UNIVERSITÀ
DEGLI STUDI DI MILANO-BICOCCA

SYLLABUS DEL CORSO

Chimica Analitica

2021-3-E3201Q106

Aims

The main objective of the course is to provide the student with the theory and operational tools in the framework of analytical chemistry necessary for the qualitative and quantitative determination of the chemical nature of a chemical sample. The student will be able to critically discuss the quality of an analytical method, to suggest ideas and solutions to analytical problems using the most common techniques and methodologies, to justify the choice of the most suitable techniques and instruments, to identify an appropriate analytical experimental plan and to document the analytical result representing its value with the associated uncertainty.
In particular, at the end of the course, the student must demonstrate that he/she has achieved the following educational objectives:

Knowledge and understanding. The student knows: the fundamental quality parameters of an analytical method; the concepts of accuracy, precision, repeatability and reproducibility; the theoretical fundamentals of calibration methods in analytical chemistry; the fundamentals and instrumental components of UV-Vis molecular absorption spectroscopy, chromatography and mass spectrometry.

Applying knowledge and understanding. The student is able to: appropriately use the common analytical laboratory instrumentation; describe the basic quality parameters of an analytical method; calculate the accuracy and precision of the experimental data; apply the principles of analytical calibration to real problems; describe the instrumentation and applications of UV-visible spectroscopy and chromatography.

Making judgements. The student is able to: write a critical report on the performed chemical analysis; judge the quality of the experimental data in terms of precision and accuracy; evaluate the information obtained from the analysis of the data; design a simple experimental plan for analytical calibration and interpret the results.

Learning skills. The student is able to: understand the principles of analytical chemistry and their methodological application to solve general analytical problems; predict what type of information will be possible to identify from the analytical data; evaluate the possibility of alternative analytical methods for solving a problem.

Communication skills. The student is able to: describe in a clear and concise written form, as well as to express orally, the objectives, the procedure and the results of the analytical experiments; carry out experimental laboratory work and develop an analytical analysis in a team-working framework.

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. Quality control: definitions of repeatability and reproducibility. Precision measures: classical standard deviation, pooled standard deviation, standard deviation of the mean, variation coefficient. Introduction to the probability distribution: Gaussian and Student probability density functions. Definition, interpretation and applications of the confidence intervals of the mean. Errors in mass and volume measurements. The significant figures of a measure and the theory of error propagation in chemical calculations. Introduction to hypothesis testing: definitions of null hypothesis, alternative hypothesis, test significance level. Student's t test for accuracy; Fischer's F test for precision; Dixon's Q test for outliers. Analysis of Variance (ANOVA): assessment of analytical and sampling variance. 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: injection system, 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 UV-Vis absorption spectroscopy to real samples.

Prerequisites

Organic Chemistry
General and Inorganic Chemistry

Teaching form

The course is divided into

- lectures (24 hours)
- practical exercises (10 hours)
- laboratory activities (20 hours)

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.

During Covid-19 emergency, lessons and laboratory will take place in a mixed mode: asynchronous/synchronous videotaped lessons and partial attendance to the chemical lab.

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 exam consists of an oral test with discussion of laboratory reports. To be admitted to the exam, students must have attended at least 75% of the laboratory activities and delivered the required lab reports. In addition to the learning ability of the fundamental notions given in the course, the following factors contribute to the definition of the final grade: quality of the reports related to the laboratory experiences in terms of completeness, accuracy and clarity; the level of acquired knowledge; autonomy of analysis and judgment; communication skills and suitability of the student's language.

Students who fail an exam can repeat it at the subsequent exam date.

During Covid-19 emergency, the lab attendance will be not mandatory. *Moreover*, t*he oral exams will be carried out online using the WebEx platform. On the e-learning page of the course, a public link for access to the examination of possible virtual spectators will be provided before every exam session.*

Office hours

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