidence), 1 (minimal competence), 2 (some competence), 3 (competent), 4 (exceptional) and 5 (outstanding). The standard for a grade of 5 for each of the laboratory grade components is given below. Primarily what distinguishes the different grades will be to what extent the work does or does not have the required elements and how well the required elements were performed.

### Style Grade

For a grade of 5 a notebook must adhere to the proper notebook style, which is set out in the Virtual Laboratory Manual. These criteria include the following:

- A clear, concise and accurate statement of purpose
- A clear and concise procedure that allows a knowledgeable reader to duplicate the experiment
- Well written description of what was done and what was observed
- Results, including graphs and tables, which are logically organized and understandable.
- Notebook properly maintained (dates, signatures, etc.)

### Results Grade

This grade will be based on what is reported in the laboratory notebook for each exercise (in all cases you must report your yield, any measured quantities and the required spectroscopic characterizations). The results grade may also include whether the yield is reasonable, and whether the measured quantities and spectroscopic data are in line with literature values.

### Conclusions Grade

A conclusion will receive a grade of 5 if it adheres to the criteria given in the Virtual Laboratory Manual for a notebook conclusion. These criteria include the following:

- States the results
- States and analyzes sources of error and how they affected the results (using an error analysis, as needed)
- Suggests how the procedure could be changed to minimize measurement errors, or to improve yields
- Puts the results in context

### Safety and Lab Practices Grade

To receive a 5 on Lab Practice a student must

- Have no accidents in lab
- Have no broken glassware
- Follow good laboratory practices at all times
- Clean up their work area and the group work areas
- Work in an efficient and safe manner

## **Progress Report**

A progress report is essentially a laboratory report that covers all research activity during a certain period. For this class you are to follow the format given in the Writing Laboratory Reports section of the Virtual Laboratory Manual, with the following changes (grouped by section).

1) Title/Title page (no change)

2) Abstract (omit)

3) Introduction (There does not need to be an extensive introduction for each exercise; a paragraph or so is sufficient. A brief accounting of how your virtual money was spent should be the last section of the introduction.)

4) Experimental (no change)

5) Results (No change, but if you have crystallographic results you will need to see me or Dr. Baughman regarding how it is to be tabulated in the results section.)

6) Discussion (no change)

7) Conclusions (No change for the science portion, but include any request for more virtual funds, along with a brief justification of the request.)

8) Acknowledgements (optional)

9) References (The full title of a research or review article does not need to be included in a reference.)

10) Tables (no change)

11) Schemes (no change)

12) Figure Legends (no change)

13) Figures (no change)

14) Supporting Information (Most of the spectral data will need to be shown as figures in the text, and so won't be supporting information. You may want to include cyclic voltammograms here, if the  $E^0$  data are tabulated in the main body of the text. Also to be included in the supporting information is bulk of the X-ray crystallography data tables. See Dr. Baughman about what needs to be done.)