SAGRADO
Universidad del Sagrado Corazón

SCHOOL OF HEALTH AND SCIENCES

SYLLABUS

TITLE:	Chemistry: Instrumental Analysis
CODE:	QUI 455
PREREQUISITE:	QUI 205
CORREQUISITE:	QUI 455L
CREDITS:	4 credits | 45 contact hours | 45 lab hours | 1 term

DESCRIPTION

Course aimed at Chemistry major students that discusses the basic principles of analytical instruments and their applications. The course covers the general aspects of conventional instrumental analysis techniques used in industrial and research laboratories. It also covers essential aspects of theory, instrumentation, and practical applications of spectroscopy, electroanalysis, and chromatography. Students will develop skills to validate an analytical method; handle instrumental analytical equipment to perform reliable quantitative chemical analysis; and learn the ethical aspects related to the handling of data in the laboratory and the reporting of the results of a chemical analysis. The course also promotes the student's autonomy in bibliographic searches; the critical analysis of scientific articles related to different areas of instrumental chemical analysis; and the development of an innovation and/or entrepreneurship project based on the critical analysis of scientific articles related to the different areas of Analytical Chemistry. The course incorporates laboratory experiences to apply theoretical knowledge by performing reliable quantitative chemical analysis, and is developed through lectures, laboratories, simulations, incorporation of web-supported technology, writing and defense of an entrepreneurship and/or innovation project, bibliographic research, and the reporting of results of a chemical analysis.

JUSTIFICATION

The development of modern science increasingly interrelates the different areas of knowledge. The complete mastery of chemical analysis methods, as well as the acquisition of the typical operational techniques to carry out such analyses, are essential for the proper characterization of a chemical system under study. In industrial laboratories such as those of large pharmaceutical companies, analytical instruments are used on a daily basis to perform a series of chemical analyses that are required by federal regulatory agencies such as the FDA and EPA. By means of these instruments, analyses can be carried out in large quantities and in a reasonably short time. Therefore, a student who aspires to major in chemistry needs a basic knowledge of the theory and operation of analytical instruments. The course's contribution to the student's profile is that he or she acquires the necessary skills to carry out a chemical analysis, and/or develop and implement an entrepreneurship and/or innovation project. In addition, the student also learns how to write a report while maintaining an ethical commitment to the integrity of the analysis carried out and the result obtained. This course is a requirement for the bachelor's degree in Chemistry. It meets the requirement to take the board exam for the Chemistry license as required by law in the Commonwealth of Puerto Rico.

COMPETENCES

The course develops the following competences in students:

- **Critical questioning**
- **Innovation and entrepreneurship**
- **Research and exploration**
- **Ethical sense and social justice**

OBJECTIVES

After completion of the course, students will be able to:

1. Critically analyze an analytical method.
2. Explain the basic theory related to the operation of an analytic instrument
3. Explain the theory and instrumentation of spectroscopic techniques.
4. Develop the theory and instrumentation of chromatographic and electrophoretic techniques.
5. Explain the theory and instrumentation of electrochemical techniques.
6. Design analytical instruments based on the coupling of mass spectrometry with spectroscopic and chromatographic techniques.

CONTENTS

I. Instrumental Chemical Analysis Method
    A. Purpose of instrumental chemical analysis
    B. Action plan for a chemical analysis
        1. Method selection
        2. Sampling

        a. Effective sampling
        b. Models
        c. Mixtures
        d. Sample integrity
        e. Separations
        f. Number of samples
    3. Sample processing
        a. Sample preparation
        b. Recovery
        c. Optimization
        d. Interference
        e. Preconcentration
    4. Analytical signal measurement
        a. Definitions
        b. Types of signals
        c. Primary types
        d. Blanks
        e. Error determination
    5. Evaluation of results
        a. Response to concentration ratio
        b. External calibration curves
        c. Standard addition calibration curves
        d. Internal standard calibration curves
    6. Results reports
    7. Inadequate analytical methodologies
    8. Figures of merit of an analytical method
    9. Interpretation of the results
C. Statistics
    1. Errors in measurements
        a. Determinate errors
        b. Indeterminate errors
    2. Normal distribution curve
        a. Curve shape

        b. Properties
    3. Mean, standard deviation, variance
    4. Linear regression
    5. T & F Tests
    6. Control chart
  D. Sample size
    1. Classifications
    2. Trace, ultra-trace, and micro-analysis
    3. Sample types

II. The Analytical Instrument
  A. General schematic design of an analytical instrument
    1. Input signal
    2. Sample holder
    3. Output signal
    4. Transducer
    5. Signal processor
    6. Recorder
  B. Analog and digital electronics
    1. Charge
    2. Voltage
    3. Electric field
    4. DC current
    5. Ohm's Law
        a. Serial circuits
        b. Parallel circuits
    6. AC current
    7. Capacitance, RC circuits
    8. Magnetic field
    9. Inductance, RL circuits
    10. Solid-state electronic components and circuits
        a. Semiconductors
        b. Diodes
        c. Transistors

        d. Transformers
        e. Voltage sources
    11. Operational amplifiers (op-amp)
        a. Operation
        b. Adder
        c. Differentiators
        d. Integrators
        e. Voltage trackers
        f. Applications
    12. Digital electronics
        a. Logic circuits
        b. Applications
    13. Records
    14. Computers
  C. Noise
    1. Definition
    2. Types of noise
        a. Chemical
        b. Electronic
        c. Environmental
    3. S/N ratio
        a. Baseline
        b. Signal amplitude
        c. Noise amplitude
    4. Sensitivity
        a. Definition
        b. Calibration curve
        c. Analytical sensitivity
    5. Limit of detection
        a. Definition
        b. Determination
    6. Limit of quantification

III. Spectroscopic Methods

A. Introduction
    1. Electromagnetic radiation (REM) and Its properties
    2. Jablonski diagram
        a. Electronic levels
        b. Vibrational levels
        c. Rotational levels
        d. Singlet and triplet states
        e. Intersystemic crossover
        f. Fluorescence and phosphorescence
    3. Beer-Lambert's Law
    4. Schematic design of a photometer and spectrophotometer
    5. REM UV and VIS sources
        a. W lamp
        b. D_2 lamp
    6. Filters and monochromators
        a. Absorption filters
        b. Interference filters
        c. Prism monochromators
        d. Grid monochromators
    7. REM detectors
        a. Phototube
        b. Photomultiplier
        c. Photodiode array (PDA)
    8. Molecular UV-VIS spectrophotometry applications
B. Infrared (IR) spectroscopy
    1. IR Absorption Theory
        a. Vibrational degrees of freedom
        b. Vibrational states
        c. Vibrational modes
        d. Harmonic oscillator
    2. Instrumentation
        a. Sources
        b. Cells

        c. Detectors
        d. Response
        e. Schematic design
    3. Raman Spectroscopy Theory
    4. Applications
C. Nuclear magnetic resonance spectroscopy (NMR)
    1. NMR Absorption Theory
        a. Nuclear Spin
        b. Energy states
        c. Radiofrequency
        d. Chemical displacement
    2. Instrumentation
        a. Sources
        b. Cells
        c. Magneto
        d. Detectors
        e. Response
        f. Schematic design
    3. NMR of ^{1}H, ^{13}C, and other cores
    4. Two-dimensional NMR
        a. NOESY
        b. COSY
    5. Applications
D. Atomic spectroscopy
    1. Atomic structure and electronic transitions
        a. Electronic configuration
        b. Electronic states
        c. Permitted transitions
        d. Atomic emission spectra
    2. Atomic absorption (AA): Instrumentation
        a. Hollow cathode tube
        b. Total consumption burner
        c. Premix burner

        d. Flame temperature and profile
        e. Graphite furnace
        f. Detectors
        g. Schematic design
        h. Applications
    3. Atomic emission (AES): Instrumentation
        a. Sources: Flame (AFS)
        b. Sources: Electric shock
        c. Schematic design
        d. Detectors
        e. Applications
    4. Plasma-Atomic Emission (ICP): Instrumentation
        a. Composition of a plasma
        b. Schematic design
        c. Detector
        d. Applications

E. Molecular fluorescence spectroscopy
    1. Variables affecting fluorescence
        a. Quantum performance
        b. Types of fluorescence electron transitions
        c. Quantum efficiency
        d. Molecular structure and rigidity
        e. Temperature
        f. Solvent
        g. pH
        h. Dissolved O_2
    2. Fluorescence as a function of concentration
    3. Excitation, fluorescence, and phosphorescence spectra
    4. Instrumentation
        a. Sources
        b. Monochromators
        c. Schematic design
        d. Detectors

5. Applications

F. Surface analysis techniques
   1. Introduction
   2. X-ray photoelectron spectroscopy (XPS or ESCA)
      a. Source
      b. Monochromators
      c. Schematic design
      d. Detectors
   3. Atomic Force Microscopy (AFM)
      a. Sources
      b. Monochromators
      c. Schematic design
      d. Detectors
   4. Applications

IV. Chromatographic and Electrophoretic Methods

A. Chromatography theory
   1. Definition
   2. Stationary phase
   3. Mobile phase
   4. Theoretical plates
   5. Resolution
   6. Selectivity factor
   7. Capacity factor
   8. Van Deemter Equation

B. Gas chromatography (GC)
   1. Gases
   2. Sample injection systems
   3. Packed and capillary columns
   4. Temperature programming
   5. Oven
   6. Detectors: TCD, ECD
   7. Applications

C. High-Performance Liquid Chromatography (HPLC)

1. Schematic design
2. Flow pumps
3. Injection system
4. Columns
5. UV-VIS detectors
6. Isocratic and gradient elution
7. Applications

D. Supercritical Fluid Chromatography (SFC)
   1. Supercritical Fluid Theory
   2. Injection systems
   3. Columns
   4. CO_2 and modifiers
   5. Oven
   6. Detectors
   7. Applications

E. Two-Dimensional Chromatography
   1. Experimental design of two-dimensional chromatography
   2. Applications

F. Capillary electrophoresis
   1. Instrumentation
   2. Electroosmotic Flow (EOF)
   3. Modalities: zone, gel, MEKC and isotachophoresis
   4. Detectors
   5. Applications

V. Electrochemistry

A. Components of a galvanic cell
   1. Electrodes
   2. Salt bridge
   3. Meter
   4. Current flow
   5. Polarity
   6. Cell potential
   7. Galvanic and electrolytic cells

B. The standard potential
    1. Definition
    2. Normal hydrogen electrode
    3. Determination of E^o
    4. Activity series
C. Nernst's equation
    1. Variables in that equation
    2. Computation of the potential of an electrode
D. Reference electrodes
    1. Calomel saturated electrode
    2. Ag/AgCl electrode
E. Circuit of a potentiostat
F. Voltammetry in general
    1. Theory
    2. Applications
G. Differential pulse voltammetry
    1. Theory
    2. Applications
H. Square wave voltammetry
    1. Theory
    2. Applications
I. Anodic clearance voltammetry
    1. Theory
    2. Applications

VI. Mass Spectrometry (MS) and Couplings
A. Fundamentals of MS
    1. General schematic design
    2. Ionization modes
    3. Ion separators
    4. M/z ratio
B. Tandem mass spectrometry (MS/MS)
    1. General schematic design
    2. SIM

        3. Applications
    C. CG-MS
        1. Interfaces
        2. Applications
    D. HPLC-ESI-MS
        1. Interface
        2. Applications
    E. ICP – MS
        1. Interface
        2. Applications
    F. CE – MS
        1. Interface
        2. Applications

LAB EXPERIENCES

A. Introduction to the Instrumental Chemical Analysis Laboratory
B. Ideation of the entrepreneurship project
C. Sampling
D. Analog and digital electronics
E. Calibration curves
F. Determination of acetic acid percentage using the Gran Method
G. Determination of %Mn in steel
H. Validation of an analytical method
I. Aqueous glucose solution analysis
J. Analytic separations
K. Presentation and defense of the entrepreneurship project

METHODOLOGY

The following strategies from the active learning methodology are recommended:

- Simulations
- Written reports
- Solving a problem posed
- Lectures

- Web-supported education
- Laboratories
- Literature review
- Oral defense of innovation and/or entrepreneurship project
- Rubrics of lab execution and oral defense of the entrepreneurship project

EVALUATION

Partial assignments	20%
Immersion experience	25%
Oral presentation	30%
Final project or exam	25%
Total	**100%**

LEARNING ASSESSMENT

The institutional assessment rubric is applied to the course's core activity.

BIBLIOGRAPHY

TEXTBOOK

Skoog, D. A., West, D. M., Holler, F. J., & Crouch, S. R. (2017). *Principles of Instrumental Analysis* (9th ed.). Cengage Learning.

REFERENCES

Andrade-Garda, J. M., Carlosena-Zubieta, A. (2017). *Problems of Instrumental Analytical Chemistry:A Hands-On Guide (Essential Textbooks in Chemistry)*. World Scientific Publishing Company.

Anjaneyulu, Y., Chandrasekhar, K. (2019). *A Textbook of Analytical Chemistry*. BSP BOOKS.

Basha, M. (2019). *Analytical Techniques in Biochemistry* (Springer Protocols Handbooks). Humana.

Barbooti, M. (2015). *Environmental Applications of Instrumental Chemical Analysis.*

Apple Academic Press.

Braunl, R. D. (2016). *Introduction to Instrumental Analysis* (2nd ed.). Pharma Med Press.

Campbell, L., Turner, W. (2015). *Standard Methods of Chemical Analysis: Instrumental Methods*. (2nd ed.). Arcler Press LLC.

Farsalinos, K. E., Gillman, I. G., Hecht, S. S., Polosa, R., & Thornburg, J. (2016). *Analytical Assessment of e-Cigarettes: From Contents to Chemical and Particle Exposure Profiles (Emerging Issues in Analytical Chemistry)*. Elsevier.

Ham, B. M., MaHam, A. (2015). *Analytical Chemistry: A Chemist and Laboratory Technician's Toolkit*. Wiley.

Ngambeki, W. (2019). *Introduction to Analytical Chemistry for University Students*. Tanzania Educational Publishers Ltd.

White, R. M., Moore, C. M. (2018*). Detection of Drugs and Their Metabolites in Oral Fluid (Emerging Issues in Analytical Chemistry)*. Elsevier

ELECTRONIC RESOURCES

American Chemical Society (n.d.) ACS Chemistry for Life. https://www.acs.org/content/acs/en.html

International Union of Pure and Applied Chemistry. (n.d.). https://iupac.org/

For more information resources related to the course's topics, access the library's webpage http://biblioteca.sagrado.edu/

REASONABLE ACCOMMODATION

For detailed information on the process and required documentation you should visit the corresponding office. To ensure equal conditions, in compliance with the ADA Act (1990) and the Rehabilitation Act (1973), as amended, any student in need of reasonable accommodation or special assistance must complete the process established by the Vice Presidency for Student Affairs.

- Students participating in the Student Support Program (PAE, in Spanish) shall request their reasonable accommodation in PAE's offices.
- Students who do not participate in PAE shall request their reasonable accommodation at the Integral Wellness Center (*Centro de Bienestar Integral*, in Spanish).

ACADEMIC INTEGRITY

This policy applies to all students enrolled at Universidad del Sagrado Corazón to take courses with or without academic credit. A lack of academic integrity is any act or omission that does not demonstrate the honesty, transparency, and responsibility that should characterize all academic activity. Any student who fails to comply with the Honesty, Fraud, and Plagiarism Policy is exposed to the following sanctions: receive a grade of zero in the evaluation and / or repetition of the assignment in the seminar, a grade of F (*) in the seminar, suspension, or expulsion as established in the Academic Integrity Policy effective in November 2022.

RESEARCH COURSES

This course may require students to practice tasks related to the research process, such as taking informed consent or assent, administering instruments, conducting interviews, observations, or focus groups, among others. These assignments are part of an academic exercise and the information collected will not be used to share with third parties or disclose it in settings other than the classroom with the professor teaching the course. Every student, as well as their professor, who will interact with human subjects as part of their research practice must be certified in ethics with human subjects in research by the Collaborative Institutional Training Initiative (CITI Program).