FACULTAD DE
CIENCIAS QUÍMICAS

ANALYTICAL CHEMISTRY III

COURSE GUIDE

BSc Chemistry

Academic Year 2023-2024

UNIVERSIDAD
COMPLUTENSE
MADRID

I.- IDENTIFICATION

COURSE NAME:	**Analytical Chemistry III**
CREDITS NUMBER:	**6**
CHARACTER:	**Compulsory**
SUBJECT:	**Analytical Chemistry**
MODULE:	**Fundamental**
UNDERGRADUATE DEGREE:	**Bachelor´s Degree (BS) in Chemistry**
SEMESTER/TERM:	**Second semester (third grade)**
DEPARTMENT/S:	**Analytical Chemistry**

LECTURERS:

Coordinator	**Lecturer:** Mª MILAGROS GÓMEZ GÓMEZ **Department:** Analytical Chemistry **Office**: QA- 322C **e-mail:** mmgomez@quim.ucm.es
Laboratory Coordinator	**Lecturer:** Mª TERESA PÉREZ CORONA **Department:** Analytical Chemistry **Office**: QA- 319B **e-mail:** mtperez@ucm.es

Theory Group E	
Theory Seminars Tutorials	**Profesora:** MARÍA PEDRERO MUÑOZ **Departamento:** Analytical Chemuistry **Office:** QA-305 **e-mail:** mpedrero@ucm.es
Theory Seminars Tutorials	**Profesora:** RIANSARES MUÑOZ OLIVAS **Department:** Analytical Chemistry **Office:** QA-319B **e-mail:** rimunoz@quim.ucm.es

Laboratories QA-307 and QA-407

Group	Semester	Lecturer	e-mail	Office	Dep.
E1	2º	Lect A Ángel Julio Reviejo García Lourdes Agüí Chicharro	 reviejo@ucm.es malagui@ucm.es	 QA-316 QA-321B	 QA QA
E2	2º				
E3	2º	Lect D Gustavo Moreno Martín	gusmoren@ucm.es	QA-402	QA
E4	2º				

II.- OBJETIVES

■ GENERAL OBJETIVE

Introducing the students to the fundamentals of instrumental electrochemical techniques as well as chromatographic and non-chromatographic separation techniques, generally employed in analysis laboratories.

Students are also expected to solve particular analytical problems from different fields: clinical, agro-food, toxicology, environment and industry; likewise, acquiring working habits and good laboratory practices and general knowledge of working procedures and safety protocols.

■ SPECIFIC OBJECTIVES

- Learn the fundamentals and application of the main electrochemical techniques.
- Learn the fundamentals and applications of chromatographic and non-chromatographic separation techniques.
- Know how to apply different sample treatment methodologies as part of the analytical method.
- Be able to choose an instrumental method either electrochemical and/or chromatographic for application to a particular analytical problem.
- Evaluate with good criteria the analytical characteristics of an analytical method.
- Acquire practical training on electrochemical and chromatographic techniques.
- Draw up a correct report that summarizes the results obtained in the laboratory following metrological and quality criteria.
- Use safely chemical products by assessing their risks.

III.- PREVIOUS KNOWLEDGE AND RECOMMENDATIONS

■ PREVIOUS KNOWLEDGE:

■ RECOMMENDATIONS:

It is recommended to have passed the following courses: *General Chemistry*, *Basic Laboratory Operations*, and *Analytical Chemistry I* from the compulsory subject Analytical Chemistry in the Fundamental Module.

It is advised to have completed the course *Analytical Chemistry II* (first semester).

IV.- CONTENTS

■ BRIEF DESCRIPTION:

Theoretical contents:

Electrochemical techniques. Chromatographic and non-chromatographic separation techniques.

Laboratory contents:

Sample treatment procedures applied to specific analysis in industrial, agro-food, clinical, environmental and social fields. Applications of the main electrochemical and chromatographic techniques.

SYLLABUS:

Theory

Unit 1: Fundamentals of the electrochemical analysis techniques

- Electrochemical reaction and electrochemical cells.
- Faradaic and non-faradaic processes.
- Factors affecting electrochemical reactions' rate and current intensity. Mass transfer processes.
- Current-potential curves in simple systems. Rapid and slow systems.

Unit 2: Potentiometric and voltammetric techniques

- Potentiometric techniques.
- Voltammetry: Linear sweep techniques. Cyclic voltammetry. Pulsed techniques. Square wave voltammetry. Stripping techniques.

Unit 3: Introduction to chromatographic techniques

- Introduction to separation techniques.
- Fundamentals of chromatography
- Chromatographic techniques classification
- Chromatographic parameters.

Unit 4: Gas and liquid chromatography

- Fundamentals of gas chromatography
- Instrumentation
- Gas chromatography applications
- Fundamentals of liquid chromatography
- Instrumentation
- Liquid chromatography applications.

Unit 5: Chromatography coupled to mass spectrometry

- Gas chromatography coupled to mass spectrometry: interfaces, sample ionization modes, specific instrumentation
- Liquid chromatography coupled to mass spectrometry: interfaces, sample ionization modes, specific instrumentation.

Unit 6: Non-chromatographic separation techniques

- Introduction.
- Solid phase extraction. Applications.
- Electrophoresis fundamentals.
- Capillary electrophoresis. Applications

Laboratory

Students will carry out a total of seven laboratory practices divided into seven 4-hour sessions. During the practical sessions, students will determine either inorganic species or organic compounds in various samples. Several sample treatments will be used, as well

as, electrochemical and chromatographic techniques. Data obtained by the different groups will be evaluated through an inter-comparison exercise.

1. Electroanalysis

- *Lab practice 1: Potentiometric determination of fluoride in tooth paste, tea and water.*
- *Lab practice 2: Determination of cadmium and lead in wine by anodic stripping voltammetry.*
- *Lab practice 3: Voltammetric techniques applied to the electrochemical study of compounds of clinical interest. Application to their determination in biological fluids.*

2. Chromatography

- *Lab practice 4: Analysis of a pharmaceutical preparation. Determination of acetylsalicylic acid and paracetamol by HPLC with ultraviolet detection. Participation in an inter-comparison exercise.*
- *Lab practice 5: Determination of the herbicide atrazine and its degradation products by HPLC with ultraviolet detection.*
- *Lab practice 6: Gas chromatography: a) optimization of parameters involved in a chromatographic method; b) identification of vegetable oils by their fatty acid composition.*
- *Lab practice 7: Beer analysis. Determination of pH, total acidity and ethanol. Participation in an inter-comparison exercise.*

V.- COMPETENCES

GENERAL:

- **CG3-MFQA:** To rigorously express the acquired knowledge in Analytical Chemistry to be clearly understood in multidisciplinary areas.
- **CG6-MFQA:** To analyze and solve qualitative and quantitative problems.
- **CG7-MFQA:** To identify emerging analytical problems and propose strategies for their solution.
- **CG8-MFQA:** Searching and exploiting effectively scientific information and techniques in the field of Analytical Chemistry.
- **CG9-MFQA:** To demonstrate knowledge on laboratory related materials and practical laboratory skills.
- **CG10-MFQA1:** Chemical materials safe handling.
- **CG10-MFQA2:** To identify and assess chemicals and laboratory procedures hazards.
- **CG11-MFQA:** Standard chemical instrumentation handling.
- **CG12-MFQA:** To analyze data from experimental observations and measurements performed in analytical laboratories.
- **CG13-MFQA:** To identify and implement appropriate measurement and experimentation-based scientific practices in Analytical Chemistry.

- **SPECIFIC:**
  - **CE4-MFQA1:** To describe the analytical process steps and be able to balance their relevance.
  - **CE4-MFQA2:** To apply the methodologies associated with sample treatment and recognize associated problems.
  - **CE6-MFQA1:** To apply the fundamentals of the main electrochemical instrumental techniques, as well as of chromatographic and electrophoretic techniques.
  - **CE6-MFQA2:** To propose a suitable electroanalytical or chromatographic technique for the identification or quantification of a certain analyte.
  - **CE7-MFQA1:** To apply the basics of chemometrics as tool for metrology and quality management.
  - **CE7-MFQA2:** To develop in students the ability to apply the main instrumental and separation techniques in the laboratory to solve specific analytical problems.

- **GENERIC:**
  - **CT1-MFQA:** Preparing and writing scientific and technical analytical reports.
  - **CT2-MFQA:** Teamwork cooperation.
  - **CT3-MFQA:** To apply critical and self-critical reasoning.
  - **CT5-MFQA:** To manage chemical information, chemical literature, and specialized databases in the Analytical Chemistry field.
  - **CT6-MFQA**: To identify the importance of Analytical Chemistry in the industrial, environmental, and social context.
  - **CT7-MFQA:** To take advantage of tools and software for the treatment of experimental results.
  - **CT11-MFQA:** Autonomous learning development.
  - **CT12-MFQA:** To develop sensitivity to environmental issues related to Analytical Chemistry.

VI. – LEARNING OUTCOMES

Once this course has finished, the student must be able to:

- Describe the fundamentals of the main electrochemical techniques.
- Distinguish i-E curves in simple electrochemical systems.
- Describe the fundamentals of potentiometric techniques and ion-selective electrodes.
- Describe the fundamentals of the main voltammetric techniques.
- Describe the fundamentals of the main chromatographic techniques.
- Calculate the main chromatographic parameters from the chromatograms' data.
- Predict the compounds elution order under certain chromatographic conditions.

- Choose the most suitable chromatographic method according to the analytes to separate.
- Explain the solid phase extraction processes.
- Describe the fundamentals of the electrophoretic techniques.
- Apply the most appropriate methods for the preparation of samples of different nature.
- Apply basic security rules in the laboratory.
- Handle electrochemical and chromatographic instrumentation.
- Interpret information obtained from analytical instruments.
- Apply statistical tools and software in instrumental analysis.
- Analyze and compare the obtained results in inter-comparison exercises.
- Prepare laboratory reports.
- Use chemical waste disposal protocols.

VII. – WORKING HOURS DISTRIBUTED BY ACTIVITY

Actividad	Presencial (horas)	Trabajo autónomo (horas)	Créditos (horas)
Lectures	25	30	2,2 (55)
Seminars	8	12	0,8 (20)
Tutorials/Guided work	2	3	0,2 (5)
Laboratory	28	24,5	2,1 (52,5)
Laboratory seminars	2	5,5	0,3(7,5)
Written assignments and exams preparation	4	6	0,4 (10)
Total	**69**	**81**	**6 (150)**

VIII.- METHODOLOGY

The contents of the subject will be given to the students in four types of in-person classes:

(a) **Lectures** will be given to the whole group and the students will be introduced to the fundamental contents of the subject. At the beginning of each unit, the main objectives will be clearly stated. At the end of each unit, exercises/problems that illustrate the contents developed in the lectures will be proposed. To make it easier to follow up the lectures, the student will be provided with some teaching material used by the teacher, either in photocopies or on the Virtual Campus. The lectures will be given using the blackboard and various audio-visual resources.

(b) **Seminars** will focus on solving numerical problems and questions based on the topics developed during the theoretical lectures and student participation will be encouraged by providing them in advance with a list of problems/exercises.

(c) **Tutorials** will be used to discuss with the teacher the proposed problems and questions related to the course curriculum, as well as specific practical examples.

(d) **Laboratory** practices will be performed in 4-hour sessions. Prior to these practical sessions, a series of seminars will be held for student preparation. Once the practical sessions have finished, a seminar will be held for the discussion of the results obtained in the inter-comparison exercises. The Analytical Chemistry Department has prepared different audiovisual material for a better understanding of the laboratory practices.

During **self-study activities,** students must solve questions, problems or quizzes related to different topics of the subject proposed by the teacher which will be evaluated as independent work activities. The general objective of these activities is that the students acquire knowledge on the usefulness of the studied analytical methods during this course for their real application in areas such as environmental, clinical, food, and/or industrial analysis, etc. Searching for bibliographical material will be encouraged.

The **Virtual Campus** will be used as a mean to promote a fluent communication between the lecturer and the students and as an instrument to make available to students the material for both lectures and problem-solving classes.

IX.- BIBLIOGRAPHY

- **BASIC:**
  - Skoog, D. A.; Holler, F. J.; Holler, F. J.; Nieman, T. A.; Nieman, T. A. *Principles of Instrumental Analysis*, 5th ed.; Harcourt Brace College: Philadelphia, 1998.
  - Christian, G. D.; Christian, G. D.; O'Reilly, J. E.; O'Reilly, J. E. *Instrumental Analysis*, 2nd ed.; Allyn and Bacon: Boston, 1986.
- **COMPLEMENTARY:**
  - José M. Pingarrón Carrazón, P. Sánchez Batanero, "*Química Electroanalítica: Fundamentos y Aplicaciones*", Ed. Síntesis, 1999.

- Bard, A. J.; Faulkner, L. R.; Faulkner, L. R. *Electrochemical Methods: Fundamentals and Applications*, 2nd ed.; John Wiley & Sons: New York etc., 2000.
- Heineman, W. R.; Heineman, W. R.; Kissinger, P. T.; Kissinger, P. T. *Laboratory Techniques in Electroanalytical Chemistry*, 2nd.ed., rev. and aum.; Marcel Dekker: New York etc., 1996.
- Luis María Polo Díez, *"Fundamentos de Cromatografía",* Editorial Dextra, 2015.
- *Gas Chromatography*, First.; Poole, C. F., Ed.; Elsevier: Oxford, 2012.
- *Liquid Chromatography: Fundamentals and Instrumentation*; Fanali, S., Haddad, P. R., Poole, C. F., Schoenmakers, P. J., Lloyd, D., Eds.; Elsevier Science: Amsterdam, 2013.
- Douglas A. Skoog, F. James Holler, Stanley R. Crouch, "*Principios de Análisis Instrumental*", Ed. Cengage Kearning, 6ª ed., 2008.
- Lucas Hernández Hernández, Claudio González Pérez, "*Introducción al análisis instrumental*", Ed. Ariel Ciencia, 1ª ed, 2002.
- Harris, Daniel, C.: "*Análisis Químico Cuantitativo*", 3ª ed., Ed. Reverté, 2007.
- Rouessac, Francis y Rouessac, Annick: *"Análisis Químico. Métodos y técnicas Instrumentales modernas"*, 1ª ed., McGraw Hill, 2003.
- Bob Ardrey, Liquid Chromatography-Mass Spectrometry: An introduction. Wiley 2003.
- R. Kellner, J-M. Mermet, M. Otto, M. Valcárcel, H.M. Widmer, eds. Analytical Chemistry. Wiley-VCH 2004, 2nd edition.

X.- ASSESSMENT PROCEDURE

Student's assessment will be performed by in-person and guided activities in which the student participates. The students' course grade will be set between 0 and 10 points. To pass the course it will be mandatory to get 5 out of 10 points.

Attendance to face-to-face activities (lectures, seminars, and tutorials) is mandatory.

To be able to access the final evaluation, the student must have participated in at least 70 % of the total face-to-face activities.

Students who have failed the theory and/or the laboratory will be entitled to a final exam of the failed part in the July call.

■ THEORY WRITTEN EXAMS: 65 %

In the ordinary call, a final exam will be held, which will constitute a **65 %** of the final grade for the subject. In order to access the overall grade for the subject, it will be necessary to obtain at least a grade of **4.5** in the final exam.

All the exams will consist of problems and theoretical questions related to the course content. The maximum score for each question will be given in the exams.

Evaluated competences:

CG3-MFQA, CG6-MFQA, CG7-MFQA
CE4-MFQA1, CE6-MFQA1, CE6-MFQA2, CE7-MFQA1
CT3-MFQA, CT11-MFQA

LABORATORY 20%

The attendance to the laboratory sessions and seminars is **mandatory**. An unjustified absence in the laboratory may be a sufficient reason to fail the course. Group changes will only be made with a certification that justifies the change.

The **laboratory** represents the **20%** of the final grade of this course. Before starting the laboratory sessions, the student must have watched the related videos uploaded on the Virtual Campus. Questions on these videos will be asked and graded, and the resulting grade that will be evaluated within the practical part of the laboratory. At the end of each laboratory practice, written questions related to the work performed can be asked. Besides, a laboratory report regarding the work carried out will be submitted. At the end of the laboratory sessions, there will be a written exam that includes the fundamentals, working methods and calculations performed in the laboratory, and that may include multiple choice questions.

If, during the report assessment, plagiarism is detected or the results and questions included in the report are not related to the work done in the laboratory, the student will **FAIL** the laboratory.

The **final score of the laboratory** will be the average of the written exam score (50%) and the score obtained in the practical sessions (50%), that will be calculated from the personal work of each student in the laboratory (active participation, questions, and report). To pass the laboratory a minimum score of 4 out of 10 points in the laboratory exam is required, as well as a minimum score of 5 in the laboratory practical work. **Failing the laboratory means failing the course**.

Students who have not passed the laboratory in the ordinary call must take, in the July call, a written/practical exam of the practices carried out in the laboratory.

For those students who fail the course but have passed the laboratory (with a minimum score of 5,0), **the laboratory score will be kept for one year**, and it will not be necessary to retake the laboratory.

Evaluated competences:

CG8-MFQA, CG9-MFQA, CG10-MFQA, CG11-MFQA, CG12-MFQA, CG13-MFQA
CE4-MFQA2, CE6-MFQA1, CE7-MFQA1-2
CT1-MFQA, CT2-MFQA, CT3-MFQA, CT5-MFQA, CT6-MFQA, CT7-MFQA, CT11-MFQA, CT12-MFQA

SELF-WORK, GUIDED ACTIVITIES AND ACTIVE PARTICIPATION: 15%

This section will consider problems, questions or quizzes proposed by the lecturer, for grading throughout the course of the different topics included in the subject syllabus, it constituting a **15 %** of the final grade of the course.

Evaluated competences:

CG3-MFQA, CG6-MFQA, CG7-MFQA, CG8-MFQA, CG12-MFQA, CG13-MFQA
CE6-MFQA, CE6-MFQA, CE7-MFQA
CT1-MFQA, CT2-MFQA, CT3-MFQA, CT5-MFQA, CT6-MFQA, CT7-MFQA: CT11-MFQA, CT12-MFQA

EXTRAORDINARY JULY EXAM

Students who have not taken, with the required attendance, the laboratory practical sessions will not be able to take this extraordinary exam.

As in the ordinary January exam, the written theory exam accounts for 65 % of the final score, and a minimum score of **4,5 out of 10** is required to be averaged with the remaining activities.
The assessment of self-work, guided activities and active participation during the course will be also taken into account in the extraordinary exam in July (15 %).

Students who have not passed the laboratory, if they have completed the required attendance to the practical sessions, will be entitled to a final theoretical and/or practical exam.

ACTIVITIES SCHEDULE

UNIT	ACTIVITY	HOURS	START*	END*
1. Fundamentals of the electrochemical analysis techniques	Lecture	5	1st week	3rd week
	Seminar/Tutorial	2		
2. Potentiometric and voltammetric techniques	Lecture	6	3rd week	5th week
	Seminar/Tutorial	2		
3. Introduction to chromatographic techniques	Lecture	4	6th week	8th week
	Seminar/Tutorial	2		
4. Gas and liquid chromatography	Lecture	6	8th week	13th week
	Seminario/Tutoría	2		
5. Chromatography coupled to mass spectrometry	Lecture	2	13th week	14th week
	Seminar/Tutorial	1		
6. Non-chromatographic separation techniques	Lecture	2	14th week	15th week
	Seminar/Tutorial	1		
Final Exam				

* Assuming 3 hours/week the first 7 weeks and 2 hours/week the remaining weeks

ACTIVITIES SUMMARY

Teaching activity	Associated competences	Lecturer activity	Student activity	Assessment procedure	P	NP	Total	C
Lectures	CG3, CG6, CG7, CG12, CG13 CE4, CE6, CE7 CT3	Explanation of theoretical concepts. Raising questions.	Participating in the questions posed by the teacher. Questions and doubts formulation.	Active participation in class evaluation the regarding the theoretical concepts	25	30	55	65%
Seminars	CG3, CG6, CG7, CG12, CG13 CE4, CE6, CE7 CT3	Application of the theory to problem solving. Raising questions..	Exercises and questions solving. Questions and doubts formulation.	Evaluation of the active participation in the resolution of practical exercises.	8	12	20	
Laboratory	CG9, CG10, CG11, CG12, CG13 CE6, CE7 CT1, CT2, CT3, CT5, CT6, CT7, CT11, CT12	Helping the student to perform the laboratory practices with explanations and methodological recommendations. Videos projection.	Completion of the proposed practices and delivery of the reports and posed questions.	Grading personal work, reports, and resolution of posed practical problems. Written exams.	28	24,5	52.5	20%
Laboratory seminars	CG12, CG13 CE6, CE7 CT1, CT2, CT3, CT5, CT6, CT7, CT11, CT12	Presenting practical aspects related to laboratory teaching. Inter-comparison exercise results discussion.	Solving practical problems and questions related to the laboratory teaching.	Attendance and evaluation of the answers to the problems related to the practices carried out. Final exam.	2	5,5	7,5	
Guided work	CG3, CG6, CG7, CG8, CG12, CG13 CE6, CE7 CT1, CT2, CT3, CT5, CT6, CT7, CT11, CT12,	Raising questions and problems. Critical evaluation of those.	Resolution of posed questions and problems individually or in groups.	Assessment of the work carried out.		6	6	15%
Tutorials	CG3, CG6, CG7, CG8, CG12, CG13 CE6, CE7	Raising questions and problems.	Resolution of posed questions and problems.	Grading of the exercises proposed by the teacher.	2	3	5	

Teaching activity	Associated competences	Lecturer activity	Student activity	Assessment procedure	P	NP	Total	C
	CT1, CT2, CT3, CT5, CT6, CT7, CT11, CT12							
Exams	CG3, CG6, CG7, CG12, CG13, CE4, CE6, CE7 CT3	Exams design and correction. Student knowledge evaluation.	Exams preparation and execution.	Evaluation of the performed exams.	4		4	65%
P : In-person; NP: Self-study; C: Evaluation