



Course guide

320063 - AQ - Chemical Analysis

Last modified: 19/04/2023

Unit in charge: Terrassa School of Industrial, Aerospace and Audiovisual Engineering
Teaching unit: 713 - EQ - Department of Chemical Engineering.

Degree: BACHELOR'S DEGREE IN CHEMICAL ENGINEERING (Syllabus 2009). (Compulsory subject).

Academic year: 2023 **ECTS Credits:** 6.0 **Languages:** Catalan, Spanish

LECTURER

Coordinating lecturer: M^a Dolores Alvarez del Castillo

Others:

REQUIREMENTS

To successfully achieve the objectives of this course, we consider very convenient having previously approved the course of Probability and Statistics (Q3) and Chemistry (Q1).

DEGREE COMPETENCES TO WHICH THE SUBJECT CONTRIBUTES

Specific:

2. CHE: Ability to design and manage applied experimental procedures, in particular to determine thermodynamic and transport properties, and to model phenomena and systems related to chemical engineering: systems with fluid flow, heat transfer, mass-transfer operations, and reactor and reaction kinetics.

3. CHE: Knowledge of material and energy balances, biotechnology, the transfer of materials, separation operations, chemical reaction engineering, the design of reactors, and the reuse and transformation of raw materials and energy resources.

Transversal:

1. EFFICIENT ORAL AND WRITTEN COMMUNICATION - Level 3. Communicating clearly and efficiently in oral and written presentations. Adapting to audiences and communication aims by using suitable strategies and means.

TEACHING METHODOLOGY

In order for students to meet the objectives and acquire the competencies described above, this subject is organized in four different types of sessions:

- Presential sessions of exposure and content application.
- Presential practical laboratory work sessions.
- Non presential independent-learning sessions.
- Non presential teamwork sessions.

Presential sessions of exposure and content application:

The lecturer will introduce the theoretical fundamentals of the subject, concepts, methods and results, which will be illustrated with relevant examples to facilitate their understanding.

Most of the topics will be covered in the following types of lectures:

- Exposition of contents with problem-solving examples.
- Problem-solving activity by the lecturer
- Problem-solving activity in small groups (2-3 students) or individually. The lecturer will mark some of the problems assigned in class and she return them to the students, so that they may track their progress over the course of the semester. These problems evaluated by the lecturer are part of the students' final marks.

Students' understanding of the content will be assessed by two exams.

Presential practical laboratory work sessions:

The students will carry out two types of experiments:

- A series of supervised experiments, without fully defined protocols, to be carried out in the laboratory on the day set by the lecturer.
- An analytical problem, chosen by students from a range of possible problems suggested by the lecturer, to be carried out in the laboratory.

Using appropriate bibliographical sources, each student will have to search for instructions on how to correctly carry out the experiment, draft a protocol for solving the analytical problem, orally present the protocol to the other students and the lecturer, carry out one of the experiments designed by a classmate in the laboratory, and draw pertinent conclusions.

Non presential independent-learning sessions.

Students are expected to spend time outside of class studying the theoretical content in order to understand it and apply it correctly. Students should divide this time between the study of theoretical fundamentals and the application of these fundamentals to solve problems assigned by the lecturer. The lecturer will mark some of the assigned problems and return them to the students, so that they may track their progress over the course of the semester. These problems will count towards students' final marks.

Non presential teamwork sessions:

Students are expected to spend time outside of class working in teams of at least 2 students to solve one of the analytical problems assigned by the lecturer. Using appropriate bibliographical sources, each student will have to search for instructions on how to correctly carry out the experiment, draft a protocol for solving the analytical problem, and orally present the protocol, to the other students and lecturer.

Each team will have tutoring/consulting time with the lecturer prior to the oral presentation of the analytical problem.

The subject has been designed to ensure that students attain Level 3 of the , "oral and written expression" competence.

Students will be given educational materials covering the theoretical basis, the experimental procedure and a bibliographic reading list for each of the experimental sessions.

Students will receive communications and notifications by means of the UPC Digital Campus, which is currently available to lecturers and students.

LEARNING OBJECTIVES OF THE SUBJECT

In this subject, students will learn to do the following:

- Identify the general steps to follow in the analytical process. Take a correct sample and make correct preparation for analysis. Select a correct analysis method based on analytical problem solving. Know how to plan and solve a problem of chemical analysis depending on the availability of instruments.
- Identify and correctly use basic laboratory equipment and instruments.
- Perform the correct treatment, evaluation and interpretation of analytical data and reporting. Applying statistics to the analytical results and draw appropriate conclusions.
- Apply the correct knowledge about chemical equilibrium. Given an aqueous solution, determine the major species in balance. Determine the effect they can have the presence of certain substances or the effect of pH on different equilibria (parasitic reactions).
- Acquire, understand and apply knowledge about the different methods of analysis. Both the gravimetric and volumetric analysis as regard to instrumental analysis. Apply knowledge about the methods of classical analysis (volumetric and gravimetric). Apply knowledge concerning potentiometric methods of analysis to make quantitative determinations. Apply knowledge concerning the spectroscopic methods of analysis to make quantitative determinations. Apply knowledge regarding the chromatographic methods of analysis to make quantitative determinations.
- Acquire the basic skills for the experimental realization and evaluation of the quality of classical and instrumental methods of analysis and their application in industrial process control.
- Display and interpret results according to different audiences and purposes.
- Communicate clearly and effectively in an oral presentation using appropriate strategies and resources. Analyze, evaluate and respond to the questions presented to him.

STUDY LOAD

Type	Hours	Percentage
Hours large group	20,0	13.33
Hours medium group	10,0	6.67
Hours small group	30,0	20.00
Self study	90,0	60.00

Total learning time: 150 h

CONTENTS

TOPIC 1: BASIC TOOLS OF CHEMICAL ANALYSIS

Description:

- Basic units of measurement. Units for expressing concentration. Significant figures.
- Stoichiometric calculations: conservation of mass, conservation of charge, conservation of protons, conservation of electrons.
- Basic equipment and instruments.
- Laboratory notebook.

Specific objectives:

On completing this topic, students will be able to:

- Correctly express an analytical result using the appropriate units and significant figures.
- Correctly carry out stoichiometric calculations for a reaction and, in particular, use the principles of conservation in stoichiometric problems.
- Identify and correctly use basic laboratory equipment and instruments (balances, volumetric material, sample-drying equipment).

Full-or-part-time: 2h 30m

Theory classes: 1h

Self study : 1h 30m

TOPIC 2: ANALYTICAL DATA ASSESSMENT: SELECTING AN ANALYTICAL METHOD

Description:

- Steps of the analytical process.
- Processing and assessment of analytical data.
- Parameters/criteria for selecting an analytical method.

Specific objectives:

On completing this topic, students will be able to:

- Identify the general steps followed in the analytical process.
- Identify potential sources of error in chemical analysis (gross, systematic and random errors).
- Identify the effects of errors (gross, systematic and random) on chemical analysis results.
- Apply the appropriate statistical treatment to random errors in chemical analysis.
- Correctly present chemical analysis results.
- Correctly calculate the confidence interval.
- Correctly apply statistical hypothesis tests.
- Detect possible gross errors. Dixon's Q test.
- Calculate the detection limits of a method.
- Identify and apply criteria for selecting a chemical analysis method: accuracy, precision, sensitivity, selectivity, robustness, ruggedness, scale of operation, time of analysis, and equipment availability.

Related activities:

Activity 1.

Delivery 1: ANOVA problem resolution

Full-or-part-time: 5h

Theory classes: 1h

Practical classes: 1h

Self study : 3h

TOPIC 3: CHEMICAL EQUILIBRIUM

Description:

- State of chemical equilibrium. Expressions of the equilibrium constant.
- Acid-base equilibrium.
- Precipitation equilibrium.
- Complex formation equilibrium.
- Redox equilibrium.

Specific objectives:

On completing this topic, students will be able to:

- Determine the main species in equilibrium given a particular aqueous solution.
- Use mass balances corresponding to various equilibria.
- Use charge balances corresponding to various equilibria.
- In the case of acid-based equilibrium, correctly use the proton balance.
- In the case of complex formation equilibrium, correctly use the ligand balance.
- Foresee the presence of substances that could lead to parasitic reactions.
- Correctly determine and use parasitic reaction rates.
- Correctly determine and use conditional constants.
- Predict the pH of a solution.
- Correctly plot and use diagrams of $\log C$ vs. pH, pL and pE.
- Correctly calculate initial and final precipitation conditions.
- Calculate whether particular substances can be separated by fractional precipitation.
- Correctly calculate the solubility of a particular substance under different conditions (pH, common ion effect, parasitic reactions).
- Foresee the presence of substances that could lead to changes in potential (pH, precipitation reactions, formation of complexes).
- Calculate the normal conditional or apparent potential of a redox system.
- Correctly plot, use and interpret predominance diagrams.
- Correctly determine whether a particular substance undergoes disproportionation under specific conditions.

Related activities:

Activity 1.

Delivery 2: Acid-base Balance Problem

Delivery 3: Equilibrium precipitation / complexation Problem

Full-or-part-time: 36h

Theory classes: 4h

Practical classes: 8h

Self study : 24h

TOPIC 4: CLASSICAL ANALYTICAL METHODS: Gravimetric and volumetric methods

Description:

- Gravimetric methods of analysis. Colloidal precipitates, crystalline precipitate. Homogeneous precipitation.
- Volumetric methods of analysis. Acid-base titration, complexometric titration, redox titration and precipitation titration.

Specific objectives:

On completing this topic, students will be able to:

- Gravimetric analysis:
 - Solve quantitative analytical problems by means of gravimetric analysis.
 - Differentiate between crystalline and colloidal precipitates and know what measures to apply during analysis in order to obtain the former.
- Volumetric analysis:
 - Solve quantitative analytical problems by means of volumetric analysis.
 - Predict the shape of an acid-base titration curve.
 - Predict the shape of a complexometric titration curve.
 - Predict the shape of a precipitation titration curve.
 - Identify appropriate titration endpoint indicators.

Related activities:

Activity 1:

Delivery 4: Gravimetric Analysis Problem

Delivery 5: Volumetric analysis (acid-base) Problem

Delivery 6: Volumetric Analysis Problem

Activity 2:

Students will carry out the following laboratory practicals:

- P1. Determinació and analysis of an acid-base titration curve.
- P2. Gravimetric content of the analyte in a sample

Full-or-part-time: 33h 30m

Theory classes: 2h 30m

Practical classes: 1h

Laboratory classes: 8h

Self study : 22h

TOPIC 5: ELECTROCHEMICAL ANALYTICAL METHODS

Description:

- Classification of electrochemical methods of analysis.
- Potentiometric methods of analysis. Reference electrodes and working electrodes (metallic and membrane electrodes).
- Quantitative applications.

Specific objectives:

On completing this topic, students will be able to:

- Correctly apply the general principles of potentiometric analysis to quantitative determinations.
- Identify the basic components of a potentiometric analysis.
- Identify the main reference electrodes used in potentiometric analysis.
- Identify the main working electrodes used in potentiometric analysis. Metallic and membrane working electrodes. Glass electrodes for measuring pH.

Related activities:

Activity 2:

Students will carry out the following laboratory practicals:

- P3 Determination of the content of a specific analyte in a sample problem by direct potentiometry (calibrated electrode method or standard addition)
- P4 determination of a particular analyte in a sample problem using a potentiometric titration.

Full-or-part-time: 20h 30m

Theory classes: 1h 30m

Laboratory classes: 8h

Self study : 11h

TOPIC 6: SPECTROSCOPIC ANALYTICAL METHODS

Description:

- General aspects of spectroscopy. Interaction between matter and electromagnetic radiation.
- Basic components of spectroscopy instrumentation.
- Absorption spectroscopy. Absorbance of electromagnetic radiation. Transmittance and absorbance.
- Absorbance and concentration. Beer's law.

Molecular absorption spectroscopy. Quantitative and qualitative applications.

- Atomic absorption spectroscopy. Quantitative and qualitative applications.

Specific objectives:

On completing this topic, students will be able to:

- Correctly apply the general principles of spectroscopic analysis to quantitative determinations.
- Identify the basic components of spectroscopy instrumentation.

Related activities:

Students will carry out the following laboratory practicals:

- P5 Determination of the amount of a metal in a sample by means of atomic absorption spectroscopy.
- P6 Determination of the amount of a particular analyte in a sample by means of molecular absorption spectroscopy.

Full-or-part-time: 20h 30m

Practical classes: 1h 30m

Laboratory classes: 8h

Self study : 11h

TOPIC 7: CHROMATOGRAPHIC ANALYTICAL METHODS

Description:

- General theory of column chromatography. Chromatographic resolution, capacity factor, column selectivity, column efficiency, peak capacity, non-ideal behaviour.
- Optimisation of chromatographic separations. Use of capacity factor, selectivity and efficiency to optimise resolution.
- Gas chromatography (GC) instrumentation. Mobile phases. Columns. Stationary phases. Injection system. Temperature control. Detectors.
- Quantitative and qualitative applications of GC.
- High-performance liquid chromatography (HPLC) instrumentation. Mobile phases (isocratic elution and gradient elution). Columns. Stationary phases (normal phase, reverse phase). Injection system. Detectors.

Specific objectives:

On completing this topic, students will be able to:

- Correctly apply the general principles of chromatographic analysis (GC, HPLC) to quantitative and qualitative determinations.
- Identify the basic components of chromatography instrumentation.

Related activities:

Full-or-part-time: 14h

Theory classes: 2h 30m

Self study : 11h 30m

TOPIC 8.CONTENT INTEGRATION

Description:

Solving an analytical problem. Determining the content of a specific analyte in a real sample. Students must look for information, must write a work procedure to solve the problem in the lab (standard protocol working PNT) and must present the protocol to their peers and teacher. The same students who have created the PNT must resolve questions posed by classmates about this PNT.

The PNT will be executed by other colleagues in the laboratory to resolve experimentally the analytical problem, extracting appropriate conclusions.

Specific objectives:

- Apply knowledge about the different methods of analysis.
- Learn to plan and solve problems in chemical analysis depending on the availability of instruments.
- Take a sample and make a right preparation for analysis.
- Select a correct analysis method based on analytical problem solving.
- Display and interpret results according to different audiences and purposes.
- Communicate clearly and effectively in an oral presentation using appropriate strategies and resources.
- Analyse, evaluate and respond to the questions presented to him.

Related activities:

Activity 2

Students will carry out the following laboratory practicals:

- P7 analytical problem solving

Full-or-part-time: 12h

Laboratory classes: 6h

Self study : 6h

ACTIVITIES

ACTIVITY 1. PROBLEM RESOLUTION

Description:

Application of theoretical foundations to solve the problems posed by the teacher. The problems posed by the teacher will be evaluated by him and returned to the student so that the student knows what their level of attainment of knowledge throughout the semester.

Specific objectives:

- Perform the correct treatment, evaluation and interpretation of analytical data. Applying statistics to the analytical results and draw appropriate conclusions.
- Apply the correct knowledge about chemical equilibrium. Given an aqueous solution, determine the major species in balance. To determine the effect they can have the presence of certain substances or the effect of pH on different equilibria (parasitic reactions).
- Correctly apply the knowledge in relation to gravimetric analysis
- Correctly apply the knowledge in relation to volumetric analysis.

Delivery:

Delivery 1: ANOVA problem (0,15% final mark)

Delivery 2: Equilibrium acid-base problem (0,15% final mark)

Delivery 3: Equilibrium precipitation / complexation problem (0,15% final mark)

Delivery 4: Gravimetric analysis problem (0,15% final mark)

Delivery 5: acid-base volumetric analysis problem (0,20% final mark)

Delivery 6: Volumetric Analysis Problem (0,20% final mark)

Total: 10% of the FINAL mark

Full-or-part-time: 15h

Self study: 15h

ACTIVITY 2: LABORATORY

Description:

- P1 Determination and analysis of an acid-base titration curve.
- P2. Gravimetric content of the analyte in a sample
- P3 Determination of the content of a specific analyte in a sample problem by direct potentiometry (calibrated electrode method or standard addition)
- P4 determination of a particular analyte in a sample problem using a potentiometric titration
- P5 Determination of metal content by atomic absorption in a sample.
- P6. Determination of the analyte content by molecular absorption spectroscopy.
- P7 analytical problem

Specific objectives:

- Acquire the basic experimental skills for the realization and evaluation of the quality of classical and instrumental methods of analysis and their application in industrial process control.
- Apply knowledge about the different methods of analysis.
- Learn to plan and solve a problem of chemical analysis depending on the availability of instruments.
- Take and prepare a sample for analysis.
- Select a correct analysis method depending on the analytical problem.
- Effective oral and written expression

Material:

Documents accessible on Athenea campus

- "Manual practices"
- "How to make a laboratory diary"
- "How to make a lab report"
- "General rubric for the laboratory reports"
- "How to make a working protocol"
- "Waste laboratory Management "
- "How to make an oral presentation"

Delivery:

Laboratory reports and work at laboratory: 20% final mark

Oral and Written expression : 10% final mark

Full-or-part-time: 30h

Practical classes: 30h

ACTIVITY 3: EXAM 1

Description:

Evaluation of the achievement of objectives relating to items 1, 2, 3 and 4

Delivery:

Total: 30% final mark

Full-or-part-time: 3h

Self study: 3h

ACTIVITY 4: EXAM 2

Description:

Evaluation of the achievement of objectives relating to items 4, 5, 6 and 7

Delivery:

Total: 30% final mark

Full-or-part-time: 3h

Self study: 3h

GRADING SYSTEM

- First exam: 30%
- Second exam: 30%
- Problem-solving activities: 10%
- Laboratory: 20%
- Oral and written expression: 10%

For those students who meet the requirements and submit to the reevaluation examination, the grade of the reevaluation exam will replace the grades of all the on-site written evaluation acts (tests, midterm and final exams) and the grades obtained during the course for lab practices, works, projects and presentations will be kept.

If the final grade after reevaluation is lower than 5.0, it will replace the initial one only if it is higher. If the final grade after reevaluation is greater or equal to 5.0, the final grade of the subject will be pass 5.0.

EXAMINATION RULES.

The Attendance in the laboratory is mandatory. The Laboratory work required the use of gown and safety goggles.

The unsatisfactory results of the First exam will redirected through a written test that will be held in the same day as the second exam. All the students enrolled in the course can access to this second-chance.

The second-chance exam results will have a score between 0 and 10, and the score obtained will replace the initial score of the exam 1, as long as the score is higher.

BIBLIOGRAPHY

Basic:

- Burriel Martí, Fernando [et al.]. Química analítica cualitativa. 18a ed. Madrid: Paraninfo, 2001. ISBN 8428312532.
- Harris, Daniel C. Análisis químico cuantitativo [on line]. 3a ed. Barcelona: Reverté, 2016 [Consultation: 10/06/2022]. Available on: https://www-ingebook-com.recursos.biblioteca.upc.edu/ib/NPcd/IB_BooksVis?cod_primaria=1000187&codigo_libro=7708. ISBN 9788429194159.
- Harvey, David. Química analítica moderna. Madrid: McGraw-Hill, 2002. ISBN 8448136357.
- Skoog, Douglas A.; West, Donald M.; Holler, F. James. Fundamentos de química analítica [on line]. 4a ed. Barcelona: Reverté, 1996-1997 [Consultation: 31/03/2023]. Available on: https://www-ingebook-com.recursos.biblioteca.upc.edu/ib/NPcd/IB_BooksVis?cod_primaria=1000187&codigo_libro=12251. ISBN 8429175563.

Complementary:

- Skoog, Douglas A. [et al.]. Principios de análisis instrumental. 5a ed. Madrid: McGraw-Hill, 2003. ISBN 8448127757.