

Course Unit	Instrumental Methods of Analysis		Field of study	Chemistry	
Bachelor in	Chemical Engineering		School	School of Technology and Management	
Academic Year	2022/2023	Year of study	2	Level	1-2
Type	Semestral	Semester	1	ECTS credits	6.0
				Code	9125-755-2102-00-22
Workload (hours)	162	Contact hours	T -	TP 24	PL 36
			TC -	S -	E -
			OT -	O -	

T - Lectures; TP - Lectures and problem-solving; PL - Problem-solving, project or laboratory; TC - Fieldwork; S - Seminar; E - Placement; OT - Tutorial; O - Other

Name(s) of lecturer(s) António Manuel Esteves Ribeiro

### Learning outcomes and competences

At the end of the course unit the learner is expected to be able to:

1. Describe the steps involved on chemical analysis and know sampling and sample preparation techniques.
2. Apply statistic analysis to experimental data including regression analysis for producing calibration curves.
3. Understand theoretical background and recognize/operate instrumentation for potentiometric and conductivimetric analysis, ion-selective electrodes measurements.
4. Understand theoretical background and recognize/operate instrumentation devoted to spectroscopic analysis: Ultraviolet-visible (UV-VIS) and Infra-Red (FT-IR) spectroscopy.
5. Understand theoretical background and recognize/operate instrumentation devoted to chromatographic processes: Gas Chromatography (GC) and High Performance Liquid Chromatography (HPLC).
6. Develop laboratorial skills by executing a set of experimental protocols and perform experimental data treatment and write technical reports.

### Prerequisites

Before the course unit the learner is expected to be able to:

Handle general laboratory material, demonstrates background knowledge of General Chemistry.

### Course contents

Introduction to Instrumental Methods of Analysis. Errors in Chemical Analysis. Potentiometric methods. Introduction to Chromatographic methods. Gas Chromatography (GC). High Performance Liquid Chromatography (HPLC). Introduction to spectroscopy. UV-VIS and FT-IR spectroscopy.

### Course contents (extended version)

1. Fundamentals of Instrumental Methods of Analysis
  - Classification and characteristics;
  - Analytical method: selection and validation;
  - Preliminary operations of chemical analysis.
2. Errors in Chemical Analysis
  - Types of errors in classical chemical analysis;
  - Mean and standard deviation;
  - Error distribution: sample, population, Gauss distribution;
  - Distribution of sample mean: central limit theorem;
  - Confidence intervals for mean: the t-Student distribution;
  - Parametric tests: data comparison;
  - Calibration curves on Instrumental Methods of Analysis;
  - Data rejection.
3. Potentiometric Methods
  - Galvanic cell;
  - Reference electrodes;
  - Oxidation-reduction electrodes;
  - Membrane electrodes;
  - Analytical methods in potentiometry;
  - Ion-selective electrodes.
4. Introduction to Chromatographic Methods
  - Concept of chromatography and classification of chromatographic methods;
  - Basic concepts of operation;
  - Selectivity, efficiency and resolution;
  - Column efficiency measurements: (HETP) and number of plates;
  - Calibration methods: external standard and internal standard methods;
  - Examples of qualitative and quantitative analysis.
5. Gas Chromatography (GC)
  - Chromatographic techniques: elution, frontal and displacement analysis;
  - Description of the chromatographic system;
  - Selection of operating conditions;
  - Advantages of using temperature programming;
  - Chromatographic data analysis.
6. Liquid Chromatography
  - Classification of chromatography;
  - Description of the chromatographic system;
  - Selection of operating conditions;
  - Advantages of using solvent programming;
  - Chromatographic data analysis.
7. Introduction to Spectroscopy
  - Electromagnetic radiation: amplitude, frequency, wavelength and wave number;
  - Electromagnetic Spectrum: Visible (VIS), ultraviolet (UV) and infrared (IR);
  - Absorption of radiation VIS, UV and IR;
  - Electronic, vibrational and rotational transitions;
  - Beer-Lambert Law. Absorbance, transmittance and molar absorptivity.
8. UV-VIS Spectroscopy
  - Electronic transitions;
  - Solvent selection. Solvent cutoff;
  - Chromophoric groups;
  - Instrumentation and operating principles;
  - Quantification using UV-VIS. Mixtures analysis;
  - Visible Spectrum and color.
9. FT-IR Spectroscopy
  - Equipment and basic principles;

**Course contents (extended version)**

- Vibrational modes;
- Liquid and solid sample handling techniques;
- Interpretation of the IR spectra.

**Recommended reading**

1. Miller, J. , Miller, J. , Miller, R. (2017). Statistics And Chemometrics For Analytical Chemistry. Seventh Edition. Pearson Education Limited. Collins Classics.
2. Skoog, D. , West, D. , Holler, F. , Crouch, S. (2014) Fundamentals of Analytical Chemistry (9th ed. ). Belmont, CA, USA. Cengage Learning.
3. Rouessac, F & Rouessac, A. (2007). Chemical Analysis - Modern Instrumentation Methods and Techniques (2th ed. ). West Sussex, England: John Wiley & Sons Ltd.
4. Skoog, D. , Holler F. , Crouch, S. (2018). Principles of Instrumental Analysis (7th ed. ). Cengage Learning, Boston, CA, USA.

**Teaching and learning methods**

Theoretical-practical (TP) classes: Exposition of concepts, discussion and presentation of examples. Guided resolution of exercises and critical analysis of results. Equipment demonstration. Practical-laboratorial (PL) classes: Execution of 6 laboratory assignments. Experimental data analysis and reports elaboration.

**Assessment methods**

1. Alternative 1 - (Regular, Student Worker) (Final, Supplementary)
  - Laboratory Work - 60% (Execution of laboratory protocols and written reports. Resolution of assignments.)
  - Final Written Exam - 40% (Theoretical-practical concepts.)
2. Alternative 2 - (Regular, Student Worker) (Special)
  - Final Written Exam - 60% (Conceitos laboratoriais (inclui exame prático-laboratorial))
  - Final Written Exam - 40% (Theoretical-practical concepts)
3. Alternative 3 - (Student Worker) (Final, Supplementary)
  - Final Written Exam - 60% (Conceitos laboratoriais (inclui exame prático-laboratorial))
  - Final Written Exam - 40% (Theoretical-practical concepts)

**Language of instruction**

English

**Electronic validation**

António Manuel Esteves Ribeiro	Hélder Teixeira Gomes	Ramiro José Espinheira Martins	Paulo Alexandre Vara Alves
02-10-2022	22-10-2022	22-10-2022	05-11-2022