Chemistry 302: INSTRUMENTAL ANALYSIS

Dr. Brettell, Spring 2009
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Lab Sections 01, 02, 71

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## INSTRUMENTAL ANALYSIS LABORATORY

In the Instrumental Analysis laboratory, students will be working individually in groups of three (3) or four (4) to complete the twelve (12) experiments. Each individual experiment will be described in the laboratory manual or handout provided by the instructor. Each student should read and understand the material described in the laboratory manual and handouts before performing the experiments. The laboratory manual will also contain a detailed list of the requirements for the laboratory reports.

**WORKING IN GROUPS:** Each group of students will cooperate to perform an experiment together. One laboratory report is to be submitted by each group of students for each of the twelve (12) experiments.

Before beginning an experiment, the group should select a group leader. <u>Each member of the group should serve as leader for at least three (3) of the twelve (12) group experiments</u>. The responsibilities of the group leader are as follows:

- ➤ Coordinate the work required to complete the experiment and prepare the report. Try to delegate tasks as equally as possible among the group members.
- ➤ Make sure that all of the group members have a complete set of the data obtained in the laboratory.
- Assemble the parts of the report.
- ➤ Make sure that the other group members have an opportunity to review and revise the entire report before it is submitted.
- Ensure that the report is submitted to the instructor by the due date. Reports are due the next laboratory class after the completion of the experiment, with the exception of the final experiment, which is due 48 hours after checkout day (the last lab period of the semester).

Note that it is NOT the responsibility of the group leader to write the entire report.

The writing of the report should be a group effort. In general, all members of a group will receive the same grade for an experiment. Therefore **EACH** group member is responsible for insuring that her work is reflected in the report. In the event that there is disagreement among the group members concerning the content of any part of the report, any member is free to submit her own version of the topic in question.

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## LAB SCHEDULE

Attendance in the laboratory is **MANDATORY**. All twelve (12) experiments must be completed as scheduled and reports submitted when due. The laboratory will operate on a semi-open lab policy. That is, lab work is possible at times other than and including the officially scheduled lab period. As you all have had two plus years of training in Chemistry, it is expected that you can work safely with a minimum of supervision. An instructor must be present at all times.

There will be twelve (12) laboratory experiments. Students will work in groups of three (3) or four (4) on all experiments. For each experiment, each group will submit **one** laboratory report along with answers to the post-lab questions **two** weeks after completing the lab. **Each individual student will submit her own laboratory notes with the report.** 

Laboratories will be performed on a rotating schedule with the class divided into three groups. During any laboratory period, each group of students will be working on a different experiment. Hence, the specific dates on which a given experiment will be performed will differ for each group of students. A detailed schedule is provided below:

Week #	Date	Exp#	Title of Lab Experiment	Student Schedule		
1	20-Jan		Check In; Laboratory Orientation; Review of	Grp I	Grp II	Grp III
			Laboratory Safety Policies and Procedures			
				Week of	Week of	Week of
				<u>1/27</u>	<u>2/3</u>	<u>2/10</u>
2	27-Jan	1	Determination of Balance Error	Grp I	Grp III	Grp II
3	3-Feb	2	UV-Visible Spectrophotometry, pKa of an Indicator	Grp II	Grp I	Grp III
4	10-Feb	3	1-H NMR Determination of a Drug Mixture	Grp III	Grp II	Grp I
				Week of	Week of	Week of
				<u>2/24</u>	<u>3/3</u>	<u>3/17</u>
5	17-Feb		No Scheduled Lab- Catch-up			
6	24-Feb	4	Fluorometry, Analysis of Quinine in Tonic Water	Grp I	Grp III	Grp II
7	3-Mar	5	Infrared Spectrophotometry	Grp II	Grp I	Grp III
	10-Mar		Spring Break			
8	17-Mar	6	Basic Gas Chromatography/Mass Spectrometry;	Grp III	Grp II	Grp I
			Analysis of an Unknown Drug of Abuse			
				Week of	Week of	Week of
				<u>3/24</u>	<u>3/31</u>	<u>4/7</u>
9	24-Mar	7	Atomic Absorption: Analysis of Gunshot Residue	Grp I	Grp III	Grp II
10	31-Mar	8	GC, Qualitative & Quantitative Analysis of Alcohols	Grp II	Grp I	Grp III
11	7-Apr	9	GC/MS of Fire Debris Accelerants	Grp III	Grp II	Grp I
				Week of	Week of	Week of
				<u>4/14</u>	<u>4/21</u>	<u>4/28</u>
12	14-Apr	10	HPLC: Qualitative Analysis of Goldenseal Alkaloids	.Grp I	Grp III	Grp II
13	21-Apr	11	Pyrolysis GC/MS of Paints	Grp II	Grp I	Grp III
14	28-Apr	12	LC/MS/MS of a Drug Mixture	Grp III	Grp II	Grp I
	5-May		Friday Lab Section Only		_	

It is unavoidable that the lecture and laboratory will not move at the same pace, nor cover subjects in the same order. It is therefore, imperative that you learn as much as you can about each experiment by reading BEFORE you come to the lab. Be sure to read the appropriate chapters in the lecture text and the lab description before performing an experiment.

# LABORATORY NOTEBOOKS

Suggested laboratory notebook: Laboratory Notebook, W.H. Freeman and Company.

A copy of your laboratory notes from your notebook must be attached to your laboratory report.

### LABORATORY REPORTS

The laboratory reports required for this course in Instrumental Analysis should be detailed accounts of your work in the laboratory, including an organized collection of your measurements, summary of your computed results, and answers to a set of questions to be completed at the end of each experiment.

Laboratory reports should be concise, well organized, and in correct English. They should be on 8-1/2 x 11 inch paper with reasonable margins. Typewritten reports are preferable. It is strongly recommended that you use a word processing program such as Wordperfect<sup>®</sup> or Word<sup>®</sup>. However, NEAT, legible handwritten reports (in blue or black ink only) are acceptable. The pages of the report should be bound together in some manner, such as stapled or in a report folder.

Reports are due the next laboratory class after the experimentation is completed, with the exception of the final experiment, which is due 48 hours after checkout day (the last lab period of the semester).

# **POST-LAB QUESTIONS**

A set of post-lab questions will be included in the handout for each experiment. These questions are designed to test and extend your understanding of the experiment. The answers to these questions should be organized by number, as in the experiment handout.

You may also include any observations or comments concerning your work. The instructor would particularly appreciate criticisms of the experiment in general or suggestions for improving the laboratory experience.

#### **GRADING**

The grade for the laboratory portion of the course will be based on performance with respect to two items: the actual experiments and reports, and an evaluation by the instructor. There will be twelve (12) laboratory experiments. Each report will be graded separately. The grades for individual experiments (12 total) will be averaged to produce a single grade applying to all of the experiments. This laboratory report grade will constitute 90% of the final laboratory grade.

The remaining 10% of the final laboratory grade will be assigned by the instructor based on the student's performance with respect to such attributes as:

- Compliance with laboratory safety rules
- Competence with respect to laboratory techniques
- Completeness and organization of laboratory notebook
- Organization and efficiency when performing experiments
- General attitude in the laboratory and the degree of cooperation and contribution when performing experiments with a partner.

In summary, the laboratory grade will be computed as follows:

(0.90)(Experiment Average) + Instructor Evaluation (0 - 10 %)

The laboratory grade will count as 25% of the Instrumental Analysis Course grade.