VNIVERSITAT DE VALÈNCIA

Course Guide
34232 Analytical chemistry laboratory II

COURSE DATA

Data Subject	
Code	34232
Name	Analytical chemistry laboratory II
Cycle	Grade
ECTS Credits	6.0
Academic year	2016 - 2017

Study (s)

Degree	Center	Acad. year	Period
1108 - Degree in Chemistry	Faculty of Chemistry	3	First term

Subject-matter

Degree	Subject-matter	Character
1108 - Degree in Chemistry	6 - Analytical chemistry	Obligatory

Coordination

Name	Department
LLOBAT ESTELLES, M JOSE	310 - Analytical Chemistry

SUMMARY

Analytical Chemistry Laboratory II is a core course taught in the third year (fall semester) of the Degree in Chemistry for a total of 6 ECTS credits.

This course familiarizes students with the most common analytical instrumental techniques (both the fundamentals of the technique and the optimization of the chemical and instrumental working conditions) and the treatment of the analytical signals produced by each technique. In their laboratory work students will acquire an awareness of the risks of using the instrumentation for each technique and the importance of respecting the safety rules in each case.

The course also introduces students to the field of application for these techniques and how the techniques are used to solve a wide range of problems.

Experiments are conducted using optical methods of analysis, electroanalytical methods, and chromatographic and electrophoretic separation methods.

PREVIOUS KNOWLEDGE

Relationship to other subjects of the same degree

There are no specified enrollment restrictions with other subjects of the curriculum.

Other requirements

To successfully complete this course, students should have acquired experience of working with instrumental techniques and knowledge of the main instrumental analysis techniques. They must therefore have passed Analytical Chemistry II and be in the process of taking Analytical Chemistry III. This will enable them to relate the contents of the theoretical lectures with the practice sessions conducted in Analytical Chemistry Laboratory I.

OUTCOMES

1108 - Degree in Chemistry

- Develop capacity for analysis, synthesis and critical thinking.
- Show inductive and deductive reasoning ability.
- Demonstrate leadership and management skills, entrepreneurship, initiative, creativity, organization, planning, control, leadership, decision making and negotiation.
- Solve problems effectively.
- Demonstrate ability to work in teams both in interdisciplinary teams and in an international context.
- Demonstrate ability to communicate information, ideas, problems and solutions to both specialist and non-specialist audiences and using information technology, as appropriate.
- Learn autonomously.
- Demonstrate the ability to adapt to new situations.
- Acquire a permanent sensitivity to quality, the environment, sustainable development and the prevention of occupational hazards.
- Demonstrate knowledge of the main aspects of chemical terminology, nomenclature, conventions and units.
- Interpret the variation of the characteristic properties of chemical elements according to the periodic table.
- Demonstrate knowledge of the main types of chemical reaction and their main characteristics.
- Demonstrate knowledge of the principles of thermodynamics and kinetics and their applications in chemistry.
- Demonstrate knowledge of the principles, procedures and techniques for the determination, separation, identification and characterisation of chemical compounds.

- Show knowledge of the metrology of chemical processes including quality management.
- Demonstrate knowledge and understanding of essential facts, concepts, principles and theories related to the areas of chemistry.
- Solve qualitative and quantitative problems following previously developed models.
- Recognise and analyse new problems and plan strategies to solve them.
- Evaluate, interpret and synthesise chemical data and information.
- Handle chemicals safely.
- Carry out standard experimental procedures involved in synthetic and analytical work, in relation to organic and inorganic systems.
- Handle the instrumentation used in the different areas of chemistry.
- Interpret data from observations and measurements in the laboratory in terms of their significance and the theories that underpin them.
- Evaluate the risks in the use of chemicals and laboratory procedures.
- Relate theory and experimentation.
- Recognise and evaluate chemical processes in daily life.
- Understand the qualitative and quantitative aspects of chemical problems.
- Develop sustainable and environmentally friendly methods.
- Relate chemistry with other disciplines.
- Students must be able to apply their knowledge to their work or vocation in a professional manner and have acquired the competences required for the preparation and defence of arguments and for problem solving in their field of study.
- Students must have the ability to gather and interpret relevant data (usually in their field of study) to make judgements that take relevant social, scientific or ethical issues into consideration.
- Students must be able to communicate information, ideas, problems and solutions to both expert and lay audiences.
- Students must have developed the learning skills needed to undertake further study with a high degree of autonomy.
- Express oneself correctly, both orally and in writing, in any of the official languages of the Valencian Community.
- Have basic skills in the use of information and communication technology and properly manage the information obtained.

LEARNING OUTCOMES

The learning outcomes for this course, which are contained in the Degree document under Analytical Chemistry, are:

1. To have the theoretical and practical knowledge needed to plan, apply and manage the most suitable analytical method for tackling environmental, health, industrial, food or any other problem related to chemical substances. (CG8, CG10, CE4, CE15, CE24, CE26)

2. To demonstrate the principles, procedures and main instrumental technologies used in chemistry for the determination, separation, identification, characterization and behaviour of chemical compounds. (CE6, CE8, CE19, CE24)

3. To clearly explain phenomena and processes related to Analytical Chemistry. (CG1, CG2, CE2, CE13)

4. To understand and rigorously use bibliographical information and technology related to chemical analytical processes. (CG7, CE16)

5 To understand and be able to use the various forms of measurement for studying chemical processes and instrumental technologies used to determine the behaviour of chemical species. (CE1, CE10)

6. To recognize and appreciate the behaviour of chemical substances for our daily lives. (CE23)

7. To use technical information to choose the best method for solving a real problem . (CG3, CG4)

8. To apply the quality criteria of the analytical results. (CG10)

9. To demonstrate knowledge of sustainable analytical methods. (CE25)

10 To have the theoretical and practical knowledge needed to manage chemical residues and safety procedures in the laboratory. (CE17, CE21)

11 To clearly explain experimental phenomena using the theories that support them. (CE20, CE22)

12. To develop experimental procedures for analysing organic and inorganic compounds. (CE18)

13. To skillfully treat errors, handle error propagation of the magnitudes measured in the laboratory, and manage computer programs for processing experimental information. (CE16, CT3)

14. To draft records, reports and the normal work procedures used in a laboratory. (CG3, CG7, CG9, CE10, CT1)

15. To demonstrate interpersonal skills also from the gender perspective. (CG6)

16. To demonstrate sensitivity to environmental issues. (CG10)

17. To demonstrate the ability to rigorously manage information. (CG7)

18. To demonstrate leadership ability also from the gender perspective. (CG3)

19 To effectively perform his or her tasks as a member of a team also from the gender perspective. (CG5)

20. To solve problems rigorously. (CG4, CG5, CE14, CE15, CE24)

These learning outcomes should ensure that on successful completion of Analytical Chemistry Laboratory II students will be able to:

• use personal and collective protective equipment in the laboratory appropriately.

• keep a laboratory notebook that reflects the essential data for the work done.

• handle materials and basic analytical instrumentation and take analytical measurements.

• link the observations found using several applied instrumental techniques to the corresponding theoretical background.

• develop procedures for preparing working solutions (sample, standards and reagents) in accordance with sustainability parameters.

• apply various sample treatment procedures.

• use various calibration strategies.

• perform the calculations needed to transform analytical signals into concentrations, mass or percentages.

• explain the purpose or objectives and the fundamentals and experimental procedure for each practice session.

• justify, on the basis of the experiments conducted, the methodological differences involved in solving analytical problems based on the type or concentration of the analyte in the sample, the physical state of the sample matrix, the complexity, etc.

• prepare analytical reports that reflect the results of the practice sessions, expressing the quantities with significant figures and the appropriate units.

• interpret the results obtained analytically in the treatment of data arrays for various kinds of problems (environmental, industrial, clinical, etc.).

• deal appropriately with the waste generated during practical sessions.

• justify the importance of reducing the environmental impact of the analytical methods by selecting reagents, minimizing their masses and volumes, and selecting residues that can be processed.

DESCRIPTION OF CONTENTS

1. Optical Methods of Analysis

This unit comprises numerous practice sessions in which several optical methods of analysis are used to determine analytes in samples of various compositions.
Many sessions also involve studying the effects of different chemical and instrumental variables. The scheduled practice sessions are:
1.1) The determination of binary samples by ultraviolet/visible absorption spectroscopy: use of derivative spectra and multiple linear regression.
1.2) The analytical applications of infrared absorption spectroscopy.
1.3) The influence of instrumental variables on molecular fluorescence: the spectrofluorimetric

determination of quinine in tonic water.
1.4) Analysis of condensed milk (part 1). Polarimetric determination of sucrose.
1.5) Analysis of condensed milk (part 2). Determination of calcium by flame atomic absorption spectroscopy.
1.6) The determination of lithium in natural waters: how the variables influence the analytical signal.

2. Electroanalytical Methods

The practice sessions in this thematic unit use some of the most common electroanalytical techniques, e.g. potentiometric and electroplating techniques. One practice session uses voltammetric determination and therefore involves the study and visualization of current-potential curves. The scheduled practice sessions are:
2.1) The use of selective electrodes for the potentiometric determination of fluoride in toothpaste.
2.2) The electrogravimetric determination of copper in brass.
2.3) Analytical applications of voltammetric techniques with a gold electrode.

3. Chromatographic and Electrophoretic Methods

The three practice sessions in this thematic unit involve different separation techniques: chromatographic methods (column liquid chromatography and gas chromatography) and electrophoretic separation. The scheduled practice sessions are:
3.1) The determination of phenols in water by liquid chromatography after concentration with solid phase extraction.
3.2) The determination of low volatility substances by gas chromatography.
3.3) The analysis of pharmaceutical products by capillary electrophoresis.

WORKLOAD

ACTIVITY	Hours	% To be attended
Laboratory practices	48,00	100
Tutorials	12,00	100
Development of group work	15,00	0
Development of individual work	10,00	0
Study and independent work	28,00	0
Readings supplementary material	10,00	0
Preparation of evaluation activities	10,00	0
Preparation of practical classes and problem	15,00	0
Resolution of online questionnaires	2,00	0
TOTAL	**150,00**	

TEACHING METHODOLOGY

The materials (scripts, guidelines, schedule, questions, etc.) will be made available to students via the virtual classroom before the beginning of the introductory session. Before work in the laboratory, each block of sessions will begin with a seminar to:

• explain the general rules of Analytical Chemistry Laboratory II.

• explain how the course will develop and discuss the various sections of this course guide.

• introduce skills students have not previously acquired but are necessary for the subject and, if appropriate, discuss any previous knowledge required.

• provide the information and materials students will need to prepare the first practice session or first block of sessions.

The course is structured around the following axes:

(i) preparation of the practice to be conducted, (ii) laboratory work, (iii) treatment of the results and (iv) laboratory notebook and analytical reports.

(i) Preparation of the practice to be conducted. With the script, the materials and the information provided by the lecturer, students prepare each practice session before coming to the laboratory.

The script will be accompanied by a series of questions on the practical session that students must write their answers to.

Students must prepare an outline of their work before they start their practice session. This serves to verify that they have understood the task and will prove useful when conducting their laboratory work.

Also in preparation for the practice session they must perform the calculations they need to prepare the solutions that will be used in the session.

The lecturer will review the material prepared by the student before the beginning of the practice session.

The aim of this preparation is to ensure that, before entering the laboratory, students understand what they need to do and why.

(ii) Laboratory work. After reviewing the students' materials, the lecturer will discuss any aspects that need to be clarified before the students start their practice session. The students will then prepare the solutions (reagents, standards and samples) and begin the session.

The sessions are conducted in pairs. Sometimes, however, in order to encourage teamwork, several pairs may share their results. At this stage of the work the lecturer will encourage a positive attitude from students toward their scientific work.

Keeping a laboratory notebook during their practical sessions is an important part of the students' laboratory work. At no time should the notes in the laboratory notebook be re-written.

(iii) Treatment of the results. The treatment of the results will begin in the laboratory. The lecturer will first guide the students in this task before the students complete the work by themselves. When presenting the results of the laboratory sessions, students must draw appropriate figures and tables to collect the data and use the correct units and significant figures. As the aim of this stage is to develop the students' analytical skills, they must not only calculate the results they obtain in the laboratory but also analyse them as well as their previous estimates.

(iv) Laboratory notebook and analytical reports. One aim of this course is to teach students to present their work appropriately by keeping a laboratory notebook and producing an analytical report.

Students must learn to keep a laboratory notebook of the work they carry out. The lecturer will periodically review the notebook. This notebook must be presented by the student after each block of sessions and at the end of the course by the deadline set by the lecturer.

When recording details in their notebook, students must remember that all scientific work should be capable of being reproduced by others. All raw data must be recorded with precision and information on the work conducted and observations found must be specified.

EVALUATION

FIRST CALL

Students' grades will be calculated from the weighted average of the following four activities: preparation and experimental technique; results of analyses of the samples; laboratory notebooks and analytical reports; and written examinations.

1. Experimental work and results:

The following aspects will be evaluated:

(a) Preparation of the practice sessions.

(b) Laboratory work: the student's abilities, interest and attitude will be taken into account when continuously evaluating his or her progress and work during the practice sessions. In particular, the student's implementation of proper experimental techniques in all laboratory operations will be assessed.

(c) Laboratory notebook

(d) Analysis of test samples: At each practice session students must analyse a sample of unknown composition and/or concentration.

The quality of the results is considered to reflect the quality of the student's experimental work.

(ii) Examinations:

After each block of sessions students will take a written examination on the concepts and skills they have acquired in the block.

The overall grade is calculated as the weighted average of the four sections. The score for each section must be at least 4.5 in order to apply the average. The minimum total score to pass the course is 5.0.

Experimental work and results	Examinations
70%	**30 %**

Students who obtain less than the minimum score in any section must take a written examination (worth 30%) and a practical examination in the laboratory (70%) in order to pass the course.

Students who do not obtain the minimum score in one of these three sections must take a written examination (worth 30%), and/or a practical laboratory examination worth 70% of the total score in order to pass the course.

Attendance at all seminars and laboratory sessions is compulsory. In the case of excused absences, students can recover up to three sessions by attending other practical groups provided the teaching requirements of the laboratories allow. The marks awarded for any session not recovered in this way will be zero. Students will fail the course if they are absent from or fail to recover more than two laboratory sessions.

SECOND CALL

In the second call the final grade is obtained by applying the same weighting criteria as in the first call. There will be a written examination (worth 30%) and a practical examination in the laboratory (70%).

NOTE: This course is excluded from the regulations on advance calls for completing graduate studies (Degree Committee agreement of 26/03/2015).

REFERENCES

Basic

- MAURÍ, A.; LLOBAT, M. Y HERRÁEZ, R. Laboratorio de Análisis Instrumental. Madrid: Servei de Publicacions de la UV y Reverté, 2010. ISBN 9788429173956
- SKOOG, D.A.; HOLLER, F. Y CROUCH, S.R. Principios de Análisis Instrumental (6ª edición). México: Cengage Learning Editores, 2008. ISBN 9789706868299
- PINGARRÓN CARRAZÓN, M. Y SANCHEZ BATANERO, P. Química electroanalítica: Fundamento y aplicaciones. Madrid: Síntesis, 1999 ISBN 8477386633
- VALCÁRCEL CASES, M. Y GÓMEZ HENS, M. Técnicas analíticas de separación, Barcelona: Reverté, 1988. ISBN 9788429179842
- CELA, R.; LORENZO R.A. Y CASAIS, M.C. Técnicas de separación en Química Analítica. Madrid: Síntesis, 2002. ISBN 8497560280