INTRODUCTORY ANALYTICAL CHEMISTRY

CHEM 0250

S. G. WEBER, INSTRUCTOR

SYLLABUS

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

There is uncertainty accompanying every quantitative analysis. We will begin the course with a section on types of errors, the magnitude of uncertainty, and (critically) how to test the hypothesis that one result is actually different from another, or from an expected value.

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. Don't be afraid to fail in the process of learning a specific concept or applying a concept in a new domain. Searching for answers may get you a little outside your comfort zone (no textbook-level description, just a research article, for example) but the reward is significant.

PREREQUISITE UNDERSTANDING

Be thoroughly competent in algebra – manipulating equations, solving simultaneous equations, etc.

Be thoroughly competent in the relationships among numbers and their logarithms.

Be able to graph a function in excel, e.g., $y = \exp(-x/a)$.

Know the difference between "mole" and "molar".

Know the relationship among standard state free energy, enthalpy and entropy.

Know what an equilibrium constant is and how it relates to free energy.

Understand stoichiometry.

Be able to use the LeChatelier principle to predict a reaction's response to a perturbation.

LEARNING OBJECTIVES

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

Understand the differences among types of equations. There are equations that are definitions, equations that dictate *what happen*s in a physical or chemical system and equations that *come from* understanding the chemistry. We will mostly derive and use the latter.

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

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

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

TOPICS COVERED

Begins	Topic	Reading	Homework
26 Aug – 9 Sep	Unit 1. Introduction and overview. Methods, Calibration Measurements and statistics, probability distributions, statistical parameters	Read Chapter 1, Ch 2 section 2-10 on excel and as needed for lab; Ch 3, 4, 5	Ch 1 Ex D Problems 1.6, 8, 26 Ch 2 Ex E Ch 3 Ex-A, B, D Problems 3.10, 14, 15, 18, 20 Ch 4 Ex E, F, G Problems 4.2, 6 a,b, 12, 15 and 16 (they are paired) Ch 5 Ex A-D Problems: 9, 14, 15, 16, 18, 19, 20, 27, 31
11 Sep	**EXAM**		
16 Sep – 9 Oct	Chemical Equilibrium	Ch 6-11	
14 Oct	**EXAM**		
16 Oct-30 Oct	Electrochemistry and Spectrophotometry	Ch14-17, Ch 18-21	
4 Nov	**EXAM**		
6 Nov – 18 Nov	Chromatography	Ch 23-26	
20 Nov	**EXAM**		
2-4 Dec	Mass Spectrometry	Ch 22	
11 Dec	**FINAL EXAM 6:00 PM, RM 154 CHVN**		

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. I will occasionally use TopHat for reinforcement.
3. Office hours: W 12:00 – 1:00 PM or appt.

GRADING

There are 650 points. Each of the four hourly exams is worth 100 points. The final exam is worth 200 points.
Quizzes, extra credit, etc. will be 50 pt. Grades will be assigned as follows: Letter grade (minimum number of points)
A (600), A- (580), B+ (560), B (530), B- (500), C+ (470), C (420), C- (400), D (300), 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

S. G. Weber, x 48520, 603 Chemistry Building. Email: SWEBER+@PITT.EDU

RECITATION

Wednesday evenings, 7:20 – 8:10 PM, Room 154.