

**INTERAMERICAN UNIVERSITY OF PUERTO RICO**  
**CAMPUS \_\_\_\_\_**  
**DEPARTMENT OF \_\_\_\_\_**  
**CHEMISTRY PROGRAM**

**SYLLABUS**

**I. INFORMACION GENERAL**

Course Title	:	Instrumental Analytical Chemistry
Code and Number	:	CHEM 4240
Credits	:	5 credits
Academic term	:	
Professor	:	
Office hours	:	
Office phone	:	
Email	:	

**I. DESCRIPTION**

Study of the components, fundamentals and applications of typical instrumentation used for separation, identification, and quantitative analysis of chemical substances. It includes spectroscopic, chromatographic, and electrochemical techniques. Emphasis on optimization, calibration, and validation methods commonly used in instrumental analysis. Discussion of the strengths and limitations of the different analysis methods and techniques. It requires 45 hours of conference and 75 hours of closed laboratory face-to-face. Requirements: CHEM 3230, 3320 and 3330.

**II. TERMINAL OBJECTIVES**

It is expected that, upon completion of the course, the student will be able to become familiar with the fundamental concepts of instrumental analysis to:

1. Develop skills so that the student can select the most appropriate instrumental technique for a chemical analysis.
2. To develop the skills so that the student can select the most suitable conditions for an instrumental chemical analysis.
3. Develop skills so that the student can apply statistical techniques for the interpretation and treatment of experimental data.
4. Apply ethical principles in solving problems of quantitative chemical analysis, both at a theoretical and practical level.

## **COMPETENCES OF THE GRADUATE PROFILE ATTENDED IN THIS COURSE**

1. Apply descriptive and inferential statistics in the analysis of experimental results.
2. Correctly perform the most characteristic calculations of chemistry, such as concentrations.
3. Know the principles, applications, advantages and limitations of the instruments and procedures most used in chemistry.
4. Show willingness to work actively in a team and in interdisciplinary projects.
5. Show good disposition towards the use of new technologies and scientific developments, including the integration of the computer into analysis.
6. Raise awareness about the ethical and cultural values necessary for the practice of the profession.

### **III. TRAINING OBJECTIVES**

#### **1. Statistical Treatment of Analytical Data**

Upon concluding the study of this topic, the student should be able to:

- a. Apply the definition of error and explain its relationship to accuracy.
- b. Recognize and classify the determined and indeterminate errors associated with the analytical method, the instrumentation, and the analyst who performs the measurement.
- c. Explain the effect of the determined constant or proportional errors on the relative error of the final analysis result.
- d. Identify ways to correct, compensate or eliminate the determined errors of the analytical method, applying ethical principles in the process.
- e. Apply the definition of standard deviation and explain its relationship to precision.
- f. Apply the least squares method to determine the slope and intercept of the regression line, linear correlation coefficient, standard deviation of the slope, standard deviation of the regression, and standard deviation of the interpolated value.

#### **2. Preparation of solutions and application of volumetric methods of analysis**

Upon concluding the study of this topic, the student should be able to:

- a. Describe the preparation of solutions in units of % mass, % volume, % mass / volume, parts per million, Molarity, and analytical concentration.
- b. Describe the change in units of concentration from one unit to another including change from mass% to Molarity, parts per million to Molarity, and vice versa.
- c. Apply the concepts of analyte, titrant, standard solution, standardization or titration, equivalence point and end point, in titrimetric analysis problems.

- d. Calculate the content or concentration of analyte in a sample from experimental data obtained in a volumetric analysis.
- e. Apply the concept of sample dilution and that of titration of an aliquot of sample solution to problems of concentration and content determination
- f. Distinguish between direct, indirect, and back-titling, and solve quantitative problems.

### 3. Calibration Methods

Upon concluding the study of this topic, the student should be able to:

- a. Describe and calculate the limit of detection and limit of quantization in terms of concentration and instrumental signal.
- b. Describe and calculate the calibration sensitivity and linear calibration range.
- c. Describe and calculate the signal-to-noise ratio "S/N" and explain its relationship with the detection limit.
- d. Describe the different types of noise that affect the instrumental signal.
- e. Describe and apply the external standard calibration method, its procedure, sample matrix considerations, its advantages, and limitations.
- f. Describe and apply the standard addition calibration method, its algebraic derivation, its advantages, and limitations.
- g. Describe and apply the internal standard calibration method, the chemical-physical characteristics of the internal standard, its advantages, and limitations.
- h. Métodos de Calibración

### 4. Instruments for Optical Spectroscopy

Upon concluding the study of this topic, the student should be able to:

- a. Mention the different types of radiation source for optical instruments.
- b. Describe the different types of wavelength selectors including absorbance and diffraction filters, prism monochromators, and diffraction grating.
- c. Describe the effective bandwidth of the different wavelength selectors.
- d. Describe and compare the scattering power and spectral purity of the prism monochromator and the diffraction grating.
- e. Describe the characteristics of the different sample containers used in optical spectroscopy.
- f. Describe the phototube and photomultiplier tube detectors, their mode of operation, sensitivity, advantages, and limitations.

## 5. Molecular Absorption Methods in the Ultraviolet-Visible Region

Upon concluding the study of this topic, the student should be able to:

- a. Describe the radiation absorption experiment and define the transmittance and absorbance of a solution.
- b. Define Beer's law and each of its variables.
- c. Define the variables that affect absorbance and absorptivity.
- d. Describe the external standard calibration method for determining one or more components in a sample.
- e. Describe the procedure to perform a spectrophotometric titration and predict the shape of the titration curve using the spectral information of the analyte, titrant, and reaction product.
- f. Describe chemical and instrumental deviations from calibrations and ways to minimize or control them.
- g. Describe the one-ray and two-ray instruments and mention their advantages and limitations.
- h. Describe the sources of radiation for UV-Vis absorption instruments.
- i. Describe the most important electronic transitions observed in UV-visible.
- j. Describe how double bond conjugation affects the spectrum of organic compounds.
- k. Describe the effect of substituents on the spectrum of aromatic compounds.

## 6. Fluorescence, Phosphorescence and Chemiluminescence Methods

Upon concluding the study of this topic, the student should be able to:

- a. Describe the electronic state singlet, excited singlet, and excited triplet.
- b. Describe the mechanisms of deactivation of the excited state, including vibrational relaxation, internal and external conversion, crossover between systems, resonant and non-resonant fluorescence, and phosphorescence.
- c. Describe the effect of the heavy atom on fluorescence and phosphorescence.
- d. Describe the variables that affect fluorescence, including temperature, solvent, quantum yield, degree of unsaturation, and analyte geometry.
- e. Describe the chemiluminescence process and its advantages for quantitative analysis.
- f. Describe the relationship between concentration and fluorescence or phosphorescence and discuss the effect of self-absorption and self-quenching.
- g. Describe the qualitative, quantitative, and operational aspects of molecular emission techniques.
- h. Describe the components and organization of spectrofluorometers.

## 7. Methods of Atomic Absorption in Flame

Upon completion of the study of this topic, the student should be able to:

- a. Describe the characteristics of the atomic emission spectrum, atomic absorption.
- b. Describe the different types of flames, the laminar flow burner, and the safety measures necessary for its operation.
- c. Describe the process of nebulization, desolvation, sublimation, atomization, and thermal excitation.
- d. Describe the components of the atomic absorption instrument, including the geometry and operation of the hollow cathode lamp.
- e. Describe the main chemical interferences in solution and in the gas phase and the methods used to minimize them.
- f. Establish the methodology to perform a quantitative analysis, including flame selection, burner height, lamp current, and monochromator alignment.
- g. Describe the electrothermal atomization technique, its advantages and disadvantages.
- h. Describe the most common calibration methods and explain deviations from linearity.

## 8. Column Chromatography Theory

Upon concluding the study of this topic, the student should be able to:

- a. Describe the mechanism of column separation by elution and define stationary phase, mobile phase, eluent, eluate, chromatogram, retention volume, dead volume of the column, capacity factor, selectivity factor, resolution of two substances, and efficiency of column.
- b. Describe the general elution problem and describe its solution in terms of a temperature gradient, or gradient in the composition of the mobile phase.
- c. Describe the general problem of solving two compounds and describe its solution in terms of increase in column efficiency ( $N$ ), selectivity ( $\alpha$ ), or capacity factor ( $k'$ ).
- d. Describe the general detection problem and describe its solution in terms of detector selectivity, analyte derivatization, or indirect detection.
- e. Describe the most common calibration methods used in chromatography and evaluation of the purity of the bands.

## 9. Gas Chromatography

Upon concluding the study of this topic, the student should be able to:

- a. Describe the different modalities of gas chromatography.
- b. Describe the Van Deemter graph and relate it to the Van Deemter equation.
- c. Describe the applicability and limitations of gas chromatography.
- d. Describe the components and organization of the gas chromatograph.

## 10. Liquid Chromatography

Upon concluding the study of this topic, the student should be able to:

- a. Describe the different modalities of liquid chromatography including normal phase, reverse phase, ion exchange, size exclusion, and ion pair formation
- b. Describe the application of the Van Deemter equation to liquid chromatography.
- c. Describe the different types of bonded phase and their applications.
- d. Describe the components and organization of a high-pressure liquid chromatography (HPLC) instrument.
- e. Describe the variation of the capacity factor of an analyte as a function of the polarity of the mobile phase and the length of the carbon chain in a reversed phase column.

## 11. Mass Spectrometry Methods

Upon concluding the study of this topic, the student should be able to:

- a. Describe the components and organization of a mass spectrometer with electron ionization and chemical ionization.
- b. Describe the application of mass spectrometry in quantitative analysis including hybrid chromatography and mass spectrometry methods.
- c. Describe the advantages and limitations of mass spectrometry applied to quantitative analysis.

## 12. Potentiometric Methods

Upon concluding the study of this topic, the student should be able to:

- a. Describe the different types of indicator electrode used for quantitative analysis.
- b. Describe the most common calibration methods and their applications.
- c. Describe the advantages and limitations of potentiometric methods.

## 13. Infrared Spectroscopy Methods

Upon concluding the study of this topic, the student should be able to:

- a. Describe the general theory of infrared spectroscopy and locate the most common bands applied to quantitative analysis.
- b. Describe the components and organization of an infrared spectrophotometer.
- c. Describe the different types of instrument manipulation and calibration.
- d. Describe the applications for quantitative analysis, the advantages, and the limitations.

#### IV. ACTIVITIES

##### A. Laboratory Practices

Exp. #	Experiment Title
1	Spectrophotometric determination of phosphorus in cola drinks
2	Potentiometric determination of the amount of active ingredient in a commercial antacid
3	Determination of lead in unknown samples
4	Simultaneous determination of dyes in commercial beverages by Visible Spectroscopy
5	Gas Chromatography (GC). Theoretical experiment (subject to change)
6	Quantification of caffeine by spectrophotometry

##### B. Teaching Strategies

It is recommended to use strategies such as the following:

- a. Troubleshooting
- b. Use of the calculator and computer programs for data processing
- c. Group work
- d. Laboratory experiences
- e. Questions to the group
- f. Multiple choice exams and problem solving.

#### V. EVALUATION

Evaluation criteria	Punctuation	% of the final grade
Partial 1	100	15
Partial 2	100	15
Parcial 3	100	15
Final exam	100	15
Oral presentation	100	10
Laboratory	100	30
Total	600	100 %

To establish the final grade for the course, the one that appears below will be used:

<b>A</b>	<b>B</b>	<b>C</b>	<b>D</b>	<b>F</b>
100-85	84-75	74-65	64-55	54-0

## VI. SPECIAL NOTES

- A. **Auxiliary services or special needs:** All students who require auxiliary services or special assistance must request them at the beginning of the course or as soon as they become aware that they need them, through the corresponding registry, in the Guidance Office with the \_\_\_\_\_.
- B. **Honesty, fraud, and plagiarism:** Dishonesty, fraud, plagiarism, any other inappropriate behavior in relation to academic work constitute major infractions sanctioned by the General Student Regulations. Major offenses, according to the General Student Regulations, may result in suspension from the University for a defined period of more than one year or permanent expulsion from the University, among other sanctions.
- C. **Use of electronic devices:** Cell phones and any other electronic device that could interrupt the teaching and learning processes or alter the environment conducive to academic excellence will be disabled. Urgent situations will be addressed, as appropriate. The handling of electronic devices that allow accessing, storing, or sending data during evaluations or exams is prohibited.
- D. **Compliance with the provisions of Title IX:** The Federal Higher Education Law, as amended, prohibits discrimination based on sex in any academic, educational, extracurricular, athletic activity or in any other program or employment, sponsored or controlled by an institution of Higher education regardless of whether it is carried out on or off the premises of the institution, if the institution receives federal funds.

In accordance with current federal regulations, our academic unit has appointed an Assistant Title IX Coordinator who will aid and guidance in relation to any alleged incident that constitutes discrimination based on sex or gender, sexual harassment, or assault. You can contact the Assistant Coordinator \_\_\_\_\_, extension \_\_\_\_\_, or email \_\_\_\_\_.

The Normative Document entitled **Norms and Procedures to Address Alleged Violations of the Provisions of Title IX** is the document that contains the institutional rules to channel any complaint that is presented based on this type of allegation. This document is available on the website of the Inter American University of Puerto Rico ([www.inter.edu](http://www.inter.edu)).



## VII. RECURSOS EDUCATIVOS

- A. Textbook: Principles of Instrumental Analysis Sixth Edition, Douglas A. Skoog, F. James Holler and Stanley R. Crouch, D.R. 2008 por Cengage Learning Editores, S.A.de C.V., a Company of Cengage Learning, Inc.  
ISBN-13:978-607-481-390-6  
ISBN-10:607-481-390-6
- B. Laboratory Manual: Prepared by the Department
- C. Supplemental reading:
1. Campbell, Oswald L.; "Investigation of experimental parameters"; J.Chem.Educ.; 68(1991)784.
  2. Cohen, Ruben D.; "Why do random samples represents populations so accurately?"; J.Chem.Educ.; 68(1991)902.
  3. Lisesnky, George, Reynolds, Kelly; "Chloride in Natural Waters. An environmental application of a potentiometric titration."; J.Chem.Educ.; 68(1991)334.
  4. Sherren, Anne T. "The use of real-life samples for unknowns in analytical chemistry"; J.Chem.Educ.; 68(1991)598.
  5. Abel, Kenton B., Hemmerlin, William M.; "Significant Figures"; J.Chem.Educ.; 67(1990) 213.
  6. Burness, James H.; "An efficient method for the treatment of weak acid/base equilibria"; J.Chem.Educ.; 67(1990)224.
  7. Davis, Edith; "A revised approach to solving redox equations"; J.Chem.Educ.; 67(1990)671.
  8. Ewing, Golen W.; "Safety in the Analytical Laboratory"; J.Chem.Educ.; 67(1990) A158.
  9. Herman, D.P.; Booth, K.K.; Parker, O.J.; Breneman, G.L.; "The pH of any mixture of monoprotic acids and bases"; J.Chem.Educ.; 67(1990)501.
  10. King, D.Whitney; Kester, Dana R.; "A general approach for calculating poliprotic acid speciation and buffer capacity"; J.Chem.Educ.; 67(1990)932.
  11. Logier, Claudia; Olivieri, Alejandro; "Calculation of solubilities of Carbonates and Phosphates in water as influenced by competitive acid-base reactions."; J.Chem.Educ. 67 (1990)934.
  12. Larson, John W.; "Error analysis in spectrophotometric determinations and the environmental consequences of a reduction in the ozone layer"; J.Chem.Educ.; 67(1990)943.
  13. Sharpe, Alan G.; "The solvation of halide ions and its chemical significance" J.Chem.Educ. 67 (1990)309.
  14. Thompson, Ralph J.; "The extent of acid-base reactions"; J.Chem.Educ.; 67(1990)220.

15. Dean, R.B.; Dixon, W.J.; "Simplified Statistics for small number of observations."; *Anal.Chem.*; 23(1951)636.
16. Gorin, G.; "Indicators and the basis for their use"; *J.Chem.Educ.*; 33(1956)319.
17. Mac Alpine, R.K.; "Change in pH at the equivalence point"; *J.Chem.Educ.*; 25(1948)694.
18. Bard, A.J.; Simpsons, S.H.; "The general equation for the equivalence point potential in oxidation-reduction titrations"; *J.Chem.Educ.*; 37(1960)364.
19. Pungor, E.; "Theory and applications of anion selective membrane electrode"; *Anal.Chem.*; 39(13)1967 28A.

### VIII. BIBLIOGRAPHY

1. Kenkel, John, *Analytical Chemistry for Technicians*, 3rd Edition, CRC Press, 2003. ISBN: 1-5667-0519-3
2. Sawyer, Donald T. and William R. Heineman, *Chemistry Experiments for Instrumental Methods*, 2nd Edition, John Wiley, 2002. ISBN: 0-471-21496-5
3. Rouessac, Francis and Annick Rouessac, *Chemical Analysis: Modern Instrumental Methods and Techniques*, 1st Edition, John Wiley, 2002. ISBN: 0-471-97261-4
4. Rubinson, Kenneth A. and Judith F. Rubinson, *Contemporary Instrumental Analysis*, 1st Edition, Prentice Hall, 2000. ISBN: 0-13-790726-5
5. Robinson, James W., *Undergraduate Instrumental Analysis*, 5th Edition, Marcel Dekker, 1994. ISBN: 0-8247-9215-7

**Revision Date: November 2018**