INTRODUCTORY ANALYTICAL CHEMISTRY

CHEM 0250

S. G. WEBER, INSTRUCTOR

SYLLABUS

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OVERVIEW

One of the most important questions answered by chemists is, "What is the composition of this object, and what does its composition imply about function?" From the earliest times, the analysis of ores (gold!) was an important pursuit. Today, analytical chemistry answers an uncountable number of important questions. Some of these are quite prosaic, such as, "Is this bridge girder mostly rust or mostly iron?", while some are surprising, such as, "If I had an analysis of the composition of these archeologically important pieces of pottery, then I could establish where this civilization began, and something of its movements over time!", and some are quite dramatic, such as, "Am I pregnant?"

Chemical equilibria form the foundations of classical Analytical Chemistry. Chemical reactions that are fast and that tend to reach equilibrium were the basis of the earliest analytical chemistry. Precipitates from solutions to which various reagents were added were used to identify the metals in samples of things like minerals, fertilizers, pigments, alloys, etc. From a detailed understanding of equilibria grew the possibility to predict which reactions would or would not occur in a certain set of conditions. This understanding led to *selectivity*, "At pH 3-4 I can precipitate Fe^{3+}, but not Fe^{2+}. I have a selective method of precipitating Fe^{3+} in the presence of Fe^{2+}". This is one example of how understanding equilibria leads to clever ways to get answers about composition.

Chemical equilibria are very important in biology, pharmacology, and medicine, in the environment, in basic chemical research, in geology, really everywhere.

Instruments began to replace purely chemical reaction-based measurements in the early-mid 20th century. Pittsburgh was actually at the forefront of research and development of so-called emission spectrometers that could measure quantitatively the metal composition of alloys, minerals, etc.. We will discuss absorbance and (to a lesser degree) fluorescence spectrophotometry that are related. The single most important analytical method is chromatography, which happens to operate based on chemical equilibria. While developing equilibrium concepts in a way that follows the book and the laboratory part of the course (CHEM 0260), you will learn about some very useful and sophisticated chromatographic techniques.

ON LEARNING

1. Learning requires a commitment. I expect you to be committed to learning, to be curious, to be willing to express your questions in class, to participate.
2. Do you understand the material? How do you know? It is an accomplishment to be able to listen/read statements and then agree/disagree with the speaker/text. However, this recognition of accurate/inaccurate statements is not enough. You must be able to explain how and why. I recommend this self-test. When studying/reading/doing problems, at an appropriate point, close the book and explain to a study partner, your cat, the wall, the "what, how and why" of the phenomenon or approach to a problem. And speak out loud. If you don't understand the material, you will not be able to express the idea cogently. Go back and read more, do a few more problems, and try again.
3. READ THE MATERIAL BEFORE CLASS. If I fall to the floor unconscious, you should be ready to pick up the chalk and keep the lecture going. Maybe try a little CPR first, though.
4. Phones and laptops: I will appoint one person to keep her/his eye on texts for urgent messages from university security. Please, no social texting, messaging, tweeting, etc. during class. However, you might have a question about something that comes up in class. I invite you to use a laptop or smartphone to look something up related to class so that you can add to the discussion. I only ask that you signal your intention by a quick raising of your hand.

ON TEACHING

1. There will not be big surprises on exams. You will know what you need to know. You will know what information will be provided on each exam ahead of time, and you will be able to propose that I add more information.

2. You are resourceful – you would not be "here" if you were not. You can apply the basic and limited understanding from undergraduate textbooks to some challenging current scientific and measurement problems. It gets you a little outside your comfort zone (no textbook-level description, just a research article, for example) but the reward is significant.
3. Remember learning long division? You worked on it in class with the teacher strolling around the room helping. It is a little impractical in a class this size to do exactly this, but there will be times when it benefits you to DO what you are learning about.

LEARNING OBJECTIVES

Some of these objectives are redundant. We will see the same concept in different forms, helping you to generalize.

Understand measurement problems
- Simultaneous equilibria
- Types of quantitative measurements
- Statistical significance

Apply concepts of
- Statistics
- Chemical equilibria
- Potentiometry
- Spectrophotometry

Semiquantitative chemical reasoning
- Rapid assessment of chemical systems

Quantitative chemical reasoning
- Problem solving

Analytical chemistry
- Goals
- Concepts
  - detection limit
  - limit of quantitation
  - linear dynamic range
  - dynamic range
  - selectivity
  - sensitivity
- Unit operations

Applying understanding to research problems

SCHEDULE OF MEETINGS AND OTHER DETAILS

Week	Begins	Topic	Reading	Homework
35	27 Aug	Unit 1. Measurements and statistics probability distribution, statistical parameters	***See "Getting up to speed" on p. 7 of this document.*** Read Chapter 1 orient yourself, Ch 2 section 2-10 on excel; Ch 3 on errors, significant figures, and propagation of errors, 4 up to 4C, skip 4C, then do 4D-E	Exercises 4E, F, G Problems 4.2, 4.6 a,b, 4.12, 4.15 and 16 (the are paired)
36	**5 Sept**	Precision, confidence, significance, detection limit	Ch 5	Ch 5 Ex: A-D. Problems: 9, 14, 15, 16, 18, 19, 20, 27, 31
37	10 Sept	Regression		
38	17 Sept 19 Sept	Quiz on acid/base *Exam 1*	Ch 6 understanding is **assumed** (it's Gen Chem 2 material)	
39	24 Sept	Chromatography, partitioning, distribution diagrams. Simultaneous equilibria,	Ch 7 (titration) Ch 8: sections 8-4 and 8-5 Ch 9	Ch 7 15 Ch 8 Ex. A, F, G, I. Probs. 18, 19, 24 Ch 9 Ex I, J. Probs 28, 29, 30, 31 33, 39, 42
40	**1 Oct**	Buffers and acid/base titrations	Ch 10 Ch 11	Ch 10: Ex A, B, Da, E; Probs. 5, 13, 14, 16, 17, 19, 23, 24, 29, 30, 31ab, 32, 40 (isoelectric)
41	9 Oct	Use of protic equilibria in chromatography. Spider sex hormones.	Ch 23 Sections 23-1 – 23-3 Ch 24 Section 24-1	

42	15 Oct	UV vis spectroscopy and spectrophotometry Fluorescence and fluorescence microscopy		Ch 17 Ex A, B, D; Probs. 1, 2, 6, 8, 10
43	22 Oct	EDTA and competitive equilibria – CA^{2+} transients in neurons, immunoassays		Ch 11 Ex A, B; Probs 11.1, 2, 3, 4, 14, 15, 17, 18, 23, 32, 34
44	29 Oct 31 Oct	*Exam 2* Electrochemistry		Ch 13 is Gen Chem review – use as needed.
45	**5 Nov**	Potentiometry, sensors		Ch 14 Ex A, B, C, E; Problems 1a, b; 2, a, b; 3, 4, 6, 7, 8, 28, 30, 31, 32, 33
46	12 Nov	Other applications of electrochemistry and electrokinetics of ions – Ion exchange chromatography, Electrophoresis, conductance sensors (carbon nanotubes)		
47	19 Nov	*Exam 3*		
48	26 Nov	Mass spectrometry Ch 21		Ch 21: EX A, B, C, Da, E. Probs: 1, 2, 3, 4, 5, 6, 8, 9, 10, 12, 16, 20, 21
49	3 Dec	Chrom Ch 22.4-5, (Ch 25) Proteomics		Ch 22: Probs 28, 29, 31, 32, 33, 38
50	10 Dec	*6-8:00pm FINAL*		

COURSE MATERIALS AND ADMINISTRATIVE MATTERS

1. Textbook: "Quantitative Chemical Analysis" 9^{9h} Ed., Daniel C. Harris, Freeman. I am told that this comes bundled with a solutions manual for problems (if you buy at the bookstore).
2. Communication: I will use CourseWeb/Blackboard routinely to distribute materials. Note that this system can only send emails to your pitt.edu address. I will at various times set up blog-like capabilities for discussion. Questions that arise during study can be sent to me by email. I will do my best to answer by email. I will always send the question and answer to the whole class after removing any personal information or identity information from your question.
3. Office hours: MW 12:00 – 1:00 PM.

GETTING UP TO SPEED

Several concepts familiar to you from general chemistry are required background. In addition, a couple of excellent examples of analytical measurements illustrate the types of procedures that analytical measurements require. Thus, you will need to read and do exercises on this background material very early in the term. Much of this will not be explicitly covered in lectures – rather it will be assumed that you know this material.

Chapter 0	A great read	
Chapter 1	A good refresher on basic quantities and units.	Do exercises
Chapter 2	Read this to prepare for the lab	
	Read section 2-10 on Excel	
Chapter 3	Skim significant figures rules, but pay attention closely to the "real rule" on page 54.	Do Exercises
Chapter 6, 7	Review concepts of K_{eq}, free energy, chemical equilibria, precipitation, monoprotic acid/base problems, pH	Exercises 6A, E, H, K

GRADING

There are 1000 points. Each of the three hourly exams is worth 200 points. The final exam is worth 400 points.
Grades will be assigned as follows: Letter grade (minimum number of points)
A (930), A- (900), B+ (870), B (830), B- (800), C+ (770), C (730), C- (700), D (600), F (0)

READING

It is essential to read assigned material before the meeting. The relevant reading material should be read BEFORE THE CLASS MEETS. You may find it valuable to work in groups.

HOMEWORK

Homework assignments are given in the schedule. Homework will not be graded.

INSTRUCTOR INFORMATION

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RECITATION

Wednesday evenings, 7:20 – 8:30 PM, Room 154.