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**CHE 340L-012  
ANALYTICAL CHEMISTRY LABORATORY  
FALL 2007**

**Learning Outcomes:** This course's student learning outcomes fulfill the following programmatic goals of the Department of Chemistry.

*At the end of this course students will be able to:*

- *Demonstrate the ability to carry out experiment protocols using modern instrumentation and methods in analytical chemistry*
- *Apply computer related technologies (i.e. various graphing programs) to interpret analytical data*
- *Explain the limitations of various analytical measurements and methods of analysis*
- *Utilize analytical techniques to collect accurate and precise data*
- *Evaluate the reliability of data through error analysis*

**Lab Manual:** All experiments will be made available on Blackboard for you to print out prior to class.

**Lab Notebook:**

A bound notebook of approximately 10" X 7 1/2" (i.e. black marble "Composition Tablet") is required for submitting weekly reports. A notebook with a spiral binding will not be permitted.

**Attendance:** Access to the lab (SC 427) will be limited to the scheduled 4 hours and 50 minutes (Thursday 1:00-5:50 pm) per week. No one will be allowed to work outside these hours.

**Experiments:** Over the course of the semester you will conduct several different laboratory experiments. Your grade will depend upon the precision and accuracy of your results along with a report write-up within your lab notebook (including observations, discussion, grammar, spelling, use of units, and significant figures). **The precision/accuracy of your results will be a large portion of your grade for an experiment.**

**Formal Lab Reports:** In addition to the lab write-ups in your notebook, you will also turn in **1** formal lab report during the semester and **1** industrial report.

**Grading Scheme:**

Lab Write Ups (Notebook) = 75%  
Formal/Industrial Lab Write Reports = 25%

**Grading Scale:** The letter grade for the course will be based on the final percentage scale

<u>Course Grade</u>	<u>Percentage</u>
A	90-100
B	80-89
C	70-79
D	60-69
F	0-59

**CHE 340L-011(Tuesday Section)  
Analytical Chemistry Lab**

**Tentative Lab Schedule Fall 2007**

<u>Exp #</u>	<u>Title of Exp. and Page No.</u>	<u>Date</u>	<u>Results (Notebook) Due Dates (Monday)*</u>
1	Check in and Calibration of Pipet	8/23	8/27
2a	Soda Ash Detmn. (Indicator Method)	8/30	9/17
2b	Soda Ash Detmn. (Potentiometric Method)	9/6	9/17
3	Cation Exchange Detm. of K <sup>+</sup> and H <sup>+</sup> ions in solution	9/13	9/24
4	Determination of Ca and Mg in Water	9/20	10/1 ‡
5	Determination of Iron in Iron ore	9/27	10/8
6	Potentiometric Detmn. of Iron in FAS	10/4	10/17
7	Microscale Determn. of Ascorbic Acid in Vitamin C Tablets	10/11	10/22
8**	Ion Selective Electrodes/ Electrodeposition of Cu	10/18	10/2
9**	Ion Selective Electrodes/ Electrodeposition of Cu	10/25	11/5
10**	HPLC / AA / Spect. Detm. Mn	11/1	11/12 †
11**	HPLC / AA / Spect. Detm. Mn	11/8	11/19
12**	HPLC / AA / Spect. Detm. Mn	11/15	11/30
	Open lab (make-up day)	11/29	12/10
	Checkout	12/6	

\* Lab notebooks (reports) are due in my office, SC 443, by **1:00 pm** on the due dates shown above.

**Note: Except for 10/04 (experiment 6) all due dates are on Mondays.** Late reports will not be accepted.

\*\* For these experiments involving specialized instrumentation, the class will be divided into groups of 2/3 students each. Each group will perform only one of these experiments per week. The report for that experiment will be due on the due date shown above.

† Formal Report for this experiment (Spect. Detm. Mn)

‡ Industrial Report (Detm. Ca and Mg Water)

*Make up day: If you receive a low grade on an experiment, you may elect to repeat the determination on a different sample. This may be obtained from the instructor. The final grade recorded for the determination will be the grade earned on the repeat analysis minus 10 points. The repeat determination has to be carried out during regular lab hours. No more than one experiment may be repeated.*

# Policies and Procedures for Analytical Chemistry Laboratory

## I. LAB NOTEBOOK

Each student must turn in their own laboratory notebook. Your notebook should reflect your ability to document what you do in lab. A notebook is not always the “prettiest” document, but one should be able to repeat an experiment by following your notebook. You should always write in your notebook in PEN. Original graphs, computer printouts, and chromatograms or photocopies must be turned in with the lab report. When graphs, computer printouts, and chromatograms are included in the lab notebook, they must be folded and stapled into the notebook.

### Format for Laboratory Reports- Notebook (You will keep this format for all Experiments)

1. The lab notebook must contain a Table of Contents with experiment names and the page numbers on which experiments start and have pages numbered. Below is a brief example of a Table of Contents.

<u>Table of Contents</u>		
<b>Experiment Number</b>	<b>Title of Experiment</b>	<b>Page Number</b>
1	Calibration of a Pipet	1
2	The Spectrophotometric Determination of Manganese in Steel	5

2. TITLE SECTION - This section must contain the following at the top of the page: a) Experiment title b) Experiment number c) Date d) Sample number
3. OBJECTIVE - This section must contain an explanation of the objective of the experiment. This must be written in *present* tense.
4. PRINCIPLE - This section must contain an explanation of the principles upon which the experiment is based and provides quantitative results. Within or at the end of this section, provide any and all chemical reactions that occur or which are used in the experiment with an explanation of the purpose of each reaction. This must be written in *present* tense.
5. PROCEDURE - This section must contain a brief overview of the procedure used in an experiment. Make sure you describe all major facets of the procedure. It is not necessary to describe the intricate details of the procedure. **DO NOT** copy the procedure from the laboratory manual. Also, indicate any changes made from the laboratory manual procedure. The procedure must be written in past tense and third person.
6. OBSERVATIONS (Data) - This section must include all pertinent observations made in the laboratory for an experiment. Include, in a tabular format, all data collected during the experiment. For example, for the Determination of Soda Ash experiment, the following format could be used:

Standardization of NaOH:	<u>Trial 1</u>	<u>Trial 2</u>	<u>Trial 3</u>
Mass of weighing bottle + pure KHP (g)	17.1352	16.6911	16.1301
Mass of weighing bottle (g)	16.6901	16.2651	15.6805
Mass of pure KHP (g)	0.4451	0.4260	0.4496
Final buret reading (mL)	40.85	38.66	42.24
Initial buret reading (mL)	18.35	17.04	19.48
Volume of titrant (mL)	22.50	21.62	22.76

7. CALCULATIONS - This section **must** contain **sample** calculations based on actual data from the experiment. If there are multiple calculations that are identical, only one calculation needs to be provided, but provide all of the results for identical calculations in this section even if the calculation is provided for only one result. Typical calculations for an experiment include the following:
- Determination of stock or primary standard solution concentrations.
  - Determination of secondary standard solution concentrations.
  - Determination of concentration(s), mass(es), or weight percentages in a sample **(This must be performed for all sample components)**.
  - Calculations of standard deviation and 95 % confidence limits for the analysis of 3 or more independent samples (This is not necessary if one sample is analyzed multiple times with an instrument such as the atomic absorption spectrophotometer).
8. RESULTS - This section must contain the results for an experiment in a tabular format. Also, report the average value of results from different trials. Results must be in the correct number of significant figures (four in most cases)
9. CONCLUSION - Explain the sources of error which could have led to systematic error in the experiment. Include a brief discussion of the significance of the data and observations.

## **II. HELPFUL TIPS FOR ALL LAB REPORTS**

Write in third person:

Good: *The solution was heated.*

Not so good: *We heated the solution.*

Have agreement in tense throughout the report:

Good: *The injection was repeated three times. For each analysis a new background was collected.*

Not so good: *The injection is repeated three times. For each analysis a new background was collected.*

Have agreement between subject and verb:

Good: *The spectra were analyzed.*

Not so Good: *The spectra was analyzed.*

Word Choices:

"came out" = eluted

"setup"=configured

"analyzed" = completed or performed

Don't start sentence with: prepositions, abbreviations, or numbers

Figures and tables should have captions with them.

Figure 1. Signal-to-noise ratio as a function of spectral resolution.

### III. INDUSTRIAL REPORT

Industrial laboratory reports are written from a different perspective than a typical formal laboratory report, which simulates the style of a peer-reviewed scientific journal where new scientific findings are shared with the greater scientific community. Industrial reports are written instead from the perspective of creating an, easy to read, document conveying results for management and customers primarily, and containing supporting material and the documentation requirements to comply with quality assurance needs after results.

**Title:** Use a specific, professional title indicating both the specific analyte and the method/instrumentation by which it was measured.

**Results:** Only the essential data should be shown here. The gory detail is in your datasheets and notebook. Immediately give the relevant results of the analysis in a highly concise form. This is not the place for explanations, justifications, or discussion.

**Procedure:** A brief exposition of what was done. Established procedures may be referenced without being explained, but any modifications should be briefly mentioned and justified.

Procedure *Examples:* EPA method 234.2 (revision 1.4.2) was used unmodified.

Method 1 of Analytical Chemistry 340 Laboratory Manual, issued 2007, XX-XX, was used with the following modifications: Computer controlled Mark 500 pH interfaces were utilized instead of the Zahner 1.4 pH meters due to malfunction of the Zahner units.

**Supporting Material:** Include in this section any graphs or transient data items that need to be calculated in the procedure, such as: Extinction coefficients, calibration curves, internal standard response levels, standardized titrant concentrations, etc... (The figures should have headings etc.)

**Quality Assurance Information:** Include here information from the method that allows you to track the results of a series of measurements. Identify the specific instrumentation used and any specific calibrated glassware. Give the values of any data that tracks the functionality of the instruments or lab ware used.

Quality Assurance *Example:* Calibrated Buret "KW1" was used, along with Zahner pH meter serial number 1331-ZH-1. Buffers (pH 4.01 and 9.48) were used to calibrate the pH meter with a resulting slope of 57 mV.

**Signature Lines :** Include two signature lines in the following order:

\_\_\_\_\_  
Analyst \_\_\_\_\_  
Date

\_\_\_\_\_  
Supervisor - Dr. Bullen or Dr. Edwards \_\_\_\_\_  
Date

**Include a MEMO to Supervisor:** Along with the memo you would attach the industrial report. The memo is the opportunity to 'highlight' the report and provide a discussion of items to your supervisor. If appropriate, discuss or explain sources of error and propose remedies for such errors. If you can draw conclusions about the procedure or instrument.

## IV. FORMAL REPORT

The Formal report should be typed, contain clearly labeled sections, include sample calculations in the results section, integrate charts, graphs, tables of data within the report, and use proper grammar.

### It will contain:

1) Title Page: The title page should include [experiment title, name, dates of experimental work, course title, unknown sample name/number, and date submitted]

2) Abstract: This is not an introduction, but a brief (short paragraph) outline of what was accomplished and the results.

3) Introduction: Here you should provide the overall objective and background of the experiment. The theory of a particular technique and experiment should be described. You **must** write this description in your own words, and use appropriate references/figures when necessary.

4) Experimental: This is a description of the particular instrument and procedure used for an experiment. Explain how data was collected.

5) Results and Discussion: These two sections are often found in chemistry journals combined together. In this section a narration presenting the results obtained (ie. graphs, charts, tables) during the experiment and discussing what the data means (data interpretation) should be presented. You also would include comments on the analysis and the limitations and advantages of the technique.

6) Conclusion: Paragraph summarizing results and presenting a final conclusion.

7) Literature References: You must correctly list all references used throughout the report. The literature references should be presented in standard American Chemical Society (ACS) format.

**Note: You will turn in your notebook along with your formal and industrial reports.**

## V. ADDITIONAL GRADING POLICIES

### Grading and Rejection of a Result:

For each experiment only the reported average value of the results will be graded; however, you should include all individual values obtained, indicating which, if any, have been omitted in calculating the final "average" value. (The decision to reject a result rests entirely up to you but must be justified.)

### Recalculations:

If the instructor detects or suspects that a reported value is erroneous due to an error in calculations, they may return the notebook ungraded and marked "Recalculate." The subsequently reported (recalculated) result will be graded with a penalty for recalculation.

### Unknowns:

Your access to unknowns is **NOT** unlimited. If you require more unknown, this will result in a penalty in your grade for a particular experiment.

## **Policies of the Department of Chemistry at Northern Kentucky University**

- All items on syllabi are subject to change by the instructor.
- Students are responsible for reading and understanding all items on the syllabi. Any items not understood must be brought to the attention of the instructor within the first two weeks of class.
- The work you will do in any course is subject to the Student Honor Code. The Honor Code is a commitment to the highest degree of ethical integrity in academic conduct, a commitment that, individually and collectively, the students of Northern Kentucky University will not lie, cheat, or plagiarize to gain an academic advantage over fellow students or avoid academic requirements.
- Cheating will not be tolerated. In accordance with the Code of Student Rights and Responsibilities, faculty members have the right to determine actions to be taken when a student is caught cheating.
- Faculty members reserve the right to dismiss or to have removed a disruptive student from their classrooms.
- Students with disabilities who require accommodations (Academic adjustments, auxiliary aids or services) for this course must register with the Disability Services Office. Please contact the Disability Service Office immediately in the University Center, Suite 320 or call 859/572/6373 for more information. Verification of your disability is required in the Disability Services Office for you to receive reasonable academic accommodations.

### **Lab policies:**

- Students not adhering to safety policies for laboratories may be dismissed from lab.

## **Department of Chemistry Student Learning Outcomes**

1. Explain the major concepts and experimental findings in the chemical sciences.
2. Demonstrate the ability to carry out experimental protocols using modern instrumentation and methods.
3. Utilize critical thinking skills to apply concept knowledge and adapt experimental techniques to: a) form and test hypotheses and b) solve scientific problems.
4. Compile, critically evaluate, and interpret scientific information and data.
5. Effectively communicate scientific information through written and oral means.
6. Apply effective group strategies to solve scientific problems.
7. Evaluate the relationships between chemistry and mathematics, physics, biology, and other disciplines and between chemistry and society.
8. Apply computer technology and other technologies in the comprehension, interpretation, and presentation of the chemical sciences.