

Analytical Chemistry and Electroanalysis

Code: 102487
ECTS Credits: 12

Degree	Type	Year	Semester
2502444 Chemistry	OB	2	A

Contact

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Use of Languages

Principal working language: catalan (cat)
Some groups entirely in English: No
Some groups entirely in Catalan: No
Some groups entirely in Spanish: No

Prerequisites

The subject of Fundamentals of Chemistry must be approved.

Objectives and Contextualisation

In this subject you have to acquire the knowledge and essential skills of Analytical Chemistry that a graduate in Chemistry must have. It is a fundamental subject that allows the student to acquire the basic training in Analytical Chemistry necessary for most graduation profiles. With this objective, the principles of Analytical Chemistry, the qualitative analysis, the treatment of analytical results, the quantitative gravimetric and volumetric analysis, and the introduction to instrumental analysis techniques, in particular the electrochemical analysis techniques, are addressed.

This compulsory subject is the most basic of the area of knowledge of Analytical Chemistry with a dedication of 12 ECTS (9 theoretical and 3 practical). The knowledge acquired in this subject has a direct impact on the learning of the later subjects called Spectroscopic Methods of Analysis and Separation Techniques. On the other hand, the knowledge acquired in this subject is fundamental to be able to understand from other areas of knowledge, in accordance with the multidisciplinary nature of Analytical Chemistry.

Competences

- "Interpret data obtained by means of experimental measures, including the use of IT tools; identify their meaning and relate the data with appropriate chemistry, physics or biology theories."
- Apply knowledge of chemistry to problem solving of a quantitative or qualitative nature in familiar and professional fields.
- Develop synthesis and analyses studies in chemistry from previously established procedures.
- Handle chemical products safely.
- Handle standard instruments and material in analytic and synthetic chemical laboratories.
- Learn autonomously.
- Manage the organisation and planning of tasks.
- Obtain information, including by digital means.
- Operate with a certain degree of autonomy and integrate quickly in the work setting.
- Reason in a critical manner
- Recognise and analyse chemical problems and propose suitable answers or studies to resolve them.
- Resolve problems and make decisions.

- Show an understanding of the basic concepts, principles, theories and facts of the different areas of chemistry.
- Show sensitivity for environmental issues.
- Use IT to treat and present information.
- Work in a team and show concern for interpersonal relations at work.

Learning Outcomes

1. Classify electroanalytical and optical analysis methods, and how they are used.
2. Classify the classic methods of gravimetric and volumetric analysis based on acid-base equilibriums, complexation, redox and precipitation.
3. Describe the principles involved in electrochemical and optical analysis methods.
4. Employ information and communication technology in the documentation of cases and problems.
5. Employ the principles of electrochemistry and (optical) spectrophotometry to solve analytical problems.
6. Ensure the good state and use of instruments and reagents.
7. Evaluate the danger and risks of the use of samples and reagents, and apply suitable safety precautions for each case.
8. Explain the basic operations of electroanalytical and optical equipment.
9. Identify and situate safety equipment in the laboratory.
10. Identify the statistical methods for the treatment of the results of analyses to obtain information on their quality.
11. Interpret analysis results and assess their quality, relating them to the previous information on the sample.
12. Interpret the results obtained from analytical problems, as well as their quality parameters.
13. Learn autonomously.
14. Make adequate use of laboratory materials and instruments.
15. Manage the organisation and planning of tasks.
16. Obtain information, including by digital means.
17. Operate with a certain degree of autonomy and integrate quickly in the work setting.
18. Plan the right strategy in the different stages of the analytical procedure to solve the problems being addressed.
19. Properly use the computer tools required to resolve, expose and interpret analytical problems.
20. Reason in a critical manner
21. Recognise the stages of the analytical procedure in chemical analysis.
22. Reproduce the instructions supplied in a laboratory protocol for gravimetric, volumetric, electrochemical and optical analysis.
23. Resolve a collection of instrumental analysis problems.
24. Resolve problems and make decisions.
25. Select appropriate laboratory material for an analytical determination.
26. Show sensitivity for environmental issues.
27. Solve gravimetric and volumetric analysis problems, based on acid-base equilibriums, the formation of complexes, redox and precipitation, applying statistical methods to the treatment of the results.
28. Use IT to treat and present information.
29. Use suitable strategies for the safe elimination of reagents.
30. Work in a team and show concern for interpersonal relations at work.

Content

The theoretical contents have been structured in 13 lessons distributed in 3 thematic modules that are detailed below. The experimental contents have been distributed in 12 practical sessions, described in the 4th thematic module.

PROGRAM: THEORY AND PROBLEMS (9 ECTS credits) (82 classroom hours)

PART I: INTRODUCTION AND TREATMENT OF RESULTS

Lesson 1: Basic concepts in AQ: Object and definition of Analytical Chemistry. Division of Analytical Chemistry. Stages of the analytical procedure. Sampling. Determination. Sample pre-treatment. Dissolution and disintegration. Classification of analytical methods: classical and instrumental.

Lesson 2: Statistical evaluation of analytical data. Quantitative analysis: the experimental error. Significant numbers. Statistics: limitation of experimental measures. Systematic and random errors. Accuracy and precision. Calibration and parameters of the line. Calibration methods.

PART II: CLASSICAL METHODS. VOLUMETRICS AND GRAVIMETRIES

Lesson 3: Introduction to Volumetric Methods. Concept and methods The analytical reaction Valuation curve (eg precipitation assessment). Equivalence point and end point. Indicators: chemical and instrumental spectrophotometric and potentiometric titration cell).

Lesson 4: Acid-base volumes. Buffer solution Buffer capacity Neutralization curves. Strong and weak protoliths, monoprotic and polyprotic. Acidimetry and alkalimetry. Titrating solutions. Primary types Acid-base indicators.

Lesson 5: Volumetric complex formation (EDTA). Valuation curves. Valuing agents. Metalochromic indicators.

Lesson 6: Redox volumetries. Redox potential. Redox and acidity. Valuation curves. Oxidations and previous reductions. Standard solutions. Indicators. Valuations with strong oxidants. Valuations with strong reducers. Determination of the COD.

Lesson 7: Volumetries of precipitation. Valuation curves. Detection of the end point. Indicators.

Lesson 8: Gravimetry. Formation and evolution of precipitates. Nucleation and growth. Impurification of the precipitates. Quantitative gravimetric chemical analysis. Precipitation in homogeneous medium. General operations of gravimetric analysis.

PART III: INSTRUMENTAL METHODS. ELECTROCHEMICAL METHODS OF ANALYSIS

Lesson 9: Fundamentals of electrochemistry. Electrochemical cells: galvanic and electrolytic. Nernst equation. Standard potential. Faradaic current and non-faradaic. Ohmic fall. Polarization. Overpotential Mechanisms of mass transfer: migration, convection and diffusion. Classification of electroanalytical techniques.

Lesson 10: Electrogravimetry: Introduction. Fundamentals of electrogravimetry. Electrogravimetry with constant applied potential. Electrogravimetry with controlled cathodic or anodic potential.

Lesson 11: Coulombimetry: Introduction. Basics of coulometry. Potentiostatic Coulombimetry: anodic or cathodic controlled potential. Chemical coulombimeters. Amperostatic Coulombimetry: coulometric estimations.

Lesson 12: Voltammetry: Introduction. Fundamentals of voltammetric techniques. Microelectrodes in voltammetry. Classical polarography: electrode of mercury droplets. Polarographic waves. Current limit of diffusion. Residual current. Half-wave potential. Curves intensity-potential. Ilkovic equation. Hydrodynamic voltammetry: voltammetric sensors. Qualitative and quantitative applications.

Lesson 13: Potentiometry: Introduction. Classification of the indicator electrodes. Reference electrodes. Liquid union potential. Ion-selective electrodes. Glass electrode Direct potentiometry: calibration of the electrodes and standard addition.

MODULE IV: EXPERIMENTATION IN THE LABORATORY

The practical contents have been distributed in 8 laboratory practices that will be carried out in 12 sessions of 4 hours. The distribution of practices in sessions can be seen in the following table.

The total number of hours of dedication is 3 ECTS (75 hours of total work), considering the face-to-face time (laboratory) and the non-contact time (preparation of practices, preparation of reports and examination). The laboratory practices will be carried out in a single block upon completion of the theoretical contents corresponding to all the teaching units.

PROGRAM: PRACTICES (3 ECTS credits) (50 laboratory hours)

Session	Practice	On-site
1	P1: Determination of phosphoric acid in a carbonated cola beverage	4
2, 3	P2: Gravimetric determination of Ni content in an alloy	8
4	P3: Determination of water hardness	4
5	P4: Determination of ascorbic acid in vitamin C drugs	4
6, 7	P5: Inorganic qualitative analysis. Analysis of cations	8
8	P6: Determination of the concentration of acetic acid in vinegar	4
9	P7: Determination of Fe^{2+} content in water	4
10, 11, 12	P8: Construction and evaluation of a Ag / AgCl electrode for the determination of chloride ion in a tomato juice	12

Methodology

The activities necessary to acquire the competences of this subject include theory classes, problem classes, seminars and laboratory practices.

Theory classes

The theory classes will be expository with audiovisual support that will be available to students in the Moodle Classroom of the subject.

To reinforce the learning, formative activities will be proposed that can be carried out in group or individually. The activities are designed to promote the learning of specific competences as well as to develop transversal competences.

The training activities will be carried out inside and/or outside the classroom and aim to solve problems and/or search for information. The activities carried out outside the classroom must be delivered within the set period. Some of these activities will be in English.

Seminars will be comprised by small groups to solve doubts or work on concepts or problems with higher difficulty.

On the other hand, it will require autonomous work by the student with the aim of reinforcing knowledge from the reading and understanding of the proposed reference books, web pages or books that can be provided for specific topics.

Classes of problems

In the classes of problems, the contents of the theory classes will be developed. The statements of the problems will be delivered before the classes so that they can be worked on by the students previously, in this way they will be able to solve the doubts that arise.

Classes of practices

For the realization of laboratory practices, students will be given a script of practices in which the objectives, the basis of the experimental procedure, the results that are intended to achieve and some relevant issues will be indicated. It is very important that, prior to the execution of the practice, the students have read the script to understand the experiment they are going to perform and try to answer the questions posed later. Each day of practices, the students will dispose 4 h to carry out the laboratory experiments in a teacher-led manner and 1-2 hours of autonomous work for reading and understanding the script of practices and to make a report with the results obtained, the discussion of them and the most relevant conclusions.

Safety warning in the laboratory: The student who is involved in an incident that may have serious safety consequences may be expelled from the laboratory and suspend the subject.

Activities

Title	Hours	ECTS	Learning Outcomes
Type: Directed			
Seminars	4	0.16	8, 10, 20, 21, 24, 30, 28
Type: Supervised			
Cooperative activities	10	0.4	2, 5, 3, 8, 15, 26, 16, 17, 20, 21, 30, 19, 28

Assessment

The evaluation will be done continuously through three partial exams, training activities and laboratory practices.

The final grade of the subject will be calculated by weighting each of the different parts of theory of the subject and laboratory practices. The calculation of the final grade of the subject will be carried out according to the expression:

$$\text{FINAL NOTE} = 0.8 \times \text{GLOBAL NOTE THEORY} + 0.2 \times \text{GLOBAL NOTE PRACTICES}$$

where,

- GLOBAL NOTE THEORY = NOTE EXAMINATIONS (out of 10) x 1 + NOTE TRAINING ACTIVITIES (OVER 10) X 0.05

$$\text{NOTE EXAMINATIONS} = 0.25 \times \text{Partial Exam 1} + 0.30 \times \text{Partial Exam 2} + 0.45 \times \text{Partial Exam 3}$$

- GLOBAL NOTE PRACTICES = 0.5x Note Laboratory + 0.5 x Note Practice Exam

$$\text{LABORATORY NOTE} = 0.9 \times \text{NOTE REPORTS} + 0.1 \times \text{NOTE ATTITUDE}$$

To pass the subject you must obtain a GLOBAL NOTE of THEORY rating greater than or equal to 5 (out of 10 points) and a GLOBAL NOTE of PRACTICES greater than or equal to 5 (out of 10 points). In case one of these two conditions is not met, the FINAL NOTE that will be published in the minutes will be a maximum of 4.5 (out of 10 points).

When the GLOBAL NOTE of THEORY by continuous evaluation is less than 5 (out of 10 points), there will be the possibility of doing a final exam of the whole subject. To participate in the recovery the students must have been previously evaluated in a set of activities the weight of which equals a minimum of two thirds of the total grade of the subject (2 of the three partial exams) and the grade of The global theory grade is at least 2. For the people who have taken the final exam, the qualification of the training activities will be taken into account in the same way as in the continuous evaluation by partial exams. In this case, the following expression will be used:

- GLOBAL NOTE THEORY = NOTE FINAL EXAM (above 10) x 1 + NOTE TRAINING ACTIVITIES (out of 10) x 0.05

When the note of the practical exam is less than 4 (over 10 points), the subject can not be approved by continuous evaluation by partial, and must be submitted to the written exam of practices of repechage that will be carried out on the same day as the final exam. In this case the GLOBAL PRACTICAL NOTE will be calculated with the following expression (without restrictions of minimum note in the exam of practices of repechage):

- GLOBAL NOTE PRACTICES = 0.5 x LABORATORY NOTE + 0.5 x NOTE PRACTICAL EXAMINATION (of repechage)

The qualification of "NOT EVALUABLE" will be obtained in the following cases:

- There is no note of laboratory practices (attendance to the classes of practices is compulsory).
- The student does not show 2 of the 3 partial exams and does not present the final exam.

In case of failing the subject and approving the practices, only the GLOBAL PRACTICE NOTE for the following course may be retained if it is equal to or greater than 6 (out of 10 points).

Other important considerations:

- The student who is involved in an incident that may have serious safety consequences may be expelled from the laboratory and suspend the subject.
- In the event that the student performs any irregularity that may lead to a significant variation of the grade of an evaluation act, the final grade of this subject will be 0.

Assessment Activities

Title	Weighting	Hours	ECTS	Learning Outcomes
Laboratory practices	20	55	2.2	13, 4, 15, 9, 11, 12, 26, 17, 18, 21, 22, 25, 19, 14, 29, 7, 6
Partial 1	14	43	1.72	13, 10, 20, 21, 24, 28
Partial 2	24	87	3.48	13, 2, 1, 15, 26, 16, 20, 22, 24, 30, 19, 28
Partial 3	30	95	3.8	13, 1, 5, 3, 8, 16, 17, 20, 24, 23, 27, 30, 19, 28
Training activities	12	6	0.24	13, 2, 1, 5, 3, 8, 15, 10, 16, 20, 21, 24, 30, 28

Bibliography

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Skoog, West, Holler, Crouch, Fundamentos de Química Analítica, 8^a edició, Thomson Editores Spain, Paraninfo, Madrid, 2005.