UNIVERSITÀ
DEGLI STUDI DI MILANO-BICOCCA

COURSE SYLLABUS

Analytical Chemistry and Laboratory

2223-1-E2702Q087

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 define the concepts related to the quality parameters of an analytical method; will be able to suggest ideas and solutions to analytical problems using the most common techniques and methodologies; will be able to justify the choice of the most suitable techniques and instruments; will be able to identify an appropriate analytical experimental plan and will be able 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 has achieved the following educational objectives:

Knowledge and understanding. At the end of the course the student knows: the fundamental quality parameters of an analytical method; the concepts of accuracy, precision, repeatability and reproducibility; the fundamentals of calibration methods in analytical chemistry; the fundamentals and instrumental components of ultraviolet and visible molecular absorption spectroscopy.

Applying knowledge and understanding. At the end of the course the student is able to: appropriately use the common analytical laboratory instrumentation; describe the basic quality parameters of an analytical method; judge the accuracy and precision of the experimental data; describe the principles of analytical calibration; describe the instrumentation and applications of UV-visible spectroscopy.

Making judgements. At the end of the course the student is able to: write and justify a critical report on the analytical methods used and the information obtained from the analysis of the data; realize a simple experimental plan for analytical calibration and interpret the results.

Learning skills. At the end of the course 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. At the end of the course 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. The significant figures and the theory of error propagation in chemical calculations. Sampling, standardization and calibration. Validation of the analytical method. Theoretical and instrumental principles of UV-visible and IR spectroscopy. Practical experiences in the chemical laboratory will be carried out in order to provide manual and operational skills.

Detailed program

Introduction to analytical chemistry and its applications. Objectives of the chemical analysis: qualitative and quantitative analysis. Definitions of: technique, method, procedure, protocol, measurement, sample, analyte, standard, blank, replicates, matrix. Description of the phases of the chemical-analytical process. Definition of the chemical-analytical system. Basics of sampling. Main analytical operations for sample preparation. Definition of the concepts of instrumental signal, calibration and replicas for estimating the uncertainty of the analytical result.

Errors in experimental measurements: systematic and random errors. Definition of precision and accuracy. Accuracy and precision estimates. Definition of standard deviation, pooled standard deviation, standard deviation of the mean, coefficient of variation. Definition, interpretation and application of the confidence intervals of the mean. Definition of significant figures of a measure. Uncertainty propagation rules in arithmetic operations. Introduction to statistical tests for hypothesis testing in analytical chemistry. Student's t-test for accuracy. Fisher's test for precision.

Calibration theory. Definition of calibration and inverse prediction. Ordinary least squares. The parameters of the calibration model. The quality of the calibration model. Definition of sensitivity and white signal. Standard estimate error. Uncertainty of inverse prediction. Calibration methods: external and internal standard. Matrix effects, standard addition method (single and multiple).

Validation of the analytical method. Repeatability and reproducibility. Test for outlier data. Recovery test. Limits of detection and quantification. Linearity, range, selectivity, sensitivity, robustness. Analysis of variance (ANOVA).

Introduction to spectroscopy, equations and main properties of electromagnetic radiation. Interactions between matter and electromagnetic radiation: definitions of absorption and emission. Definition of Transmittance and Absorbance. Definition of the Lambert-Beer law, its parameters and definition of the fields of applicability, its specifications and limitations. Experimental and theoretical absorbance and blank correction. Definition and characteristics of UV-visible absorption spectra. Introduction to IR spectroscopy.

Instrumental components for UV-visible spectroscopy: sources, monochromators, detectors. Single-beam, double-beam spectrophotometers. Errors in absorbance measurements: relative precision on absorbance. Qualitative and quantitative applications of UV-visible absorption spectroscopy. Determination of substances in mixtures. Spectrophotometric titrations. Absorption by charge transfer. IR spectroscopy: mention of qualitative and quantitative applications.

Practical experiences in the laboratory include five activities related to the course contents.

Prerequisites

Principles of general and inorganic chemistry

Teaching form

The course is divided into lectures, practical exercises and five laboratory experiences. In the lectures the theoretical notions are given on the addressed topics. The practical exercises are functional to the development of skills to analyse analytical data. Laboratory experiences include the application of the principles and concepts introduced during the lectures. The slides of the lessons 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 and additional material are provided, on the e-learning page of the course. Teachers suggest a textbook. For each laboratory experience, a document is provided (on the e-learning page of the course) describing in detail the principles and operating conditions. Instructions for writing a report are available on the e-learning page of the course.

Semester

Second semester

Assessment method

The exam consists of a written test and an oral test with discussion of laboratory reports:

- the written test includes 30 multiple choice questions on the topics presented in the lectures; students with at least 18 correct answers can access the oral exam;
- the topics presented in the lectures and the reports of laboratory experiences are discussed in the oral exam.
- the final mark depends on the written test, oral exam and quality of laboratory reports

To be admitted to the exam, students must have attended at least four of the five laboratory experiences. It is also necessary to have submitted the reports relating to all laboratory experiences, for which it is necessary to receive a positive evaluation. 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. The evaluation relating to the behavior and management of the workstations in laboratory activities also contributes to the definition of the grade.

During the course, two partial tests are also provided to the students: the first one in the middle and the second one at the end of the course. Each partial test consists of 20 multiple choice questions and it is considered positive with at least 12/20 correct answers. Students with both positive tests can access the oral exam. Students who pass both partial test with a cumulative score equal to or greater than 30/40 correct answers, can access to a reduced oral test. This will mainly focus the discussion on laboratory activities in relation to the fundamental themes of the course.

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

Office hours

Teachers receive students in their offices upon an e-mail request.

Sustainable Development Goals