

# CHEM 231 - QUANTITATIVE ANALYSIS LABORATORY

## SUMMER QUARTER, 2009

Instructor: Ed Thorne  
Office: Disque Hall, Room 316  
Phone: (215) 895-1331  
Email: thorneej@drexel.edu  
Office Hours: One hour before class each week and/or by appointment

Writing Intensive Tutor: Ben Riblett  
Email: [@gmail.com](#) (Please put ATTN: Ben Riblett CHEM 231)

### COURSE OBJECTIVE

The objective of CHEM 231 Laboratory is two-fold. First and foremost, this is a laboratory course whose primary objective is to introduce classical "wet" methods of analytical chemistry, in addition to absorption spectrophotometry and Beer's Law. Second, the entire quarter will be devoted to the statistical treatment of experimental data obtained via gravimetric, volumetric, and instrumental methods of analysis. As a result, the accuracy and precision of the results obtained will be a large focus for this course.

In addition to the experimental aspect, the CHEM 231 Laboratory has also been designated as the Writing Intensive Course for the Drexel Chemistry curriculum. As such there will be a series of writing assignments throughout the term that are designed to introduce you to the various forms of technical writing.

### COURSE MEETINGS

Thursday Evenings 6:00 P.M. to 9:30 PM in Disque Hall, Room 409

### REQUIRED COURSE MATERIALS

- Laboratory Experiments for Quantitative Analysis by Edward J. Thorne.
- Bound Laboratory Notebook
- Safety Glasses/Goggles
- Access to Microsoft Excel

### ATTENDANCE/LAB ASSIGNMENT DUE DATES

There are **NO make-up labs**. If you cannot attend a scheduled lab, you must notify me in advance. If no such notification is given beforehand and no valid excuse is given afterwards, you will receive a zero for the lab. The instructor is the judge of a valid excuse. Please note that **proof** of the validity of an excuse may also be required.

Hard copies of lab reports and electronic data (which will be submitted via email) are due no later than one week after the experiment has been completed, according to the schedule in this syllabus. Late reports will receive a full letter grade reduction per week if no valid excuse can be demonstrated. The instructor is the judge of a valid excuse.

## COURSE GRADING SCHEME

### QUIZZES (5 %)

There will be random pre-lab quizzes.

### SUBJECTIVE GRADE (5 %)

This grade is based on promptness (arrives on time, finishes on time), preparation for lab, level of organization, ability to work independently, cleanliness of work area, analytical technique observed, and overall attitude.

### LABORATORY NOTEBOOK (20 %)

The student will write the following sections in his/her lab notebook **prior** to coming to the lab:

- a) Experimental Purpose – 1 sentence
- b) Introduction/Theory – 2 pages minimum
- c) Answers to Pre-Lab Questions – must make a legitimate attempt
- d) Experimental Plan: a bulleted list that must be written in the student's own words and not merely copied from the lab book
- e) Data Tables

**The instructor will check the pre-lab requirements (a-e) before the experiment is started.**

- f) Experimental Observations
- g) Data – all data will be written in the lab notebook, not in the lab manual. Sections (e) and (f) will be written in the notebook **during** the actual experiment.

### LABORATORY REPORTS (20 %)

One week after the experiment has been completed; the student will submit a summary lab report consisting of (in the designated order):

- a) Title Page
- b) Data Tables – including the raw data (data collected when you are in lab) and the statistical treatment of the data (Q-test, mean, standard deviation, etc.). Because of the relatively few number of data points, and to clearly demonstrate your understanding of the concepts, students are **not** permitted to use the Excel functions for standard deviation and 95% confidence interval.
- c) Discussion: 1 full-page double-spaced font size 10 of discussion is the minimum required amount and should include possible sources of error.
- d) Conclusions: three sentences maximum
- e) Answers to the Post-Lab Questions

The student will **bring a printed copy** of the lab report and data tables to the lab to be submitted to the instructor. The student must also **submit an electronic copy of the Data Tables with associated calculations via email** using Microsoft Excel.

### QUANTITATIVE UNKNOWNNS (25 %)

A grade will be assigned based on the accuracy and precision of the results obtained. For quantitative results, the following grading scale based on percent relative error (% RE) will be employed.

GRADE	WET CHEMICAL EXPERIMENT	INSTRUMENT ORIENTED EXPERIMENT
A+ = 100	0.00-0.10 %	≤ 0.5 %
A = 95	0.10-0.20 %	0.5-1.0 %
A- = 91	0.20-0.30 %	1.0-2.0 %
B+ = 88	0.30-0.40 %	2.0-3.0 %
B = 85	0.40-0.55 %	3.0-4.5 %
B- = 81	0.55-0.75 %	4.5-6.0 %
C+ = 78	0.75-1.00 %	6.0-7.5 %
C = 75	1.00-1.25 %	7.5-9.0 %
C- = 71	1.25-1.50 %	9.0-12.0 %
D = 65	1.50-2.0 %	12.0-15.0 %
F = 0-60	> 2.0 %	> 15.0 %

### WRITING INTENSIVE LAB REPORT (WILR) (15%)

The results obtained during the volumetric and gravimetric analysis of chloride (weeks 4 and 5) will be combined and written-up as a detailed lab report, 15-20 pages in length. The student will work closely with the Writing Intensive Tutor for this course throughout the term to write 2 drafts prior to the final submission. The tutor for this quarter is Ben Riblett ( [@gmail.com](mailto:benriblett@gmail.com)). In the subject line, please put "ATTN: CHEM 231: Ben Riblett."

Each student is **required** to meet individually with the tutor at least twice during the term for 25-30 minute sessions. Each meeting missed will result in a letter grade deduction from the overall grade received on this assignment.

### FINAL EXAM (10%)

There will be a cumulative final exam covering fundamental topics presented in the laboratory experiments.

**SEQUENCE OF EXPERIMENTS FOR CHEM 231  
SUMMER, 2009**

WEEK	DATE	EXPERIMENT	EXPERIMENT TITLE	REPORT STYLE	DUE DATE
1	6/25/09		Check in and discussion of safe laboratory practices		
2	7/2/09	1	Volumetric Glassware & Data Treatment	Lab Report	7/9/09
3	7/9/09	2	Volumetric Determination of Water Hardness (via EDTA titration)	Lab Report	7/16/09
4	7/16/09	3	Gravimetric Determination of Cl	WILR	7/27/09* 8/27/09‡
5	7/23/09	4	Volumetric Determination of Chloride by Fajans' Method	WILR	7/30/09* 8/27/09‡
6	7/30/09		<b>Individual Meetings to Review First Draft of WILR</b>		
7	8/6/09	5	Acid-Base Titration and Purity of Potassium Hydrogen Phthalate (KHP)	Lab Report	8/13/09
8	8/13/09	6	Volumetric Determination of Iron <b>Second Draft of WILR</b>	Lab Report	8/20/09
9	8/20/09	7	Spectrophotometric Analysis: Determination of Mn in Steel (Spectronic 20 Method)	Lab Report	8/27/09
10	8/27/09		Check Out <b>Final Version of WILR Due</b>		

\* Electronic Data (Excel Spreadsheet) Due Only

‡ Final Version of Writing Intensive Lab Report Due

**GUIDELINES AND REQUIREMENTS FOR WRITING INTENSIVE LABORATORY REPORTS**

## **A. GENERAL INFORMATION**

1. The Writing Intensive Lab Report (WILR) is a requirement of this course to demonstrate your ability to clearly and effectively communicate scientific principles and laboratory data. As such, it is an extensive treatment of a specific unknown you are required to analyze. For this course, the WILR will be required for the chloride unknown sample you will analyze by both the gravimetric and volumetric methods. In essence, you should think of this report as being a document that thoroughly describes what is being done, the theoretical principles behind the experiment(s), a presentation of the data, sample calculations to demonstrate how the acquired data is converted into meaningful values, and the sound conclusions that can be derived from the exercise. The final reports should be approximately 15 to 20 pages in length for a document printed in 9-point Geneva or Verdana font size and double-spaced with one-inch margins on all sides. That is the format of this handout you are reading.
2. Any material taken from a textbook or handbook is to be properly referenced in the approved manner. References in the main body of the report will be made using superscripted numbers in parentheses, with the main bibliographic information listed at the very end of the report. All references **must** include each of the following: (a) title of the book or journal, (b) the name of the author, (c) the pages, (d) the volume number if appropriate and year of publication, and (e) the publisher. When in doubt, consult an English handbook or the instructions to authors printed in the front of scientific journals such as *Analytical Chemistry*.
3. All reports are to be typewritten. The only exception to this requirement is the incorporation of mathematical equations that may be overly tedious to format properly. In that case, space should be left in the main body of the report and the equations handwritten in their appropriate location. Use one side of the page only and indicate each section as described below. If the report is not "sectioned" it will be returned for rewriting.
4. Each report will have a cover sheet when it is submitted. The cover sheet may be obtained from the instructor when the final report is submitted.
5. Any reports that do not follow the prescribed format will be returned to the student for rewriting.

Rewriting a report applies only to format errors (improperly sectioned reports, improperly constructed graphs, etc.) and **NOT** for incomplete content such as weak conclusions, incorrect calculations, etc.

## **B. FORMAT OF WILR**

Writing a lab report is just as much a part of experimental chemistry as the actual laboratory manipulations. Great inventions, discoveries, new synthetic techniques, etc. are useless unless they can be accurately and clearly related to the rest of the scientific community. Every scientific journal in press at the present time has its own format requirements for manuscripts and authors must adhere to the prescribed format as a requirement for publication. Similarly, there is a format that must be adhered to for the WILR. Outlined below are the expectations/requirements for the WILR. While they may at first seem lengthy and tedious, many of the manipulations become second nature once they have been done a few times. While this is not a technical writing course as such, at this stage of your academic career you are expected to be able to write in a clear and concise fashion, following accepted rules of grammar. The reports will not be graded on a basis of penalizing strictly for grammatical errors such as punctuation, etc., but points will be deducted for flagrant violations of writing practices. In the past, students have submitted reports with incomplete sentences and other errors that are more a result of carelessness and/or laziness than grammatical errors. Most important of all, proofread your reports before they are submitted. Reports submitted with incomplete sentences and other serious grammatical errors will be penalized heavily, frequently 50% or more of the point value for the section of the report where they are found. The WILR must have the sections listed below, **each written as its own section**. Combining sections will result in point reduction penalties and/or returning the report for rewriting.

1. **ABSTRACT** - This section is very brief and typically can be written in 40 to 60 words. It gives a broad overview of the primary objective of the experiment and the result(s) that were generated. The final result(s) obtained must be given, along with the confidence limits and/or results of the propagation of errors calculations if appropriate.
2. **INTRODUCTION** - Within this section should be a statement of the basic theoretical principles

within the experiment and **all equations** being used in the treatment of the experimental data. The only exceptions to this rule are simple and/or standard calculations such as converting grams to moles or determination of molar mass from the atomic mass of the elements making up a compound. The equations are to be incorporated within your discussion and not merely a “laundry list” of mathematical expression that will be used. If in doubt as to what this means, refer to the Introduction section for the appropriate experiment in your lab manual. Accompanying this statement must be a definition of each parameter that appears in the equation, along with any restrictions on its units. A discussion of the relative merits of gravimetric vs. volumetric procedures is appropriate. Also indicate the requirements and limitations of each method. Do not assume that the reader is very familiar with either mode of analysis. In essence, treat your Introduction as the information you would need to know to be able to perform the experiment (without the detailed experimental procedure steps). In addition, you must include a brief qualitative description of the apparatus/method, clearly indicating any limitations. It is not necessary to include the detailed procedure for the actual experiment unless you have deviated from the lab manual directions. This section is not to be recopied or slightly reworded from the lab manual. Any report where this has been done will receive a grade of zero for this section.

3. **DATA** - The data collected in the laboratory must be recopied in a neat fashion, in table format if appropriate. Since you will have already submitted this information in an Excel spreadsheet, printing that and attaching it as part of the report is appropriate.
4. **SAMPLE CALCULATIONS AND RESULTS** - If there are several calculations of the same type to be made, all of which require the same equation, show in detail one calculation of each type, including all numerical values and units where appropriate, and then perform all of the remaining calculations using MICROSOFT EXCEL. You may find it more convenient to handwrite the sample calculations because of the formatting of some of the mathematical equations used. In this section, that is acceptable. All remaining calculations in the lab report must be done using a computer. The number of significant figures in the final reported values should reflect the actual number of figures in the measurements made but the intermediate values as determined in the individual calculations need not adhere to this rule. The computer output of the calculations is to

be included in this section. When in doubt as to exactly what belongs in this section, the simplest means of determining this is to include each item requested in the lab manual under the heading TREATMENT OF THE DATA.

6. **CONCLUSIONS AND DISCUSSIONS** - Comment on the success/failure of the experiment in light of its goals and your experimental results. Conclusions not based on the experimental results are not valid. For example, in a previous report on determining the pK of an indicator, a student concluded "*as the pH of a solution increased, its hydrogen ion concentration decreased*". While this statement is technically correct, it is a statement of a defined scientific fact and is not in any way based on the experiment that was performed and as such is not acceptable. Attempt to explain why the precision may have been poor (if yours was). Also, since you have studied the SAME UNKNOWN by two different methods, a detailed discussion of the relative results is in order. How do their means compare? What about their confidence intervals, etc.? Does one method seem "better" than the other? On what do you base that statement?
  
7. **ANSWERS TO QUESTIONS** - At the end of each experimental write-up in the lab manual there are questions regarding the experiment under the heading. Answer these as completely as possible in light of the work you have performed.

### **C. USE OF PERSONAL COMPUTERS**

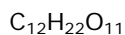
All repetitive calculations must be performed using a computer. It must be understood that the computer is a tool that will help you to perform your work: it does not dictate the format. The requirements listed for these operations are not unusual and any reasonably well-equipped software package will handle them quite nicely. For example, MICROSOFT EXCEL will enable you to complete the assignments in this course according to the above requirements. Similarly, when typing the report, the format used must be standard format that is accepted by the scientific community. For example, when using a spreadsheet such as MICROSOFT EXCEL, very large or very small numbers will be expressed in scientific notation as a part of the software limitations. These numbers will not be expressed in the standard notation such as  $6.02 \times 10^{23}$ , but rather it will be expressed as 6.02 E+23.

However, when you type your final report, this notation will be accepted only in the spreadsheet output. When typing very large or very small numbers (as well as variables which are subscripted or superscripted in text), only the standard format will be accepted. MICROSOFT WORD is equipped to handle these requirements quite well with the **SUBSCRIPT** AND **SUPERSCRIPT** selections. Listed below are examples of acceptable and unacceptable formats. If necessary, it is better to write in the correct format by hand than to type the incorrect format. If these formats are not adhered to, significant point reduction penalties will be imposed.

**CORRECT FORMAT**

$$P_1V_1 = P_2V_2$$

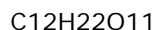
$$6.02 \times 10^{23}$$



**UNACCEPTABLE**

$$P1V1 = P2V2$$

$$6.02 \times 10^{23} \text{ or } 6.02 \text{ E}+23$$



**D. DEADLINES**

To prevent getting too far behind on this assignment, two drafts **MUST** be submitted during the quarter. These do not need to be complete but they must indicate that an appropriate level of progress is being made on completing the assignment. For example, the first draft can be mostly the Introduction and theoretical principles about both methods but include calculations related to only the gravimetric part and then present the calculations on the volumetric part in the second draft. The due dates for the drafts are as follows:

First Draft (minimum 5 pages in length)	Thursday July 30
Second Draft (minimum 10 pages in length)	Thursday August 13
Final Report Submission	Thursday August 27

NOTE: FAILURE TO SUBMIT THE NECESSARY DRAFTS (OR AN INADEQUATE DEMONSTRATION OF SUITABLE PROGRESS) WILL RESULT IN A 30 POINT REDUCTION FOR EACH DRAFT.