

INSTRUMENTAL ANALYSIS LABORATORY 1008-318

Please see the web page (<http://www.cis.rit.edu/class/scha318/>) for a list of instructors and their contact information.

Introduction

Welcome to 1008-318, Instrumental Analysis. In this laboratory you will do five experiments, each using a different instrument available in the department. As the name of the course suggests, you will be analyzing different samples using instrumental methods. The emphasis is on understanding the strengths, weaknesses, similarities, differences, and appropriate use of the instrumental methods, and in accordance with that, the wet chemical preparation has been minimized. Of course, the necessary wet chemistry is extremely important and must be done carefully. The instruments you will use include research instruments and other department *workhorses*. In many cases we have only one suitable instrument in the department. The instruments are located in room 08-A207 and 08-A155.

<u>Experiment</u>	<u>Instrument*</u>
Determination of Mg by Atomic Absorption	SpectrAA-10
Fluorescence Spectroscopy of Quinine	LS50B Fluorescence Spectrometer
UV Recording Spectroscopy	Shimadzu UV-Vis Spectrometer
Composition of Complexes in Solution	HP Diode Array Spectrometer
NMR Spectroscopy	Bruker DRX-300 NMR

*Subject to change due to the purchase of new instruments.

You are expected to do all your laboratory work during your regular lab period. All this can add up to a real challenge, but with planning and good preparation everything should go smoothly. **This means read the protocol before coming to lab.** Also the experiments work quite well and are a good demonstration of the theory discussed in class.

Scheduling

Once you have chosen a lab partner, please select an experiment sequence and inform your instructor. This schedule coordinates use of the instruments and allots the right amount of instrument time for each experiment. You will probably have to perform one or more experiments before the appropriate theory has been discussed in class. In this case, you are expected to seek out and digest applicable theory and techniques. Lab reports are due one week after the assigned completion date of each experiment, which are scheduled for the following dates:

- Experiment 1. Week 1 & 2
- Experiment 2. Weeks 3 & 4
- Experiment 3. Weeks 5 & 6
- Experiment 4. Weeks 7 & 8
- Experiment 5. Weeks 9 & 10

It is very important that you keep up with the submission of your lab reports as the end of the quarter comes very quickly. For each week that lab reports are late, the maximum possible grade for the lab will be reduced by 5 points. For example, if you pass a lab in two weeks late, your maximum grade would be a 10. If you earn a 9, you would get a 9, but if you earn 11 or higher, the grade would still be only a 10. The fifth lab report must be turned in early enough for your instructor to read it and post your grade. Please inquire as to this date for your specific lab section.

Grading

The course grade is based on 5 lab reports, each 20% of the total, with the following breakdown.

- Abstract 2
- Introduction and Theory 3
- Experiment and Instrument 2
- Data Analysis 3
- Graphs and tables 2
- Discussion 4
- References 2
- Style, Grammar, Format, and discretionary. 2

Lab Reports and Notebooks

Experiments are to be performed by groups of two students but lab reports and unknowns are to be done on an individual basis. Each of you should record information and data taken during the experiment in your own lab notebook. Even though experiments utilize lab partners, all reports are to reflect an individual effort. Lab reports should be prepared using a word processor using 8.5" x 11" paper and pages should be numbered. The format should be similar to a journal article (see *Analytical Chemistry*, *JACS*, etc.) with some differences in emphasis due to the nature of the course. I would like to see more instrumental details than one normally finds in journal articles. The general format includes:

A **Title Section** for your report that should include the title of the experiment, your name, your partner's name and the date(s) on which the experimental data were obtained, and the date the lab report is due.

A brief **Abstract** summarizing the work done and reporting major results, including numerical results and their precision, instrumental technique used, instrument used, and the results of the analysis of any unknown. This is not an introduction or a purpose. It should summarize the highlights of the experiment: *e.g.*, concentration or identity of unknowns, wavelengths for peak maxima, effect of varying experimental parameters on signals (heating, cooling, bubbling oxygen), and comparison of results obtained by two or more methods. The abstract is extremely important and should only be written after your results have been evaluated completely.

An **Introduction** or **Theory** section describing the basis for the experiment. In general, present the theory behind the measurements that you will perform. You will know if you have done a good job if you can easily explain each of your results in the discussion section. Keep your theory pertinent to the actual measurements taken, use your own words, and use references where appropriate.

The more detailed **Experimental and Instrumental** section need not include a rehash of the procedure described in the handout but should include specific details about how solutions were made up from the solutions available in the lab. The preparation of solutions should be presented in a table form. Also a block diagram of the instrument should be presented. Cite your source of this diagram correctly and a description of its components and how it works should be provided.

The **Results** section should include tables of data, graphs, and data analysis. A description of the data presented in tables, figures and calculations should be included to increase clarity of reading.

- a. Tables should be numbered consecutively and consist of a row and column format, with a title at the top of the table. Tables should be designed for ease and clarity of reading.
- b. Figures should be numbered consecutively with a title at the bottom of the figure. The title should not just be the axes labels on the figure. The X- and Y-axes should be labeled, including units. All lines should be determined by linear regression. Data points should be made with distinct symbols.
- c. Data analysis should include the determination of the concentration of any unknowns and the equations for any linear regression curves that are obtained. Your data analysis must include a propagation of errors error analysis.
- d. Any spectra obtained directly from the instrument should be attached at the end of the report and referred to in your results section. On the spectrum, mark all pertinent information: sample identity and concentration if known, wavelength (for fluorescence and UV-Visible), absorbance (for UV-Visible), etc.

A **Discussion** section which should contain the following:

- a. any observations you made during the experiment,
- b. a discussion relating your results to the theory,
- c. a brief summary of any sources of error associated with your results, and
- d. answers to numbered questions in the handout.
- e. propagation of errors error analysis

Please feel free to include comments and suggestions that might improve the labs.

References should be in the style of the current chemical literature. Each reference is numbered according to when it is first used and thereafter is referred to by that number. The references are listed in order at the end of the report. For these labs, if you only have one or two references, you are doing something wrong. There should not be a separate bibliography section.

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Name: _____

Graded by: _____

Lab: CC UV AA Fluor NMR

Grade: _____

Abstract: _____/2

-Instrument used

-Instrumental technique

-System studied

-Major results

Introduction or Theory: _____/3

-Basic theory

-Theory related to each measurement

Experimental and Instrument: _____/2

-Diagram of instrument

Results: _____/5

-Data Analysis

-Graphs and Tables

-Propagation of errors error analysis

Discussion: _____/4

-Relate each result to theory

References: _____/2

Style, grammar, and format: _____/2

Discretionary:

Late

-5 / week

Failure to clean work area

-1

Failure to come to lab prepared

-2

Absent from lab without informing partners

-2