



Course Guide

ANALYTICAL CHEMISTRY II ANALÍTICA II



FACULTY OF CHEMICAL SCIENCES
UNIVERSIDAD COMPLUTENSE DE MADRID
ACADEMIC YEAR 2022-2023



I.- IDENTIFICATION

COURSE NAME:	Analytical Chemistry II
CREDITS (ECTS):	9
CHARACTER:	Compulsory
SUBJECT:	Analytical Chemistry
MODULE:	Fundamental
UNDERGRADUATE DEGREE:	Bachelor's Degree (BS) in Chemistry
SEMESTER/TERM:	First semester (third year)
DEPARTMENT/S:	Analytical Chemistry

LECTURERS:

Subject and Laboratory Coordinator	Lecturer: ÁNGEL JULIO REVIEJO GARCÍA Department: Analytical Chemistry Office Number: QA-316 e-mail: reviejo@ucm.es
---	---

Group E

Theory Seminar Tutorial	Lecturer: REBECA M. TORRENTE RODRÍGUEZ Department: Analytical Chemistry Office Number: QA-422A e-mail: rebecamt@ucm.es
Theory Seminar Tutorial	Lecturer: MARIA GAMELLA CARBALLO Department: Analytical Chemistry Office Number: QB-435 e-mail: mariagam@ucm.es

Laboratory QA307

Gruop	Semester	Teacher	e-mail	Office	Depart ment
E1	1º	Tamara Fernández Bautista	tamarf02@ucm.es	QA-416A	QA
E2	1º	Mª Raquel Mateos Briz	marmat07@ucm.es	QA-402	QA
E3	1º	Emma Gracia Lor/ Juan José Rodriguez Bencomo	emgracia@ucm.es / juanjr10@ucm.es	QA-402/ QA-321A	QA
E4	1º	Fernando Navarro Villoslada	fenavi@ucm.es	QB-342D	QA



II.- OBJECTIVES

■ GENERAL OBJECTIVE

To introduce the students in the basic concepts of the main optical techniques used in chemical analysis, as well as to optimize, develop and apply measurement processes for obtaining quality chemical-analytical information.

It is also expected that the student will acquire the capacity to solve specific analytical problems in diverse areas including clinical, agri-food, toxicological, environmental, and industrial fields, through the employment of different optical methods.

Students' acquisition of correct working habits and clear knowledge of the laboratory work and safety regulations.

■ SPECIFIC OBJECTIVES

- Provide a solid basis about the general methodology of chemical analysis, covering from sampling to data interpretation.
- Understand and apply the basics of chemometrics for solving problems in Analytical Chemistry.
- Understand and utilize general principles of instrumental analysis.
- Understand the rationale and applications of the main optical methods.
- Understand the rationale and applications of mass spectrometry-based analysis.
- Learn how to validate an analytical method by evaluating its analytical characteristics.
- Train students to develop required optical method-based skills to be applied in the laboratory with the aim of solving specific analytical problems.
- Submit suitable reports containing obtained experimental results according to quality metrological aspects.
- Safely handle of chemical substances and assessing the hazardous effects of chemicals and chemical processes.

III.- BACKGROUND KNOWLEDGE AND RECOMMENDATIONS

■ BACKGROUND KNOWLEDGE:

It is advisable to have previous knowledge on spectroscopy taught in the course of *Physical Chemistry I* from the compulsory subject of Physical Chemistry included in the Fundamental Module.

■ RECOMMENDATIONS:

It is advisable to have successfully completed the following subjects: *General Chemistry*, *Basic Laboratory Operations* and *Analytical Chemistry I*.

IV.- CONTENTS

■ BRIEF DESCRIPTION:

***Theoretical contents:***

General basics of instrumental analysis. Introduction to chemometrics. Optical methods of analysis.

Laboratory contents:

Application of the main optical methods for solving specific problems of analytical chemistry relevance in the industrial, agri-food, clinical, environmental, and social concerns.

**■ SYLLABUS:****Theory****Unit 1: Principles of instrumental analysis**

- Basics.
- Classification of instrumental techniques.
- Basic components of analytical instruments.
- Signal-to-analyte concentration ratio.
- Selection of an analytical method according to its characteristics.

Unit 2: Introduction to Chemometrics

- Statistical data treatment. Variance and mean comparison.
- Univariate calibration. Statistical parameters.
- Basics of intercomparison exercises.

Unit 3: Introduction to optical techniques

- Electromagnetic radiation-matter interaction: adsorption and emission of electromagnetic radiation.
- Classification of optical methods
- Instrumentation used in analytical spectroscopy.

Unit 4: Ultraviolet-visible (UV-Vis) and infrared (IR) molecular spectroscopy-based analytical techniques.

- UV-Vis-based molecular absorption spectroscopy. Measurements of transmittance and absorbance. Quantitative analysis' basic law. Beer's Law and deviations from Beer's Law. Specific instrumentation. Absorbing species. Analytical applications.
- Infrared (IR) spectroscopy. IR absorption. Reflection spectrometry. Specific instrumentation. Fourier transform. Analytical applications.

Unit 5: Molecular luminescence spectroscopy-based analytical techniques

- Theory of photoluminescence. Factors affecting photoluminescence.
- Emission intensity-concentration ratio.
- Specific instrumentation.
- Analytical applications.
- Chemiluminescence: applications.

Unit 6: Atomic spectroscopy-based analytical techniques

- Types of atomic spectra. Comparison between atomic and molecular spectra. Atomic line broadening.
- Flame-based atomization spectroscopy: atomic absorption, atomic emission, and atomic fluorescence spectrometry.
- Atomic absorption spectroscopy: flame-, electrothermal-, hydride-, and cold-vapor-based atomization. Specific instrumentation.
- Atomic emission spectroscopy: atomization sources. Inductively coupled plasma (ICP)-atomic emission spectroscopy. Specific instrumentation.
- Analytical applications.

Unit 7: Introduction to mass spectrometry



- Introduction
- Mass spectrometers. Ion sources, mass analyzers, ion detectors.
- Atomic mass spectrometry. ICP-MS coupling.
- Molecular mass spectrometry. Specific features.
- Analytical applications.

Laboratory

Students will carry out a total of seven laboratory practices divided into seven 4-hour sessions.

Established laboratory practices include application of atomic and molecular spectroscopic techniques for the determination of metallic ions and both organic anions and molecules in biological, environmental and foodstuff samples.

Cooperative intercomparison exercises will be conducted for sample data treatment. The obtained results will be discussed in a seminar.

- *Lab practice 1:* Determination of phosphorus in soil samples by UV-Vis molecular absorption spectrophotometry after molybdenum blue formation. *Intercomparison exercise' participation.*
- *Lab practice 2:* Determination of selenium in biological samples by hydride generation atomic absorption spectroscopy (HG-AA). *Intercomparison exercise' participation.*
- *Lab practice 3:* Mercury determination in biological samples by cold vapor atomic absorption spectroscopy. *Intercomparison exercise' participation.*
- *Lab practice 4:* Determination of zinc, calcium, and iron in powdered milk samples by flame- and graphite furnace-based atomic absorption spectroscopy. *Intercomparison exercise' participation.*
- *Lab practice 5:* Atomic and molecular emission spectroscopy: fluorimetric determination of quinine and potassium in tonic water and wine samples, respectively.
- *Lab practice 6:* Determination of *trans* fatty acids in butter and/or margarine by Fourier transform infrared spectroscopy (FTIR).
- *Lab practice 7:* Determination of Fe(II) in wine samples by UV-Vis molecular absorption spectrophotometry after formation of a complex with 1,10-phenanthroline.

V.- COMPETENCES

■ GENERAL:

- **CG3-MFQA:** To rigorously express the acquired knowledge in Analytical Chemistry to be clearly understood in multidisciplinary areas.



- **CG5-MFQA:** To demonstrate knowledge and understanding of the essential facts, concepts, fundamentals, and theories related to Analytical Chemistry.
- **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.
- **CE6-MFQA1:** To apply the basics of the main optical methods.
- **CE6-MFQA2:** To propose a suitable optical method for the identification or quantification of a given target analyte.
- **CE7-MFQA1:** To apply the basics of chemometrics as tool for metrology and quality management.

■ **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.- RESULTADOS DEL APRENDIZAJE

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

Theory

- Apply the general methodology regarding chemical analysis.
- Classify the different instrumental methods.
- Apply basic statistics analysis to instrumental analysis.
- Use optical methods as tool to obtain both qualitative and quantitative information.
- Use statistical software to process quantitative measurement data.
- Express adequately the obtained results of the analysis and their uncertainty.
- Evaluate and compare the analytical characteristics of the different optical methods and mass spectrometry.
- Analyze the influence of the different parameters that affect optical and mass spectrometry measurements.
- Identify the structural characteristics of a molecule for its detection using optical methods and mass spectrometry.
- Assess the limitations of the different optical methods and mass spectrometry.
- Compare different optical methods.
- Select the appropriate instrumental method for the analysis of complex samples.
- Use appropriate optical and mass spectrometry based-methods depending on the analyte concentration and type of sample.

Practice

- Use the most appropriate method for sample preparation.
- Apply the safety basic laboratory rules and use the laboratory waste disposal protocol.
- Use the instrumentation of the different instrumental optical techniques.
- Analyze and compare the results obtained in the analysis.
- Apply statistical tools and software in instrumental analysis.
- Analyze and compare the obtained results in intercomparison exercises.
- Prepare laboratory reports.

VII. – WORKING HOURS DISTRIBUTED BY ACTIVITY



Activity	Attendance (hours)	Self-study (hours)	Credits (hours)
Lectures	46	54	4 (100)
Seminars	7	5,5	0,5 (12,5)
Tutorials/Guided work	3	4,5	0,3 (7,5)
Laboratory	28	24,5	2,1 (52,5)
Laboratory seminars	3	4,5	0,3 (7,5)
Written assignments and exams preparation	6	39	1,8 (45)
Total	93	132	9 (225)

VIII.- METHODOLOGY

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

- 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.
- 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.
- 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.
- 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 intercomparison exercises. The Department of Analytical Chemistry 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.



IX.- BIBLIOGRPHY

■ BASIC:

- [Skoog, Douglas A.; Holler, F. James y Crouch, Stanley R.: "Principles of Instrumental Analysis", 6ª ed., Ed. Cengage Kearning, 2008.](#)
- [Harris, D. C.: "Quantitative Chemical Analysis", 7th ed.; W.H. Freeman: New York, 2007.](#)
- [Miller, James N. y Miller, Jane C.: "Statistics and Chemometrics for Analytical Chemistry", 4ª ed, Ed. Prentice Hall, 2010](#)

■ COMPLEMENTARY:

- Skoog, Douglas A.; West, Donald M. F.; Holler, James y Crouch, Stanley R.: "*Fundamentals of Analytical Chemistry*", 8ª ed., Ed. Thomson, 2004.
- Rubinson, Kenneth A. y Rubinson, Judith F.: "*Análisis Instrumental*", 1ª ed., Ed. Prentice Hall, 20021ª ed., Ed. Prentice Hall, 2002.
- Sémus Higson, P.J.: "*Analytical Chemistry*", 1ª ed., Oxford University Press, 2004.
- Rouessac, Francis y Rouessac, Annick: "*Análisis Químico. Métodos y Técnicas Instrumentales Modernas*", 1ª ed., Ed. McGraw Hill, 2003.
- Mongay Fernández, Carlos: "*Quimiometría*", Universidad de Valencia, 2005.
- Stephen L.R. Ellison, Vicki J. Barwick y Trevor J. Duguid Farrant: "*Practical Statistics for the Analytical Scientist*", 2º ed., RSC Publishing, 2009.
- Angel Ríos, Mª Cruz Moreno y Bartolomé Simonet, Coordinadores, Técnicas espectroscópicas en Química Analítica, volumen II: Espectrometría atómica de iones y electrones. Editorial Síntesis, 2012.
- R. Kellner, J-M. Mermet, M. Otto, M. Valcárcel, H.M. Widmer, eds. Analytical Chemistry. Wiley-VCH 2004, 2nd edition.
- Hernández Hernández, Quantitative Chemical Analysis ", 1ª ed., Ed. Ariel Ciencia, 2002.

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 take the ordinary final exam in January, the student must have participated in at least 70% of the face-to-face activities.

■ WRITTEN EXAMS

65 %

January Ordinary exam: There will be only one final exam

The **final exam** represents the 65 % of the total grade of the course. In the final exam a minimum score of **4,5 out of 10** points will a prerequisite for averaging the remaining activities.



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, CG5-MFQA, CG6-MFQA, CG7-MFQA, CG12-MFQA, CG13-MFQA
CE4-MFQA1, CE6-MFQA1, CE6-MFQA2, CE7-MFQA1
CT3-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. These videos contain practice-related questions that will be used to assess the laboratory sessions. 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. At the end of the laboratory and as an additional activity, a satisfaction survey will be performed.

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.

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.

Students who have not passed the laboratory in January, provided they have completed the whole laboratory sessions, must take a written and/or practical exam in the extraordinary July exam on the practices performed 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.

Associated competences:

CG9-MFQA, CG10-MFQA1, CG10-MFQA2, CG11-MFQA, CG12-MFQA, CG13-MFQA
CE6-MFQA1, CE6-MFQA2, CE7-MFQA1
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 considers problems, questions or quizzes proposed by the professor, for grading throughout the course of the different topics included in the syllabus of the subject, it constituting **15%** of the final grade of the course.

Assessed competencies:

CG3-MFQA, CG5-MFQA, CG6-MFQA, CG7-MFQA, CG8-MFQA, CG12-MFQA, CG13-MFQA

CE6-MFQA, CE6-MFQA, CE7-MFQA1

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.

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	BEGGINIG	END
1. Principles of instrumental analysis	Lectures	2	1 ST week	1 ST week
2. Introduction to chemometrics	Lectures	8	1 ST week	3 rd week
	Seminar	2		
3. Introduction to optical techniques	Lectures	6	4 TH week	5 TH week
	Seminar	2		
4. Ultraviolet-visible (UV-Vis) and infrared (IR) molecular spectroscopy-based analytical techniques.	Lectures	9	5 TH week	8 TH week
	Seminar	2		
5. Molecular luminescence spectroscopy-based analytical techniques	Lectures	4	8 TH week	10 TH week
	Seminar	1		
6. Atomic spectroscopy-based analytical techniques	Lectures	12	10 TH week	13 TH week
	Seminar	2		
7. Introduction to mass spectrometry	Lectures	5	13 TH week	14 TH week
	Seminar	1		
Final exam				



SUMMARY OF THE ACTIVITIES

Teaching activity	Associated competences	Lecturer activity	Student activity	Assessment procedure	P	NP	Total	C
Lectures	CG3, CG5, CG6, CG7, CG12, CG13 CE4, CE6, CE7 CT3	Explanation of theoretical concepts. Raising of questions.	Participating in the questions posed by the teacher. Questions and doubts	Evaluation of the active participation in class regarding the theoretical concepts.	46	54	100	65%
Seminars	CG3, CG5, CG6, CG7, CG12, CG13 CE4, CE6, CE7 CT3	Application of the theory to problem solving. Raising questions.	Exercises and questions solving. Questions and doubts.	Evaluation of the active participation in the resolution of practical exercises.	7	5,5	12,5	
Laboratory	CG9, CG10, CG11, CG12, CG13 CE6, CE7 CT1, CT2, CT3, CT5, CT6, CT7, CT11, CT12	Help the student perform the laboratory practices with explanations and methodological recommendations. Video 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	CG9, CG10, CG12, CG13 CE6, CE7 CT1, CT2, CT3, CT5, CT6, CT7, CT11, CT12	Presenting practical aspects related to laboratory teaching. Results discussion.	Attendance and participation thorough raising questions and doubts related to the laboratory practices.	Evaluation of the active participation during the seminars related to laboratory practices.	3	4,5	7,5	
Guided work	CG3-, CG5, CG6, CG7, CG8, CG12, CG13 CE6, CE7 CT1, CT2, CT3, CT5, CT6, CT7, CT11, CT12,	Development and proposal of questions, problems, and controls. Critical evaluation of the same.	Written resolution of the proposed problems and questions, to be carried out individually or in groups. Active participation.	Assessment of the performed work		39	39	15%
Tutorial	CG3, CG5, CG6, CG7, CG8, CG12, CG13 CE6, CE7 CT1, CT2, CT3, CT5, CT6, CT7, CT11, CT12	Raising questions and problems.	Resolution of posed questions and problems. Active participation	Grading of the exercises proposed by the teacher.	3	4,5	7,5	
Exams	CG3, CG5, CG6, CG7, CG12, CG13, CE4, CE6, CE7 CT3	Design and exams correction. Student knowledge evaluation.	Exam preparation and execution.	Evaluation of the performed exams.	6		6	65%

P : In-person; NP: Self-study; C: Evaluation

