CHMC 16 Instrumental Analysis (Syllabus)

Course Description
CHMC16 is a laboratory course designed to compliment CHMC11 (Principles of Analytical Instrumentation). This course will provide a practical introduction and experience in the use of modern analytical instrumentation, and stress its increasing significance in 21st century research. Students will face a number of real world challenges and learn how to apply instrumental approaches to overcome them. Emphasis will be placed on sample preparation, instrumental operation/methods, and data interpretation for a range of pharmaceutical, biological, environmental, and industrial samples.

The lab will be a mix of demonstration and practical, depending on the instrumentation involved. Fourier Transform Infrared Spectroscopy (FT-IR), Atomic Absorption Spectroscopy (AA), Ultra Violet – Visible Spectroscopy (UV-VIS) will be considered briefly. However emphasis will not be placed on these approaches as they have been introduced in earlier years.

The focus of the course will be on the following instrumentation :

Electrospray Ionization Mass Spectrometry (ESI-MS) and High Performance Liquid Chromatography (HPLC)

Students Learn :

- Various sample preparation techniques
- The acquisition of MS and MS^n data
- The isolation of compounds from mixtures.
- The identification of compounds from fragmentation patterns
- The limitation and advantages of the technique

Nuclear Magnetic Resonance Spectroscopy (NMR)

Students Learn :

- Various sample preparation techniques
- The acquisition of basic 1D and 2D datasets
- The identification and quantification of components
- The use of simulations in spectral interpretation
- The limitation and advantages of the technique

Gas Chromatography and Gas Chromatography-Mass Spectrometry (GC-MS)

Students learn :

- Sample injection methods
- Important experimental variables
- Calculation of concentrations in unknowns
- Quantitative separations of mixtures
- Experimental design
- The limitation and advantages of the technique

CHMC16H3 - Analytical Instrumentation

The course will be split into 6 main sections. The class split into 2 groups. One Group will work on section 1-3, while the other section works on sections 4-6. Half way through the class groups will swap.

Section 1. Training on HLPC, MS, and NMR

This is a two week practical. The aim is to provide basic training on the most sophisticated pieces of instrumentation, and provide a basic understanding as to the capabilities and limitation of each technique.

Section 2. Determination of Caffeine in Tim Horton's Coffee (Analytical Emphasis)

The Situation (fictitious !!)

Caffeine in high quantities can kill. Numerous members of the public have been suffering from heart problems, sleep problems etc. They think it is connected to the Tim Horton's coffee they drink and have filed a class action law suite. Tim Horton's lawyers have hired you test the caffeine level in their coffee. They are lawyers ! they don't care how you do it, but they just need number that will "stand up" in court.

The problem.

You have at your disposal and ESI-MS, an LC-ESI-MS, and an NMR. Which instrument is most appropriate for the task ? You are going to design experiments that measure the caffeine level using **ALL** of the approaches. You are going to have to think of the advantages and disadvantages of each approach. How you could improve the methods in the future etc ?

Section 3. Determination of Components in a Pharmaceutical Tablet (Emphasis on Pharmaceutical Chemistry)

The Situation

Drug companies spend billions on quality control. They have to make sure their tablets really contain what they say they do ? Sometimes outside labs are asked to double check the work of the drug companies to make sure there is no corruption, fraud, or potentially fatal mistakes.

The Task.

We will be given a commonly used "over the counter" drug. You have at your disposal and ESI-MS, an LC-ESI-MS, and an NMR. This time you should use the information from each instrument together to try and find out what these tablets contain, and is it the same as on the label ?

Section 4. UV-VIS (Emphasis on Food Chem.)

This practical is for a single week. In UV-VIS spectroscopy you will develop an experiment to study the concentration of iced tea in commercial samples. You must describe and assess the wide spread applicability of the method you devise.

Section 5. FT-IR (Environmental Emphasis)

This will be a 2 week practical. You will look at a range of components that would commonly be found in the environment and to be become familiar with the FT-IR spectra that are produced by the different materials, and different methods of preparation. It will be important to see if characteristic signals for different types of materials are observed. This we will provide preliminary information and experience for the practical in week 2 which will deal directly with natural samples such as plant tissues directly.

Section 6. GC and GC-MS (Industrial Emphasis)

This will be a 3 week practical. Week 1. Will introduce you to the GC. You will learn the basic of the instrument, how to inject basic samples, and some of the important experimental parameters. Week 2. You will run some hydrocarbons and then gasoline. This week will introduce you to working with complex mixtures and “real” samples. Week 3. You will now run the same gasoline on a GC-MS. You should begin to appreciate the advantages of MS detection.

Assessment for CHM C16

Irrespective of which instrument you are using, we will always meet in S165.

Assessment

There will be ***no*** final exam for this course. Students will be assessed on the following criteria.

1) 5 x Lab reports. Lab reports are worth 12% each.
2) 1 x term paper (20%)
3) Ability and Performance in the Lab sessions (20%). Remember this is a lab course you will be evaluated on your involvement, safety (lab glasses, coat), you ability to work with your team members, your ideas especially in "the research project section", your ability to keep a lab manual that can both be used to verify your results, and repeat your work, your timeliness, and your ability to organize your time and leave the lab in good shape.

Lab reports are to be written individually and each student will be expected to attach their own copies of the relevant chromatograms, spectra etc with their reports. Plagiarized reports will not be accepted.