GEORGE MASON UNIVERSITY
CHEM 423- Instrumental Analysis Laboratory
Syllabus

Prof. Abul Hussam
Room: 337 or 408A (lab)
ahussam@gmu.edu

During next few weeks you are required to carry out a series of experiments designed to strengthen your understanding in instrumental techniques for modern quantitative chemical analysis. The techniques that you learn here are the basis for professional chemists working in industry, research organization and academia. This course requires that you have passed the chemistry courses: Organic Chemistry (CHEM 314), Elementary Quantitative Analysis (CHEM 321), Physical Chemistry I (CHEM 331), and Instrumental Analysis Lecture (CHEM 422), and have understood the materials. In addition, a familiarity with a spreadsheet program such as MS-Excel and a word-processing program is mandatory. The syllabus, the laboratory instruction materials, and all other information are posted on the 'Blackboard' for registered students. You must also have the textbook used in CHEM 422 for reference. The recommended textbook is: Quantitative Chemical Analysis by Harris, 8^{th} edition or later.

The instrumental analysis laboratory begins with a lecture by your instructor. The lecture materials cover basic principles of instrumental analysis, statistical analysis of data, and the discussion of the experimental sections. The class quizzes are based on these lectures and the reading materials from appropriate sections of Laboratory Manual and Instrumental Analysis text book prescribed by your instructor. A tentative lecture schedule is given below.

The list of experiments is shown in the following page. All experiments are performed in a group and each group performs different assigned topics. The instructor introduces the experiment to one group at-a-time. The instructor and an instrument specialist are available as needed. You must bring your standard lab notebook, a flash-drive, and a safety-glass. You may bring your laptop to record data as you progress.

You are required to submit a formal laboratory report for each experiment a week after the experiment is finished or on assigned dates. After you gather the experimental data, you are solely responsible for your lab report. You are not allowed to collaborate on writing your lab report. The report format is discussed in the class and an example laboratory report is posted. You are also required to take a final examination on theoretical and experimental aspects of the experiments performed. **The overall grade is based on 70% from laboratory report, 15% from the lab final exam, and 15% from homework/ lab quizzes/ lab attendance, and lab performance.**

LIST OF EXPERIMENTS

Expt. (duration)	Topics
A (2 week)	**UV-Vis Spectroscopy** 1. UV absorption spectrum of benzene in vapor and liquid phase: Effect of slit width and measurement of molar absorptivity 2. Spectrophotometric analysis of a two component system with overlapping spectra: Determination of Cu and Ni in a mixture
B (1 week)	**Fluorescence Spectroscopy** 1. Excitation and emission spectra of quinine in water 2. Determination of quinine in Tonic Water. 3. Effect of quenching agent on fluorescence intensity
C (2 week)	**Potentiometry with Ion Selective Electrode** 1. Calibration of a fluoride ion selective electrode (FISE) 2. Measurement of fluoride in tap-water 3. Auto-titration of F^- with HCl and simultaneous measurement pH and F^-. Calculation of dissociation constants for HF and HF_2^-.
D (1 week)	**Dynamic Electrochemistry** 1. Characterization of a redox couple with chronoamperometry, cyclic voltammetry and square-wave voltammetry. 2. Determination of trace Pb^{2+} and Cd^{2+} in water Anodic stripping voltammetry.
E(2 week)	**Liquid Chromatography** 1. Separation of aromatic hydrocarbons by reversed phase liquid chromatography (RPLC) 2. Measurements of column efficiency and thermodynamic partition coefficients. 3. Extraction and determination of chemical species (pesticides) in soil.
F (2 week)	**Gas Chromatography-Mass Spectrometry (GC-MS)** 1. Isothermal separation and identification of a mixture of aliphatic hydrocarbons. 2. Measurements of column efficiency and thermodynamic partition coefficients. 3. Separation, identification, and quantitative measurements of compounds in unleaded gasoline.
G (1 week)	**Basic Electronics** 1. Low-pass RC filter: Measurements of time constant and frequency response. 2. Operational amplifiers: Characteristics of a follower, inverting and non-inverting amplifier. 3. Build a pH measurement system by using operational amplifiers and record an acid-base titration curve.
H (1 week)	**Atomic (Microwave Plasma) Emission Spectroscopy** 1. Preparation of standard calibration samples for trace metals 2. Generation of calibration curves for at least three trace metals. Measurement of sensitivity, detection limit, and quantitation limit. 3. Measurement of unknown sample concentration for at least three unknowns.

Lecture Title	Lecture Topics	Dates & Duration
Data Treatment: and Error Analysis	Types of errors. Accuracy. Precision: Standard deviation, %rsd, Repeatability. T-test: Confidence interval of true mean, comparing replicate measurements by t-test. Data rejection: Q –test. Error propagation: Propagation of random errors. Concentration calibration: Errors in concentration measurement. Linearity and dynamic range. Detection limit: $dl_{signal} = s_{blank}. t_{N-1, 98\%}$ or $dl_{concn} = 3\ s_{blank} /m$. Signal/Noise = Signal / s_{noise} = 1/ (precision of measurement). Calibration of analytical systems: Direct calibration, One point calibration, Standard addition techniques, and Internal standard technique.	Hour each
Separation Chemistry: Gas and Liquid Chromatography)	Principle: thermodynamics of separation, separation efficiency, origin of band broadening, and band resolution. Capillary gas chromatography: Instrumentation, qualitative and quantitation techniques. Liquid chromatography: The role of solvent composition in reversed phase LC, and instrumentation. Discussion on experimental data analysis.	Hour each
Electrochemistry: Ion Selective Electrodes (ISE)	Principle of potentiometry: the origin of selective interface for pH and fluoride ISE. The use of F-ISE for the measurement of equilibrium constants. Data analysis by using nonlinear routine- the use of SOLVER in Xl.	1.5 hour
Electrochemistry: Voltammetry	Voltammetry: Basic principle of cyclic voltammetry for the measurement of electron transfer dynamics. Principle of pulse techniques (DPV, SWV etc). Square wave voltammetry (SWV): measurement of trace metals by stripping mode.	Hour
Spectroscopy: UV -VIS and Fluorescence	The origin of molecular spectroscopic signals. Energy level transitions. Beers law and limitations. Multicomponent analysis. Discussion on experimental data analysis.	Hour
Spectroscopy: Atomic Absorption and Emission	Origin of atomic spectroscopy. Difference between emission and absorption in atomic spectroscopy. Factors affecting emission and absorption signals. Sample preparation and data analysis.	Hour
Basic Electronics	Voltage divider, low-pass RC filter. Measurements of time constant and frequency response. Operational amplifiers: Characteristics of a follower, inverting and non-inverting amplifier. pH measurement system with operational amplifiers. Introduction of Arduino microcontroller system.	Hour

GEORGE MASON UNIVERSITY
Instrumental Analysis Laboratory (CHEM 423)
Laboratory Policies

During next few weeks, you are required to carry out a series of experiments designed to strengthen your understanding in instrumental analysis as applied to analytical chemistry. This course assumes that you have completed the required chemistry courses: Organic Chemistry (CHEM 314), Elementary Quantitative Analysis (CHEM 321), Physical Chemistry I (CHEM 331), and Instrumental Analysis Lecture (CHEM 422) and have understood the materials. In addition, a familiarity with a spreadsheet program and a word-processing program is mandatory. Apart from following the normal safety regulations, we observe the following general guidelines:

- You should come prepared for the laboratory. Bring your safety glass, the laboratory instructions, a standard lab notebook, and a flash drive. Laboratory session starts promptly at class time.

- You should know your lab schedule and your group number. To avoid overlapping schedule, you must check with your instructor on next week's lab. Accordingly, you should prepare yourself for the laboratory. You should read the laboratory instructions and plan ahead. Especially, if the experiment requires the preparation of a large number of solutions then you should do some of the calculations ahead of time. Each member of the group should share the work. You should also read the Instrumental Analysis textbook on the technique that you are going to use. This reading should be coordinated with your laboratory instructions.

- Since each group is using a different instrument or setup, it is not possible to give a general lecture. The instructor, in the beginning of the lab period shows you the basic operation of the instrument. Please remember that many of the instruments you are using are research grade and expensive. Therefore, do not venture into doing something that you are not certain about. Ask your instructor for help in such cases. You should also be prepared for situations when the instrument may not function properly and your experiment may be delayed or it may be modified by your instructor to obtain meaningful results. You should always note these situations in your notebook.

- Cleanliness is absolutely essential in a good laboratory practice (GLP). Maintain your bench space, the instrument space, and the balance area clean. Do not leave anything on the balance pan and re-zero the balance after your use. There may be many sources of errors from sloppy lab practices. Watch out for these errors. If you are not sure about the proper way of handling chemicals, apparatus and equipment, ask your instructor or the instrument specialist for a demonstration.

- Do not throw away large quantities of solutions or chemicals. Perhaps it can be stored safely or can be used by the next group assigned to do the same experiment. In this way, you conserve chemicals and time. It also makes a good environmental sense.

- After finishing the day's work examine your notebook and be certain that you have all the data necessary to write a complete lab report. Remember, you only share the raw data. Observations, derived data, graphs, results, calculation, and discussion should be your own. You must checkout with your instructor before you leave the laboratory.