

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 are the basis for professional chemists working in industry, research organization and academia. This course requires that you have completed 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 are essential. You must purchase the following instruction manual from the copy center: Instrumental Analysis Laboratory Manual (CHEM 423) by Dr. Abul Hussam. You should also have two textbooks used in CHEM 321 and CHEM 422 for reference. The recommended textbooks are: Quantitative Chemical Analysis by Harris (or any book on quantitative chemical analysis) and Instrumental Analysis by Skoog and Leary, 4<sup>th</sup> edition or later.

The following experiments are performed during the course. All experiments are performed in a group and each group performs different assigned experiments. You are required to submit a formal laboratory report for each experiment on assigned dates. The report format is discussed in the class and an example laboratory report is distributed. 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**

<b><u>Expt No.(duration)</u></b>	<b><u>Topic</u></b>
A (1 week)	Spectrophotometric Determination of Co & Ni as EDTA and Aqua Complexes
B (1 week)	Fluorescence Spectroscopy: Determination of Quinine in Tonic Water
C (2 weeks)	Atomic Emission and Atomic Absorption Spectroscopy: Trace Metal Analysis
D (2 weeks)	Potentiometry with Ion Selective Electrode: Fluoride Analysis
E (2 weeks)	Basic Electrochemistry Experiments: Linear Scan Voltammetry and Anodic Stripping Voltammetry.
F (2 weeks)	Gas Chromatography: Analysis of Hydrocarbons
G (2 weeks)	Liquid Chromatography: Separation of Organics

The instrumental analysis laboratory begins with a lecture by your instructor. The lecture materials comprise 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, Quantitative Analysis, and Instrumental Analysis text books as prescribed by your instructor. A tentative lecture schedule is given below:

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: $dl_{\text{signal}} = S_{\text{blank}} \cdot t_{N-1, 98\%}$ or $dl_{\text{concn}} = 3 S_{\text{blank}} / m$ . Signal/Noise = Signal / $S_{\text{noise}} = 1/$ (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 XI.	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

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 (Excel, QuatroPro etc.) and a word-processing program are essential. 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 manual, a laboratory notebook, and computer diskettes. 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 manual 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 manual.
- 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 rezero 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 apparatus and equipment, ask your instructor 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 that you only share the raw data. Observation, derived data, graphs, results, calculation, and discussion should be your own. **You must checkout with your instructor before you leave the laboratory.**