CHEM 325 Analytical Separations and Measurements Spring 2015

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Office Hours: MWF, 9:00-10:00, or by appt. Lecture: 8:00- 9:15 TuTh, Far. 205 Labs: LaT 301

TENTATIVE LECTURE SCHEDULE – Spring 2015

WEEK		TOPICS	CHAPTER/SECT.
1.	Jan. 13	Introduction/Terminology/Units and Calculations	0 - 2 (all)
	Jan. 15	Sig. Figs./Error/Uncertainty	3 (all)
2.	Jan. 20	Statistics/Gaussian Dist./Use of Statistics/Calibrations	4 (all)
	Jan. 22	Quality Assurance/Standard Addn./Internal Std./Sampling	5, 27 (all)
3.	Jan. 27	Equilibrium Applications/Solubility	6 (all)
	Jan. 29	**EXAM 1**	
4.	Feb. 3	Gravimetric Analysis/Combustion Analysis	26.1-26.4
	Feb. 5	Activity Coefficients/Ionic Strength	7.1-7.3
5.	Feb. 10	Factors Affecting Ionic Strength/Systematic Treatment of Equil.	7.4-7.5
	Feb. 12	Titrations/Spectrophotometric/Indicator Methods	1.5, 17.5, 26.8
6.	Feb. 17	Acid/Base Equil./Buffers/pH Titrations/Indicators	8, 9.3, 10
	Feb. 19	**EXAM 2**	
7.	Feb. 24	Complexometric Titrations/EDTA Titration Methods	11 (all)
	Feb. 26	Redox Titrations/Cell Conventions/End Point Detection	15.1-15.2
8.	Mar. 3	Redox Sample Prep/Iodimetry/Iodometry	15.3-15.7
	Mar. 5	Analytical Separations/Solvent Extraction/Counter Current Dist.	22.1, suppl.
9.	Mar. 10/12	**SPRING BREAK**	
10.	Mar. 17	Chromatographic Methods/Terminology/Retention/Resolution	22.2-22.3
	Mar. 19	Separation Efficiency/Band Spread/Van Deemter Eqn.	22.4-22.5
11.	Mar. 24	**EXAM 3**	
	Mar. 26	Gas Chromatography/Kovats Indices/Gradient Programs	23.1-23.2
12.	Mar. 31	GC Detectors/Sample Prep	23.3-23.5
	Apr. 2	Liquid Chromatography/Detectors	24.1-24.2
13.	Apr. 7	Optimization in LC/Intro to Mass Spectrometry/Spectra Interp.	24.3-24.4, 21.1-21.2
	Apr. 9	**EXAM 4**	
14.	Apr. 14	Mass Spectrometry Components/Methods	21.3-21.4
	Apr. 16	Misc. Methods/SFC/IEC	
15.	Apr. 21	IEC Detection/Ion Suppression/Direct vs Indirect Detection	25.2
	Apr. 23	Size Exclusion/SEC Thermodynamics/Calibration	25.3
	May 5	**Final Exam (Tuesday of finals week 8:00 – 9:50 am)**	

TEXTBOOK: Daniel C. Harris "*Quantitative Chemical Analysis*", 8th ed., Freeman, New York [2010].

Grading:			
	Exams (4 @ 100 pts each)	= 400*	(Note: the lowest hourly exam score
	Participation/Homework (Sapling)	= 150	will be dropped)
	Final Exam	= 150	
	Laboratory (see 3rd page)	= 250	
	totals:	850	

Final grades will be based on a the overall average of the lecture/lab points A **tentative** scale is:

88% = A 78% = B 67% = C 56% = D <56% = F

YOU MUST OBTAIN A PASSING GRADE IN LAB (60%, or 150/250 pts.) AND TAKE THE FINAL EXAM TO PASS THE COURSE. ALL LAB EXERCISES ARE MANDATORY!

Content / Learning Objectives:

- Understand the effect of uncertainty in measurement on the accuracy and precision of calculated results.
- Use statistical methods to determine and correctly report the uncertainty associated with measured results, and to evaluate/compare data sets.
- Understand the role of ionic strength on equilibria used in routine chemical analysis.
- Use chemical equilibria calculations and be able to identify appropriate end-point detection methods for a variety of volumetric analyses/titrations.
- Understand the role of thermodynamic equilibrium in chemical separations and chromatographic methods.
- Understand the operational principles of chromatographic detectors.
- Understand the operational principles of mass spectrometry, and be able to identify and discuss the role of common MS instrumental components.

Keys to Success:

- Read assigned material from text before lecture so you are at least somewhat familiar with the content.
- Come to class! It is not only an opportunity to see the material again, but it is an opportunity to ask questions. (Plus, part of your grade is based on attendance/participation).
- Keep up with the homework assignments. Not only is it part of your grade, it will help you get up to speed in mastering the material before the exams. Waiting until the last minute to start doing homework and/or preparing for exams is a recipe for failure.
- Ask questions! (The only "stupid question" is the one that doesn't get asked). Seek help as needed in a timely fashion.
- In a similar fashion, keep up to date on your lab exercises – if you get behind it is extremely difficult to catch up!

Rules of Conduct:

Under the Academic Regulations sections of the NIU catalog, it states:

"When a student's behavior in a classroom, laboratory, or other formal learning environment is such that the rights of other enrolled students to an effective learning climate are being violated, the student shall lose the privilege of attending or receiving credit in the class."

I would ask that all students respect the rights of their classmates, and conduct themselves according to the principles of common courtesy while in class/lab. In addition, any instances of academic misconduct, including cheating, inappropriate copying the work of another student, or plagiarism, will be subject to judicial review as discussed in the catalog section on Academic Integrity.

NIU abides by Section 504 of the Rehabilitation Act of 1973, which mandates that reasonable accommodations be provided for qualified students with disabilities. If you have a disability that requires accommodations, please contact me early in the semester so that I can help provide them. Before this, you must register with the Center for Access-Ability Resources (CAAR), the designated office on campus to provide services and administer exams with accommodations for students with disabilities. The CAAR office is located on the 4th floor of the University Health Services building (815-753-1303).

LABORATORY SCHEDULE

L001 (M: 2:00 - 5:50 pm) - ??? L002 (W 2:00 - 5:50 pm) – ???

WEEK OF:	EXPERIMENT	PROCEDURE* (# in text)	Points	DUE DATE (at noon)
Jan. 12	Check-in/Analytical Balance/ Spreadsheets	31-35, 43-48		
Jan. 26	Prep & Standardization of NaOH/ Purity of KHP	19-24, E1	18	Feb. 6
Feb. 2	Chloride by Mohr Method	E2	18	Feb. 13
Feb. 9	EDTA Titration of Ca/Mg	E3	23	Feb. 20
Feb. 16	Iodimetric Titration of Vit C	E4	18	Feb. 27
Feb. 23	Spectrophotometric Titration of Fe^{3+}	E5	13	Mar. 6
Mar. 2	Lab Exam/Makeups		35	
Mar. 16	Gas Chromatography I	E6	13	Mar. 27
Mar. 23	Gas Chromatography II	E7	18	Apr. 3
Mar. 30	HPLC I	E8	13	Apr. 10
Apr. 6	HPLC II	E9	13	Apr. 17
Apr. 13	LC/MS	E10	13	Apr. 24
Apr. 20	Literature Report/ Make-ups	LR	10	
Apr. 27	Lab Final/Check Out (mandatory)		35	
	General Laboratory Performance		10	
		TOTAL =	250	

*Those lab procedures identified with "E#" can be downloaded directly from the Blackboard website and will be available in the <Content/Labs> folder in advance of the lab dates.

Lab points: For all lab assignments, 2 points will be based on appropriate maintenance/documentation of the lab notebook (see attached Guidelines). The remaining points will be distributed as follows: accuracy/identification of unknown (33%), precision/quantification of unknown (33%), correct/appropriate and complete calculations (33%).

Lab Notebook: Students should purchase a 100-page notebook (carbonless copy); some are available for sale in the Chemistry Stockroom. Procedures for the experiment are to be summarized in the notebook **before** the scheduled lab period. All data are to be recorded directly in the notebook in ink! Try to maintain an organized and legible notebook (see Guidelines). Have the TA check your notebook for style and format within the first few lab meetings. The carbon/duplicate sheets will be handed in on a weekly basis for grading; points will be based on accuracy and precision, as well as on the notebook (organization/content/style). ***Note: lab reports are due on the dates indicated above. Points will be subtracted for late labs***

Safety: Goggles are to be worn in the laboratory at all times! In addition, the workbenches are to be kept clear of clutter (books, backpacks, etc.). Appropriate clothing is to be worn in the laboratory - no sandals or open-toed shoes, no shorts or sleeveless shirts. Long hair should be tied back. NO FOOD OR DRINKS IN THE LAB!

ATTENDANCE/COMPLETION OF ALL LAB EXERCISES IS MANDATORY!

Lab Content/Learning Objectives:

- Apply principles of "good lab practice" to appropriately maintain a laboratory notebook.
- Perform replicate titrations with accuracy and precision.
- Use titration data to determine concentration and/or percent purity of unknown samples.
- Apply statistical methods to evaluate data obtained in the laboratory.
- Use instrumental separation methods (GC/LC/LC-MS) to perform qualitative and quantitative analysis of mixtures.

LABORATORY NOTEBOOK GUIDELINES

Your lab notebook is intended to be a substantive record of work performed in the laboratory, in which you must record the data/results obtained from your experiments. It should contain sufficient information so that anyone reading your notebook would be able to reproduce your experiments, and evaluate your conclusions. In general, scientific notebooks follow a basic format similar to that provided below.

Suggested Format:

1. Table of Contents - Leave a few pages at the beginning of you notebook so that you can list the individual experiments, the dates on which they were performed, and the pages on which the relevant procedures and data may be found.

2. Experiments - **Each lab exercise should be dated and initialed**. In addition, the following items should be included:

   a) Title - (see Lab Syllabus) Should be listed both in the Table of Contents and on the first page of the experimental section.

   b) Objective - A brief (2 to 3 sentences) description of the purpose and goals of the analyses to be performed.

   c) Procedures - An itemized list of the sequential steps performed, including information relevant to the preparation of samples, standards, and reagent solutions, and the equipment utilized.

   d) Data - **All data obtained during the analysis should be permanently recorded, in ink, directly in the notebook! Do not write data on pieces of scrap paper!** The data should be clearly labeled (including appropriate units) so that it is obvious what the data represents, i.e. tare weight, weight of tare + sample, etc. If you anticipate accumulating a lot of data, use data tables for convenient data entry. Preparation of these tables ahead of time can save time during the lab. If you make a mistake in entering data, do not erase or obliterate the offending data. Rather, draw a single line through the erroneous value(s) and enter the correct value(s) either above or next to the mistake(s). Include graphs when appropriate.

   e) Calculations - Your treatment of the data, i.e. calculations and relevant equations, should be included. Your calculations should be clear enough so that it is obvious how the final result was achieved. Estimated error calculations and/or statistical analyses should be included in this section.

   f) Conclusions - Report your results in appropriate units and with the associated uncertainties. In addition, a brief discussion of the possible sources of error, potential interferences, etc. may be included.