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 experience with 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

Logistics

The course will be split into 4 main sections :

Section 1. RAMAN, TRXRF, CE
Section 2. NMR
Section 3. GC and GC-MS
Section 4. HPLC and HPLC-MS

Under Quercus, under people, you can find out what group you are in.

The groups are labelled A-D. Just join the session that you will be doing for that week, based on the table on the next page.

Use the following zoom links per section

Section 1. RAMAN, TRXRF, CE
https://utoronto.zoom.us/j/86584910069

Section 2. NMR
https://utoronto.zoom.us/j/81401237209

Section 3. GC and GC-MS
https://utoronto.zoom.us/j/84621132153

Section 4. HPLC and HPLC-MS
https://utoronto.zoom.us/j/87888896990

Lab Session by Group. Note Yellow is online and Green in-person (covid restrictions permitting).

In Person will occur if Covid Restrictions are Lifted

Date	RAMAN/TRXRF/IC	GC/GC-MS	HPLC/HPLC-MS	NMR	Comment
Wed 12th Jan	**A+B**	**-**	**-**	**C+D**	Online
Wed 19th Jan	**A+B**	**-**	**-**	**C+D**	Online
Wed 26th Jan	**C+D**	**-**	**-**	**A+B**	Online
Wed 2nd Feb	**C+D**	**-**	**-**	**A+B**	Online
Wed 9th Feb	**-**	**A+B**	**C+D**	**-**	Online
Wed 16th Feb	**-**	**A+B**	**C+D**	**-**	Online
Wed 23rd Feb	**----------Reading Week No Class ----------**				
Wed 2nd Mar	**-**	**C+D**	**A+B**	**-**	Online
Wed 9th Mar	**-**	**C+D**	**A+B**	**-**	Online
Wed 16th Mar	**A**	**B**	**C**	**D**	In-Person
Wed 23rd Mar	**B**	**C**	**D**	**A**	In-Person
Wed 30th Mar	**C**	**D**	**A**	**B**	In-Person
Wed 6th Apr	**D**	**A**	**B**	**C**	In-Person

Assessment

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

1) 3 x Lab reports. Lab reports are worth 33.33% each (see later, "RAMAN/TRXRF/CE" no lab report this year)

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 have 5% removed for each hour they are late unless you have a Dr's note

The submission time in Quercus will be used to determine this. Reports are due by 10am

YOU MUST UPLOAD YOUR ASSIGNMENT AS A SINGLE PDF FILE USING ASSIGNMENTS IN QUERCUS

Contacts and Office Hrs

Office Hrs. If you need to meet with me during the course feel free to e-mail me andre.simpson@utoronto.ca. I will send you a zoom link and we can chat online.

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

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.

Dates Reports are Due !!!!

Note if we have to switch things around – REPORTS ARE ALWAYS DUE ONE WEEK AFTER THE LAST CLASS ON THAT SPECIFIC INSTURMENT. NOTE FOR SECTION "RAMAN/TRXRF/CE**" there is NO lab report.**

YOU MUST UPLOAD YOUR ASSIGNMENT AS A SINGLE PDF FILE USING ASSIGNMENTS IN QUERCUS

Date	RAMAN/TRXRF/IC	GC/GC-MS	HPLC/HPLC-MS	NMR	Comment
Wed 12th Jan	-	-	-	-	
Wed 19th Jan	-	-	-	-	
Wed 26th Jan	-	-	-	**C+D**	**1st Lab Report Due**
Wed 2nd Feb	-	-	-	-	
Wed 9th Feb	-	-	-	**A+B**	**2nd Lab Report Due**
Wed 16th Feb	-	-	-	-	
Wed 23rd Feb	**----------Reading Week No Class ----------**				
Wed 2nd Mar	-	**A+B**	**C+D**	-	**3nd Lab Report Due**
Wed 9th Mar	-	-	-	-	
Wed 16th Mar	-	**C+D**	**A+B**	-	**4th Lab Report Due**
Wed 23rd Mar	-	-	-	-	
Wed 30th Mar	-	-	-	-	
Wed 6th Apr	-	-	-	-	

WE MUST RECEIVE YOUR REPORT BY 10AM ON THE DATE INDICATED IN THE TABLE ABOVE.

In Person will occur if Covid Restrictions are Lifted