# ANALYTICAL CHEMISTRY II -- CHEM 425 Spring 2013
# TENTATIVE LECTURE SCHEDULE

INSTRUCTOR: Dr. Petr Vanýsek; Office, Faraday West 418
Meeting place for all sections: Faraday West 201 at 8:00-9:15 Tuesdays and Thursdays

OFFICE HOURS: 9:30 – 10:30 Tuesdays and Thursdays. Other times by appointment only. I will help you with your problems, but when you come to see me, have your questions and problems already at least partially prepared. Bring your class notes along; I will want to see what you write down. Do not expect the instructor to give you your own private make-up class. When coming to the office hours, be prepared to share the office or the time with other students.

Laboratory sections (all meet in Faraday West 304)

| | | | |
|---|---|---|---|
| 1 | Tuesday | 17:30-21:20 | TAs: Not yet assigned by the DGS |
| 2 | Thursday | 14:00-17:50 | Prof. G. Baker as of |
| 3 | Thursday | 18:00-21:50 | 9 Jan. 2013 |

TEXTBOOK: D. A. Skoog, F. J. Holler and S. R. Crouch: Principles of Instrumental Analysis, 6th Ed. Thompson Brooks/Cole, Belmont, CA 2007. ISBN: 0-495-01201-7.

| DATE dd.mm.yy | TOPIC | CHAPTER |
|---|---|---|
| 15.01.13 | Introduction | |
| 17.01.13 | Electronics, signals and noise | 2-5 |
| 22.01.13 | Electronics, signals and noise | 2-5 |
| 24.01.13 | Electronics, signals and noise | 2-5 |
| 29.01.13 | Electronics instrumentation, handling of data | class notes |
| 31.02.13 | Radiation, spectrometer components | 6-7 |
| 5.02.13 | Radiation, spectrometer components | 6-7 |
| 7.02.13 | Test I | |
| 12.02.13 | Schemes to detect radiation | 6-7, handouts |
| 14.02.13 | Atomic spectrometry | 8-10 |
| 19.02.13 | Atomic spectrometry | 8-10 |
| 21.02.13 | Atomic spectrometry | 8-10 |
| 26.09.13 | Molecular spectrometry | 13-18 |
| 28.02.13 | Molecular spectrometry | 13-18 |

| | | |
|---|---|---|
| 5.03.13 | Test II | |
| 7.03.13 | | |
| 12.03.13 | Spring break | |
| 14.03.13 | Spring break | |
| 19.03.13 | Molecular spectrometry | 13-18 |
| 21.03.13 | Molecular spectrometry | 13-18 |
| 26.03.13 | X-ray spectrometry<br>and surface characterization | 12 & 21 |
| 28.03.13 | X-ray spectrometry<br>and surface characterization | 12 & 21 |
| 2.04.13 | X-ray spectrometry<br>and surface characterization | 12 & 21 |
| 4.04.13 | Test III | |
| 9.04.13 | Electrochemistry | 22-25 |
| 11.04.13 | Electrochemistry | 22-25 |
| 16.04.13 | Electrochemistry | 22-25 |
| 18.04.13 | Electrochemistry | 22-25 |
| 23.04.13 | Electrochemistry | 22-25 |
| 25.04.13 | Electrochemistry | 22-25 |
| 30.04.13 | Electrochemistry | 22-25 |
| 2.05.13 | | |

**Schedule of the tests (all in FW 201):**

| | | |
|---|---|---|
| 7 February | Test I | 100 points |
| 5 March | Test II | 100 points |
| 4 April | Test III | 100 points |
| 7 May | Final 8:00 – 9:50 | 200 points |

GRADING:

Tests: 500 points

Laboratory: 280 points, as described in the laboratory handout.

Total of 780 points is possible. Percent average (earned points divided by 7.8) will be used for determining the final grade. The following is a tentative scale: 85% A, 75% B, 65% C, 55% D, less than 55% F. Note however, that you have to complete all the laboratory assignments to get a passing grade.

**ACADEMIC INTEGRITY:** In general, cheating means presenting or using work that was not done entirely by you and, in the case of in-class examination, it includes also presenting or using your work that was written outside the classroom. You may not talk or pass notes to each other on any subject. Having other materials than those

allowed for the work within your reach during test or sharing calculators is cheating as well. During tests you must put away any devices that would allow you to communicate with others or access databases. Be very careful not to use work of somebody else (a lab mate, another group's report, previous years' reports, web downloaded material) for the laboratory report. Violation of these rules will result at minimum of a zero score on your work.

**Other issues:**

- Calculators: For tests only calculators without data storage/retrieval capability can be used. The calculator function on cell phones cannot be used during tests. Cell phones must stay away out of your reach during any tests or quizzes.
- No smoking in the building, no food or drink in the class or the laboratory.
- TAPING/RECORDING OF THE LECTURE: You are encouraged to take good notes, reflecting your interpretation and understanding of the lecture. However, you are not permitted to make verbatim recording or transcription of the lecture.
- ATTENDANCE: The material in the lectures is essential for understanding the subject. Although there is no formal enforcement of attendance, due to the size of the class your absence will be clearly apparent to me. Be prepared to explain and justify your absence to me.
- CELL PHONES AND THE LIKE: Cell phones are great technology and it is great to have one with you for emergency. (Campus police: 815-753-1212). However, please, turn off your phones and other noise-making devices as a courtesy to others, and do not distract yourself by reading and sending text messages.

Note on the material included in Analytical Chemistry II and in particular that, which is *not* covered there:
The textbook is an excellent resource and as you are planning to become a chemist, keep the book as a reference, do not sell it at the end of the semester. The textbook has lot of instrumentation subjects. Some are not covered, because there is not enough lecture time in one semester. However, some are excluded on purpose, because they were (or supposed to have been) covered in other courses at the department. You should know the material – in fact, in the course of my lecture I am assuming that you know the materials. The specific excluded topics are:
Infrared spectroscopy. Covered in relation to organic compounds in CHEM 332.
NMR. Covered in CHEM 332.
Thin Layer Chromatography. Covered in CHEM 332.
and all the material covered in Analytical Chemistry I, CHEM 325 including:
Statistics and data treatment
Gravimetry and proper use of balances
Mass Spectrometry
Chromatography (GC, LC, SEC and connection to MS)

Additional syllabus material and class updated information can be found on the web:
http://www.vanysek.com/electrochemistry/425_material/2013_spring.htm

## TENTATIVE LABORATORY EXPERIMENTS

| | **EXPERIMENT** | Date (Monday of the week) | Instrumentation |
|---|---|---|---|
| 1. | Introduction to the uses of a digital multimeter, resistor and a resistor circuit | Jan. 21 | Electronics sets |
| 2. | Introduction to an oscilloscope and operational amplifiers | Jan. 28 | Electronics sets |
| 3. | Operational amplifiers, part II | Feb. 4 | Electronics Sets |
| 4. | Flame atomic absorption of cadmium | rotating schedule | |
| 5. | UV-VIS spectrometry | rotating schedule | Spec 20 |
| 6. | Spectroscopic determination of manganese in steel | rotating schedule | Spec 20 |
| 7. | Fluorescence spectrometry | rotating schedule | Fluorometer |
| 8. | Potentiometric simultaneous titration of chloride and bromide | rotating schedule | pH meter |
| 9. | Ion selective electrodes – Chloride and fluoride | rotating schedule | pH meter |
| 10. | Amperometric titration | rotating schedule | potentiostat |
| 11. | Cyclic Voltammetry | rotating schedule | potentiostat |
| 12. | Rotated disk electrode | rotating schedule | potentiostat + RDE |
| 13. | Bipotentiometric end-point indication | rotating schedule | potentiostat |

Material needed: (1) Your textbook, (2) handouts available from the web, (3) bound a page-numbered laboratory notebook, (4) pre-lab notes demonstrating that you came prepared to do the lab, (5) calculator, ruler, pencil, etc., (6) protective gear. The TA will specify protective gear and any other safety related matters. Necessary handouts will not be available in the lab. They can be downloaded on the web:
http://www.vanysek.com/electrochemistry/425_material/2013_spring.htm
For writing the laboratory reports consult the specific handout.
The experiment is completed by submitting a laboratory report to the teaching assistant. Laboratory reports are due one week after the lab was performed. The experiment, which would be due during the spring break, is due one week later. No exceptions! There is a late penalty of two points for each calendar day the report is late.
Each laboratory report is worth 20 points. You have to finish all the 13 laboratories to pass. The quality of your laboratory notebook and timely note taking is worth 20 points. The notebook will be casually inspected and initiated by the TA every lab, as you are departing. From these points the teaching assistants may subtract demerit points for safety violations, tardiness, sloppiness and other breach of common sense and good manners. There is 280

points total in the labs. (If, for serious reason, *e.g.*, failed instrument, a lab has to be cancelled, the grading will have to be adjusted accordingly, *i.e.*, fewer required points for the labs.)

## FORMAT FOR LABORATORY REPORTS

(Note that additional requirements and due-dates will be specified by the teaching assistants.)
Word processed reports are required. The length should be a maximum of 4 pages. Standard font (10 or 12 pitch, 12 and Ariel is preferred) and single spacing should be used. The page format limit cannot be achieved by judicious adjustment of font sizes and margins.
You should prepare Sections I – IV before the laboratory begins and bring it with you to the lab.

Section I **NAME**. Give your name (on top right corner), date(s) the experiment was performed and the date submitted, course number (CHEM425), section (day of the week, time), full name of the TA in charge, names of the partners, if you were split into groups (which you will be). Do not generate a separate page only as a cover page.

Section II **TITLE**. Experiment title and number (from the syllabus), identification number of the unknown and what concentration was determined for the unknown (with units and standard deviation).

Section III **OBJECTIVE**. Give a brief statement of the problem or experiment. State the parameter(s) to be determined. Do not use the text from the handouts, use your own words.

Section IV **METHOD and MATERIALS**. Describe the method to be used and the basic principle of the method. Write in your words a brief synopsis of the experiment, following the handout, but omit procedural details unless there is a difference from those given. List equipment used. If you are using one of several identical instruments (*e.g.*, a pH meter, an oscilloscope) note the inventory or serial number on your list.

Section V **CALCULATIONS**. Write all the calculations in a neat way here. Write first a general formula, using formal variables. Define the variables. Only then show a numerical calculation. (If a particular tricky equation is used, you may insert it by hand. However, learning how to do it on a word processor is a skill that will take you long way. Consider using an equation editor.)

Section VI **DATA**. Set the table conveniently to record all significant obtained data. If a large set of data is obtained as on a computer you should decide on some convenient way to reduce the number of printed points. Data output should be always supplemented (if not bypassed) by a graph.

Section VII **RESULTS & ERROR**. Write the results from section (V) here. Remember to identify the unknown sample by its number or letter. Recall the rules of error reporting and significant figures.

Section VIII **GRAPHS, DISCUSSIONS, QUESTIONS, PROBLEMS**. Report what you have learned, provide interpretation of the results. Compare with literature values of expected values. Point out accuracy and precision, possible sources of error, unusual aspects encountered and their possible effects on results, advantages and disadvantages (or limitations) of the technique, ideas for further work. Include here also answers to specific questions and exercises posed in the instructions that accompanied the assignment. This is where a graph imported into the word processor

should fit.

From the report itself, the significance as well as the eventual use of the data should be clear to a knowledgeable reader who has not read the experimental procedure. A good two-step test is: 1) Do the plots and tables stand alone? Are all the units and their symbols included (use **SI units**) and do the titles clearly state the data contained? As an example, peruse an issue of *Analytical Chemistry*. 2) Does the text adequately explain the data and point out important values? Is the language correct?

Adherence to the above requirements as well as neatness and legibility of the work will be graded in addition to the correct value of the unknown and a sound discussion of results. Some of common errors include omission of units, reporting in wrong units (Do not forget any dilution you may have done. Typically, if an unknown is issued in a volumetric flask, report concentration when diluted to the mark.), omission of the standard deviation (Make enough experiments to be able to calculate it!), too few or too many (usually) significant figures and careless graphs.

Printed 8 January 2013