UNIVERSITÀ
DEGLI STUDI DI MILANO-BICOCCA

SYLLABUS DEL CORSO

Chimica Analitica

2627-3-E3201Q106

Aims

The primary objective of the course is to provide students with the theoretical foundations and operational tools of chemical analysis necessary for the qualitative and quantitative determination of the chemical nature of a sample of matter. Students will be able to define the concepts related to the quality parameters of an analytical method; suggest ideas and solutions to analytical problems using the most common techniques and methodologies; justify the choice of the analytical techniques and instruments considered most suitable; identify an appropriate analytical experimental plan; and document the analytical result by expressing its value together with the associated uncertainty.

In particular, at the end of the course, students will be expected to demonstrate that they have achieved the following learning outcomes:

Knowledge and understanding. Students know: the stages of the chemical-analytical process; the fundamental quality parameters of an analytical method; the concepts of accuracy, precision, repeatability, and reproducibility of a measurement; the theoretical foundations of calibration methods in analytical chemistry; the theoretical foundations and instrumental components of spectroscopy, chromatography, and mass spectrometry.

Applying knowledge and understanding. Students are able to: use common analytical laboratory instrumentation appropriately; choose the analytical method most suitable for achieving the objectives of the analysis; describe the quality of an analytical method and the reliability of the results obtained; calculate the accuracy and precision of experimental data; design the experiments required for instrumental calibration; describe the instrumentation and applications of spectroscopy, chromatography, and mass spectrometry.

Making judgements. Students are able to: choose the analytical method most suitable for achieving the objectives of the analysis; design experiments according to the expected level of accuracy of the result, while also optimizing the costs and time of the analysis and minimizing environmental impacts. Ability to choose the analytical method or approach most suitable for achieving the objectives (chemical-analytical method, sampling plan) and to evaluate the reliability of the result.

Communication skills. Students are able to: describe in written form clearly and concisely and present orally, using appropriate scientific language, the objectives, procedures, and results of the work carried out; perform laboratory experimental work collaboratively and contribute to the development of the analysis of analytical results. Write a comprehensive report on the analysis performed, justifying the choice of the analytical methods used and interpreting the analytical results obtained in light of their experimental uncertainty.

Learning skills. Students are able to: understand the principles of analytical chemistry studied and their methodological framework in order to apply them correctly and appropriately to the problem to be solved; foresee what type of information can be extracted from the data under examination; evaluate the possibility of alternative analytical methods for solving the problem. Ability for independent learning in different disciplinary areas (chemistry, physics, biology, geology, ecology, mathematics) thanks to the foundations acquired, also through the integration of prior knowledge. Ability to successfully undertake subsequent courses within the Environmental Science and Technology programme and to continue studies independently in Master's Degree programmes, integrating previous and new knowledge.

Contents

- Introduction to analytical chemistry and its applications
- Errors in chemical analysis and quality parameters of experimental data
- Sampling, standardization and calibration
- Theoretical and instrumental principles of UV-Vis spectroscopy and mass spectrometry
- Theoretical and instrumental principles of analytical separations
- Chemical laboratory activities to provide manual and operational skills

Detailed program

Introduction to analytical chemistry, terminology and its applications: objectives of chemical analysis, qualitative and quantitative analysis. Definitions of analytical technique, method, procedure, protocol, measurement, analyte, standard, blank, replicates, sample matrix, interfering, signal noise. Description of the individual steps of an analytical procedure. Definition of the analytical-chemical system. Fundamentals of sampling. Equipment and some of the most common analytical chemical operations for sample preparation. Classification of analytical methods

Calculation tools and statistical tests for analytical chemistry. Errors in chemical analysis and quality parameters of experimental data: systematic and random error. Methods for detecting systematic error. Definition and estimates of accuracy and precision. Precision measures. Definition, interpretation and applications of the confidence intervals of the mean. The significant figures of a measure and the theory of error propagation in chemical calculations. Hypothesis testing and examples of test applications to the evaluation of the analytical result.

Standardization and calibration: calibration target, definition of blank and standards, ordinary least squares regression method, figures of merit for the evaluation of the calibration results. Validation of the analytical method: specificity, linearity, accuracy, precision, uncertainty on the data calculated from a calibration line, estimate of the experimental error from the calibration line. Calibration method by external standards. Matrix effects: definition and application of the recovery test.

Introduction to spectroscopy: theoretical fundamentals, equations and main properties of electromagnetic radiation. Interactions between matter and electromagnetic radiation. Definition of transmittance and absorbance. Lambert-Beer law: description, parameters, applicability framework, characteristics and limitations. Experimental and theoretical absorbance and blank correction. Instrumental components of UV-Vis absorbance spectroscopy: radiation sources, monochromators, detectors.

Mass spectrometry: theoretical principles, electronic ionization, definition of mass spectrum. Types of mass spectrometers: instrumental components of a mass spectrometer: ionization methods, mass analyzer, detector. Interfaces Chromatography - mass spectrometry.

Introduction to analytical separations, theoretical principles and equipment: classification of chromatographic methods, column chromatography and thin layer chromatography (TLC). Definition of chromatogram. Characteristics of the chromatographic column: distribution constants, retention times, retention factor, selectivity factor. Efficiency of the chromatographic column and its description; definition of plate height and number of theoretical plates. Factors that determine the efficiency of the chromatographic column. Van Deemter's equation. Resolution of the chromatographic column and effect of the factors on the resolution. The process of elution (isocratic and gradient). Gas-liquid chromatography; the separation process in gas chromatography; injection system, columns and their characteristics, capillary and packed columns, liquid stationary phases, flame ionization detectors (FID), thermal conductivity detectors (TCD), electron capture detectors (ECD). Liquid - Liquid Chromatography: characteristics of the chromatograph; sample pumping and injection systems. Types of columns. Characteristics of the stationary phase. Detectors. Ionic Chromatography. Overview of partition, adsorption, size-exclusion and affinity chromatography.

Lab activities (for a total of 20 hours) will be carried out to provide students with a deeper practical understanding of the theoretical concepts, along with manual and operational skills, on the following topics: choice of the most appropriate volumetric measuring device, recognize the significant figures of experimental data, calculate measurement uncertainty, critical assessment of analytical result reliability, calculation of a calibration line, application of volumetric analysis, chromatography (column and TLC), and UV-Vis absorption spectroscopy to real samples.

Prerequisites

Organic Chemistry
General and Inorganic Chemistry

Teaching form

The course is divided into

- 12 lectures (2 hours per lecture) in presence - delivered didactics;
- 6 laboratory activities (2 or 3 or 4 hours per activity) in presence - interactive teaching;
- 4 activities of exercises (2 hours per activity) in presence - delivered didactics;
- 1 activity of exercises (2 hours per activity) in presence - interactive teaching.

The lectures are intended to provide students with the theoretical concepts and basic knowledge about analytical chemistry. In the exercise session, the introduction to the statistical treatment of the analytical data and the characteristics and operational methods of the laboratory equipment are described. The laboratory activities are aimed at testing the principles and concepts introduced during the lectures. Laboratory activities are organized in individual and group projects.

The slides and videos of the lectures are constantly updated on the e-learning page of the course and additional contents are made available for further information on specific topics.

Textbook and teaching resource

The slides of the course along with additional material are provided through the e-learning page of the course. The following textbook is recommended: F.J. Holler, S.R. Crouch: Fondamenti di Chimica Analitica di Skoog & West (III Edizione). EdiSES, 2015. For each laboratory activity, a document is provided (on the e-learning page of the course) describing in details the principles and operating conditions. Guidelines for the correct use of laboratory equipment and writing a report are also available on the e-learning page of the course.

Semester

First semester

Assessment method

The final exam consists of an oral test, which involves the discussion of the topics presented in the lessons, exercitations and laboratory experiences, aimed at evaluating the achievement of the training objectives.

For admission to the exam it is necessary to have attended at least 75% of the laboratory activities and to have delivered the laboratory report, which is evaluated and contributes to the definition of the grade.

The oral exam can be replaced by two partial written exams, plus a simplified oral exam, according to the methods described below. Each partial test consists of 20 multiple choice questions. Students who obtain a cumulative score of the two written tests equal to or greater than 30/40 correct answers, access the simplified oral test, which will mainly concern the discussion of the laboratory activities carried out in relation to the fundamental topics of the course. The first partial test takes place during the week of teaching break and the second at the end of the course.

In addition to verifying the acquisition of fundamental knowledge and the ability to understand analytical problems and the most suitable solutions, the following factors contribute to the definition of the final mark: the quality of the laboratory reports in terms of completeness, accuracy and clarity; the level of knowledge acquired; the autonomy of analysis and judgment; the presentation skills and adequacy of the student's language; the evaluation of the behavior and management of workstations in laboratory activities.

Office hours

Teachers are always available to meet students in their offices (U1-3th floor) or Webex personal rooms, upon an e-mail request.

Sustainable Development Goals

QUALITY EDUCATION