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 provide practical hands on experience to a wide range of instrumentation. This includes Total reflection X-ray Fluorescence (TR-XRF) and Energy Dispersive X-ray Fluorescence (EDXRF), Fourier Transform RAMAN (FT-RAMAN), Atomic Absorption Spectroscopy (AA), Capillary Electrophoresis (CE), High Performance Liquid Chromatography (HPLC), Electrospray Ionization Mass Spectrometry (ESI-MS), Nuclear Magnetic Resonance Spectroscopy (NMR) and Gas Chromatography-Mass Spectrometry (GC-MS) . UV-VIS and FT-IR will be discussed 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:

High Performance Liquid Chromatography

Students Learn :

- Various sample preparation techniques, including SPE and Reflux
- Isocratic and Gradient Elution's
- The isolation of compounds from mixtures.
- The identification of compounds using various detectors
- The limitation and advantages of the technique

Electrospray Ionization Mass Spectrometry (ESI-MS)

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
- Mixture Analysis by NMR
- The use of Electronic References for Quantification

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

Students learn :

- Sample injection methods
- Correlation of Theory and Practice through Van Deemter Plots
- Important experimental variables
- Calculation of concentrations in unknowns
- Quantitative separations of mixtures
- Experimental design
- The limitation and advantages of the technique

Energy Dispersive X-ray Fluorescence (EDXRF), Atomic Absorption Spectroscopy (AA) and Total reflection X-ray Fluorescence (TR-XRF)

Students learn :

- Compare Various Methods of Metal Analysis
- Understand and contrast detection limits
- Surface vs. Bulk Analysis
- Destructive vs Non-destructive Analysis
- Calculation of concentrations in unknowns

Fourier Transform RAMAN (FT-RAMAN)

Students learn :

- Understand the role and potential of FT-RAMAN in a range of analyses
- Analyze a range of mineral and rock samples
- Interpretation of Spectral Data
- Qualification Run library matching
- Quantification

Capillary Electrophoresis (CE)

Students learn :

- Anion and Cation Analysis
- Reverse and Normal EOF
- Effects of Buffer, pH, Sample Stacking
- Indirect UV Detection
- Developing and programming methods
- Quantification

CHMC16H3 - Analytical Instrumentation

The course will be split into 4 main sections :

Section 1. Three weeks split between, RAMAN, TRXRF, CE
Section 2. Three weeks will be spent learning and applying NMR
Section 3. Three weeks will be spent on GC and GC-MS
Section 4. Three weeks will be spent learning HPLC and HPLC-MS

Unless stated otherwise we will always meet in S141.

Assessment

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

1) 4 x Lab reports. Lab reports are worth 15% 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.

Late Reports will not be accepted unless you have a Dr's note

Acknowledgement : Some sections of these practicals have modified from "Chemistry Experiments from Instrumental Methods" by Sawyer, Heineman and Beebe.

Week Number by Group

Week Number

Lab	***1***	***2***	***3***	***4***	***5***	***6***	***7***	***8***	***9***	***10***	***11***	***12***
RAMAN/TRXRF/CE	*A*	*A*	*A*	*B*	*B*	*B*	*C*	*C*	*C*	*D*	*D*	*D*
GC/GC-MS	*B*	*B*	*B*	*C*	*C*	*C*	*D*	*D*	*D*	*A*	*A*	*A*
HPLC/HPLC-MS	*C*	*C*	*C*	*D*	*D*	*D*	*A*	*A*	*A*	*B*	*B*	*B*
NMR	*D*	*D*	*D*	*A*	*A*	*A*	*B*	*B*	*B*	*C*	*C*	*C*

Do not enter research labs, unless your TA is present. If you are early wait outside.

Contacts and Office Hrs

Office Hrs : Wed 2-4.30pm. In Environmental NMR Center (take elevator to basement of the **science research building** and bang on the large double door that are located around the corner) E-mail : andre.simpson@utoronto.ca

Lab Books, Cleaning Up, and Leaving

Before Leaving

Make sure all chemicals have been returned and that all apparatus, has been cleaned an returned to its correct location. YOU WILL LOOSE MARKS IF YOU LEAVE A MESS. LOTS OF THE EQUIPMENT YOU WILL BE USING IS VERY EXPENSIVE RESEARCH EQUIPMENT, TREAT IT WITH RESPECT !!

Lab Manuals :

Lab Manuals are to be kept throughout the course. ***You must get these initialed by the instructor or demonstrator at the end of each session*** after you have cleaned up and shut down all the instrumentation properly. Lab manuals **must be handed in along** with the last lab report. **It is your responsibility to get you lab book signed each week. IF SIGNATURES OR LAB BOOKS ARE MISSING AT THE END OF THE COURSE THEN YOU WILL LOOSE MARKS.**

All students are required to make their own notes and observations in the lab books as they feel appropriate

Example Lab Report + Schedule

Overall Title (i.e. Gas Chromatography)

Name : Student Number Date :

Names of Other Student in the same practical group

Subtitle (i.e. Week 1 : Determination of Optimal Flow Rate in Gas Chromatography)

Make a note here of any special circumstances. For example : *"As is was the first week of class only 4 of the 6 compounds were available"*

Treatment of Data and Results

Here you will be expected to work chronologically through the practical write up. You are expected to carry out all the instructions in the "Treatment of Data Sections" throughout the text. You will be expected to clearly label and hand in all spectra or data collected during the practical. Each individual will be responsible to obtaining his own individual copy of any relevant data. Any appropriate experimental conditions should be clearly marked. In this section discuss any relevant finding or observations that you made during the practical.

Questions

Answer all questions throughout the text as fully as possible.

Conclusions

In this section summarize your major findings, and what you think you learnt from the practical session. Address any problems encountered. Suggest improvements that could be made to improve the results.

Repeat Format for week 2 and hand in complete report.

The practical report should be submitted in full the week following the last practical session on a particular type of instrumentation. Exact dates for handing in each assignment are given on the next page.