

Course Outline
Analytical Chemistry II: Instrumental Analysis
CHEM*3430 Summer 2015

Lecturer: Rick deLaat (SSC 3244)

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Lectures: Tues, Thur 11:30 AM - 12:50 PM MCKN 314

Office Hours: to be determined in class

Lab Supervisor: Dr. Kate Stuttaford (SSC 3113A)

Email: kstuttaf@uoguelph.ca

Labs: Tues, Thur 2:30-5:20 PM SSC 3105

Lab Teaching Assistant: Jadwiga Lyczko

Undergraduate Calendar Description:

CHEM*3430 Analytical Chemistry II: Instrumental Analysis S,W (3-3) [0.50]

This course examines methods for the separation, identification and quantification of substances in the solid, liquid and vapour states. Emphasis will be placed on modern instrumental methods and trace analysis.

Prerequisite(s): (CHEM*2400 or CHEM*2480)

Restriction(s): TOX*3300

Department(s): Department of Chemistry

Course Materials:

1. Strongly Recommended

Textbook: Quantitative Chemical Analysis by Daniel C. Harris - 8th Edition, 2010

2. Required

Lab Manual for CHEM 3430 - available from the Chemistry Department Lab Manual Sales (look for the signs by the labs). The lab manuals are available as a cash only purchase.

Safety Goggles and Lab Coat.

Scientific Calculator.

Laboratory:

Introductory labs are the week of **May 11th**. For the introductory lab, the students should bring with them the copy of the **lab manual only**; they do not need lab coats or goggles. Attendance to the introductory lab is required and students can expect to stay for approximately one hour.

During this time they will meet the TA's, review lab safety procedures, be assigned a lab partner, and get the schedule of experiments so they know which experiment to prepare for the following week.

Course Web Site:

Course and lab materials will be available through <http://www.uoguelph.ca/courselink/> under CHEM*3430 S15 (01) Analytical Chemistry II. Use your university email **id** and **password** to access courselink.

Course Evaluation:

LABORATORY	40%
SEMESTER PROJECT(s)	15 %
IN CLASS DISCUSSIONS	3%
MIDTERM	20%
FINAL EXAM	22%

Midterm will be held in class and will be 75 minutes. Date to be decided with input from the class. Final Exam will be held as determined by the registrar's office

In Class Discussions:

Participation in classroom discussions and interacting with your class colleagues is an important aspect to successful learning. If you really want to learn and succeed in this class, you will want to participate in the questions posed by your lecturer during the class and to ask your own questions. Marks will not be given for correct answers - correct answers are for the exams. Rather, we need to have you honestly participate in the class. To this end, we will provide 3% of your grade to be calculated as follows. There will be many days throughout the semester in which class discussions will be conducted. If you make a contribution to the class discussion on a given day, then it will be taken that you have participated that day. If you participate in at least 85% of the days on which class discussion are conducted you will receive 3 marks towards your final grade. If you participate for at least 60% of the days, you will get 2 marks; at least 35% will earn you 1 mark. It is known that students will do better on their exams if they participate in learning activities and class discussions and it is felt that it is important to offer this to those who are willing to make the effort. To be best prepared to participate you should read the appropriate sections of the textbook and be prepared to discuss them.

Semester Project(s):

More information will be determined during class discussions.

Missed Midterm:

A grade of zero will be assigned for a missed midterm except for valid medical or compassionate reasons. If you are excused for missing the midterm then the final exam will be worth 42% of the course.

COURSE CONTENT

Introduction:

Goals of the course. The process of chemical analysis. Instrumental analytical methods versus classical analytical methods. Selectivity and interferences in trace analysis. Figures of merit in instrumental analysis (Accuracy, Precision, Sensitivity, Dynamic Range, Detection Limits, Selectivity). Calibration methods.

Spectroscopic Methods:

Properties of electromagnetic radiation. Atomic Absorption Spectroscopy (Beer's Law, Instrumental components [light sources, monochromators, detectors]. Atomic Emission Spectroscopy. Flames. Inductively coupled plasmas (ICP). Detector arrays. Comparison of Atomic Absorption and Emission techniques.

Mass spectrometry

Introduction to mass spectrometry (type of ions, isotopes, mass spectra). Ionization methods in atomic and molecular mass spectrometry. Mass spectrometry instrumentation. Applications of gas chromatography-mass spectrometry and liquid chromatography-mass spectrometry. Tandem mass spectrometry.

Separation Methods:

Planar and elution chromatography. The principles of chromatography. Partition coefficients (retention of analytes). Plate theory-van Deemter curves (efficiency of separation). Gas Chromatography (instrumental components and applications). Instrumental components and applications of Liquid Chromatography (normal phase LC, reversed phase LC, ion exchange LC, size exclusion LC). Electroseparations (Capillary Electrophoresis, Gel Electrophoresis, Electrochromatography)

Electroanalytical Methods:

A brief review of electrochemistry. Potentiometric methods. Reference electrodes. Ion selective electrodes. Membrane electrodes. Sensors. Voltammetry and Polarography. Dropping Mercury electrodes. Tast Polarography. Differential Pulse Polarography. Hydrodynamic Voltammetry. Stripping Voltammetry Electrochemical sensors.

CHEM*3430 Summer 2015 Readings Chapters and Sections

0 The Analytical Process 1

The "Most Important" Environmental Data Set of the Twentieth Century 1

0-1 Charles David Keeling and the Measurement of Atmospheric CO₂ 1

0-2 The Analytical Chemist's Job 6

0-3 General Steps in a Chemical Analysis 11

4 Statistics

4-8 Calibration Curves 87

5 Quality Assurance and Calibration Methods 96

The Need for Quality Assurance 96

5-1 Basics of Quality Assurance 97

5-2 Method Validation 100

5-3 Standard Addition 106

5-4 Internal Standards 109

17 Fundamentals of Spectrophotometry 393

The Ozone Hole 393

17-1 Properties of Light 394

17-2 Absorption of Light 395

17-3 Measuring Absorbance 399

17-4 Beer's Law in Chemical Analysis 400

17-6 What Happens When a Molecule Absorbs Light? 404

17-7 Luminescence 408

18 Applications of Spectrophotometry 419

Fluorescence Resonance Energy Transfer Biosensor 419

18-1 Analysis of a Mixture 419

18-2 Measuring an Equilibrium Constant: The Scatchard Plot 424

18-3 The Method of Continuous Variation 425

18-4 Flow Injection Analysis and Sequential Injection 427

18-5 Immunoassays and Aptamers 431

18-6 Sensors Based on Luminescence Quenching 433

19 Spectrophotometers 445

Cavity Ring-Down Spectroscopy: Do You Have an Ulcer? 445

19-1 Lamps and Lasers: Sources of Light 447

19-2 Monochromators 450

19-3 Detectors 454

19-4 Optical Sensors 461

20 Atomic Spectroscopy 479

An Anthropology Puzzle 479

20-1 An Overview 480

20-2 Atomization: Flames, Furnaces, and Plasmas 482

20-3 How Temperature Affects Atomic Spectroscopy 487

20-4 Instrumentation 488

20-5 Interference 493

20-6 Inductively Coupled Plasma–Mass Spectrometry 495

21 Mass Spectrometry 502

Droplet Electrospray 502

21-1 What Is Mass Spectrometry? 502

21-2 Oh, Mass Spectrum, Speak to Me! 507

21-3 Types of Mass Spectrometers 512

21-4 Chromatography–Mass Spectrometry 519

21-5 Open-Air Sampling for Mass Spectrometry 529

22 Introduction to Analytical Separations 537

Measuring Silicones Leaking from Breast Implants 537

22-1 Solvent Extraction 538

22-2 What Is Chromatography? 542

22-3 A Plumber's View of Chromatography 544

22-4 Efficiency of Separation 548

22-5 Why Bands Spread 554

23 Gas Chromatography 565

What Did They Eat in the Year 1000? 565

23-1 The Separation Process in Gas Chromatography 565

23-2 Sample Injection 577

23-3 Detectors 579

23-4 Sample Preparation 584

23-5 Method Development in Gas Chromatography 587

24 High-Performance Liquid Chromatography 595

Paleothermometry: How to Measure Historical Ocean Temperatures 595

24-1 The Chromatographic Process 596

24-2 Injection and Detection in HPLC 611

24-3 Method Development for Reversed-Phase Separations 617

24-4 Gradient Separations 623

24-5 Do It with a Computer 625

25 Chromatographic Methods and Capillary Electrophoresis 634

Capillary Electrochromatography 634

25-1 Ion-Exchange Chromatography 635

25-2 Ion Chromatography 642

25-3 Molecular Exclusion Chromatography 647

25-4 Affinity Chromatography 649

25-5 Hydrophobic Interaction Chromatography 650

25-6 Principles of Capillary Electrophoresis 650

25-7 Conducting Capillary Electrophoresis 657

25-8 Lab-on-a-Chip: Probing Brain Chemistry 665

13 Fundamentals of Electrochemistry 279

Lithium- Ion Battery 279

13-1 Basic Concepts 280

13-2 Galvanic Cells 284

13-3 Standard Potentials 287

13-4 Nernst Equation 288

13-5 E° and the Equilibrium Constant 293

13-6 Cells as Chemical Probes 295

13-7 Biochemists Use E° 297

14 Electrodes and Potentiometry 308

Chem Lab on Mars 308

14-1 Reference Electrodes 309

14-2 Indicator Electrodes 311

14-3 What Is a Junction Potential? 313

14-4 How Ion-Selective Electrodes Work 314

14-5 pH Measurement with a Glass Electrode 317

14-6 Ion-Selective Electrodes 323

14-7 Using Ion-Selective Electrodes 330

14-8 Solid-State Chemical Sensors 331

16 Electroanalytical Techniques 361

How Sweet It Is! 361

16-1 Fundamentals of Electrolysis 362

16-2 Electrogravimetric Analysis 367

16-3 Coulometry 369

16-4 Amperometry 371

16-5 Voltammetry 376

CHEM*3430 Skills

Through the content and concepts presented and the problems discussed, another purpose of this courses is to help you further develop skills that will aid you in your future courses within your program and major as well as beyond. These skills are:

ability to think critically & apply knowledge to new problems (i.e., problem solving skills)

inquiry (www.uoguelph.ca/registrar/calendars/undergraduate/current/c02/sec_d0e396.shtml)

observing and the ability to design a simple experiment

work co-operatively with others and independently

depth and breadth of understanding as well as the capacity to know when you do not understand (www.uoguelph.ca/registrar/calendars/undergraduate/current/c02/sec_d0e403.shtml)

love of learning
(www.uoguelph.ca/registrar/calendars/undergraduate/current/c02/sec_d0e427.shtml)

University Policies

E-mail Communication

As per university regulations, all students are required to check their <mail.uoguelph.ca> e-mail account regularly: e-mail is the official route of communication between the University and its students.

When You Cannot Meet a Course Requirement

When you find yourself unable to meet an in-course requirement because of illness or compassionate reasons, please advise the course instructor (or designated person, such as a teaching assistant) in writing, with your name, id#, and e-mail contact. See the undergraduate calendar for information on regulations and procedures for Academic Consideration.

Drop Date

The last date to drop one-semester courses, without academic penalty, is Friday, July 3. For regulations and procedures for Dropping Courses, see the Undergraduate Calendar.

Copies of out-of-class assignments

Keep paper and/or other reliable back-up copies of all out-of-class assignments: you may be asked to resubmit work at any time.

Accessibility

The University of Guelph is committed to creating a barrier-free environment. Providing services for students is a shared responsibility among students, faculty and administrators. This relationship is based on respect of individual rights, the dignity of the individual and the University community's shared commitment to an open and supportive learning environment. Students requiring service or accommodation, whether due to an identified, ongoing disability or a short-term disability should contact the Centre for Students with Disabilities as soon as possible.

For more information, contact CSD at 519-824-4120 ext. 56208 or email csd@uoguelph.ca or see the website: <http://www.uoguelph.ca/csd/>

Academic Misconduct

The University of Guelph is committed to upholding the highest standards of academic integrity and it is the responsibility of all members of the University community – faculty, staff, and students – to be aware of what constitutes academic misconduct and to do as much as possible to prevent academic offences from occurring. University of Guelph students have the responsibility of abiding

by the University's policy on academic misconduct regardless of their location of study; faculty, staff and students have the responsibility of supporting an environment that discourages misconduct. Students need to remain aware that instructors have access to and the right to use electronic and other means of detection.

Please note: Whether or not a student intended to commit academic misconduct is not relevant for a finding of guilt. Hurried or careless submission of assignments does not excuse students from responsibility for verifying the academic integrity of their work before submitting it. Students who are in any doubt as to whether an action on their part could be construed as an academic offence should consult with a faculty member or faculty advisor.

The Academic Misconduct Policy is detailed in the Undergraduate Calendar.

Recording of Materials

Presentations which are made in relation to course work—including lectures—cannot be recorded or copied without the permission of the presenter, whether the instructor, a classmate or guest lecturer. Material recorded with permission is restricted to use for that course unless further permission is granted.

Resources

The Academic Calendars are the source of information about the University of Guelph's procedures, policies and regulations which apply to undergraduate, graduate and diploma programs.

Special Thanks to

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the past instructors for this course who generously lent their notes from past semesters.