FACULTAD DE
CIENCIAS QUÍMICAS

ANALYTICAL CHEMISTRY I

COURSE GUIDE

BSc Chemistry

Academic Year 2023-2024

UNIVERSIDAD
COMPLUTENSE
MADRID

I.- IDENTIFICATION

COURSE NAME: Química Analítica I
CREDITS (ECTS): 9
CHARACTER: Compulsory
SUBJECT: Analytical Chemistry I
MODULE: Fundamental
UNDERGRADUATE DEGREE: Bachelor´s Degree (BS) in Chemistry
SEMESTER/TERM: Annual (second year)
DEPARTAMENT/S: Analytical Chemistry

LECTURERS:

Coordinator	**Lecturer:** **Department:** **Office number:** **e-mail:**	Mª TERESA PÉREZ CORONA Analytical Chemistry QA-319B mtperezc@ucm.es
Laboratory Coordinator	**Lecturer:** **Department:** **Office number:** **e-mail:**	ARACELI GONZÁLEZ CORTÉS Analytical Chemistry QA-322D aracelig@ucm.es
Group E		
1st Term **Lectures** **Tutorials** **Seminars**	**Lecturer:** **Department:** **Office number:** **e-mail:**	MARÍA LUZ MENA FERNÁNDEZ Analytical Chemistry QB-342F mariluz@ucm.es
2nd Term **Lectures** **Tutorials** **Seminars**	**Lecturer:** **Department:** **Office number:** **e-mail:**	MARÍA GAMELLA CARBALLO Analytical Chemistry QB-439 mariagam@ucm.es

Laboratory QA307

Group	Semester	Teacher	e-mail	Office	Department
E1	2º	Verónica Serafín González-Carrato	veronicaserafin@ucm.es	QA-435	QA
E2	2º	Emma Gracia Lor	emgracia@ucm.es	QB-405	QA
E3					
E4					

II.- OBJETIVES

GENERAL OBJETIVE:

The main aim is to introduce students to the methodology of Analytical Chemistry, so that they acquire a clear and up-to-date knowledge of the analytical process, its principles, and applications to quantitative analysis of titrations and gravimetric methods. The basic aspects of some sample treatment methods will be studied.

Students are also expected to become proficient in laboratory work and analytical problem solving, and to learn how to choose the most appropriate analytical method in selected situations.

After completing the course, the student should be able to interrelate the different types of equilibria in solution, to calculate the concentration of the different species and to select analytical methodologies based on chemical equilibria for the determination of compounds in different samples. At the end of the syllabus, the student should have acquired adequate training to understand Analytical Chemistry subjects in higher courses.

SPECIFIC OBJECTIVES:

The specific objectives are to:

- Become familiar with the general methodology of chemical analysis, from obtaining the sample to the interpretation of results.
- Familiarize the student with the evaluation of the analytical characteristics of an analytical method.
- Become familiar with the application of ionic equilibria to titrations and the importance of titration and gravimetric analyses as absolute methods of analysis.
- Understand the influence of secondary reactions in each of the equilibria and to evaluate their analytical implications.
- Know the sample treatments that allow the application of the methods of analysis studied.
- Acquire practical training in titrations and gravimetric analysis methods.

III.- PREVIOUS KNOWLEDGE AND RECOMMENDATIONS

PREVIOUS KNOWLEDGE:

Nomenclature and chemical formulation (both inorganic and organic). Periodic table of the elements and most frequent oxidation states. Adjustment of reactions. Expressions of Concentration. Course in *General Chemistry*.

RECOMMENDATIONS:

It is recommended to have passed the following courses in the first year: General Chemistry, Basic Laboratory Operations and Applied Computer Chemistry.

IV.- CONTENTS

BRIEF DESCRIPTION:

Theoretical contents:

The Analytical Process and measuring and transducing of the Analytical Signal. Chemical equilibria in aqueous solutions and their application to quantitative analysis. Titrations and gravimetric analyses. Liquid-liquid equilibria. Sample pretreatment.

Laboratory contents:

Examples of applications to acid-base, complexometric, precipitation, and redox equilibria and titrations. Examples of gravimetric applications. Examples of sample pretreatment methodologies.

SYLLABUS:

THEORY

Unit 1: Analytical Chemistry and the Analytical Process.

- Introduction to Analytical Chemistry.
- Aims and objectives of Analytical Chemistry.
- The Analytical Process.

Unit 2: Introduction to Sample Pretreatment.

- Introduction to Sample Pretreatment.
- Sample preparation methods for inorganic compounds.
- Sample preparation methods for organic compounds.

Unit 3: Acid-Base Equilibria for Polyprotic Species.

- Polyprotic acids or bases: methods for calculating pH and the concentration of species in polyfunctional acid and base equilibria.
- Buffer solutions for polyprotic species.
- The composition of an anionic system in equilibrium (polyprotic acids, bases, or complex ions) as a function of the pH value.

Unit 4: Applications of Acid-Base Titrations.

- Introduction.
- Acid-Base Titrations.
- Titration curves of polyprotic acids and bases.
- Acid-Base Indicators: detection of the end point. Potentiometric titrations.
- Applications of Acid-Base Titrations.

Unit 5: Complexes in Analytical Chemistry.

- Types of complexes/ligands and equilibria constants.
- Metal chelate complexes.
- Conditional Formation Constant

Unit 6: Applications of EDTA Titrations.

- Complexometric titration curves.
- Metallochromic indicators.
- Types of complexometric titrations.

- Applications of complexometric EDTA titrations.

Unit 7: Precipitation Equilibria and Applications: Precipitation Titrations and Gravimetric Analyses.

- Solubility and solubility product.
- Conditional solubility product.
- Species separation by precipitation.
- Precipitation titrations curves. Examples.
- Gravimetric analyses: types of gravimetry.
- Practical Gravimetric analyses: precipitation and volatilization.

Unit 8: Redox Equilibria and applications of Redox Titrations.

- Standard Conditional Potential.
- Redox systems of water.
- Redox titration curves. Redox indicators.
- Oxidizing and reducing agents.
- Applications of redox titrations.

LABORATORY

Practical sessions in the laboratory

1. Determination of protein nitrogen in flour by using the Kjeldahl´s method.
2. Determination of vitamin C (ascorbic acid) in pharmaceutical preparations by redox titration.
3. Determination of water hardness by complexometric titration.
4. Potentiometric titration of a chloride-iodine mixture.
5. Gravimetric determination of Ni(II) in steel.
6. Indirect determination of Ca in milk by redox titration.

V.- COMPETENCIES

GENERAL:

- **CG1-MF1:** To recognize chemical-analytical processes in everyday life.
- **CG2-MF1:** To recognize the importance of Analytical Chemistry in different contexts and relate it with other areas of knowledge.
- **CG5-MF1:** To demonstrate knowledge and understanding of the essential facts, concepts, principles, and theories related to Analytical Chemistry.
- **CG6-MF1:** To analyze and solve qualitative and quantitative problems.
- **CG7-MF1:** To recognize and assess new analytical problems and plan strategies to solve them.
- **CG8-MF1:** To evaluate, interpret and synthetize data and chemical information in Analytical Chemistry.
- **CG9-MF1:** To demonstrate knowledge of laboratory materials and practical skills in Analytical Chemistry.
- **CG10-MF1:** Safe handling of chemical materials.
- **CG10-MF2**: To recognize and assess hazards in the use of chemicals and laboratory procedures.

- **CG11-MF1:** To handle basic analytical instrumentation.
- **CG12-MF1:** To interpret data from observations and measurements in the analytical laboratory.
- **CG13-MF1:** To develop and implement good scientific practice in measurement and experimentation in Analytical Chemistry.

■ **SPECIFIC:**

- **CE4-MFQA1:** To describe the steps of the analytical process and know how to assess the importance of each of them.
- **CE4-MFQA2:** To apply the methodologies and recognize the problems associated with sample collection and processing.
- **CE5-MFQA1:** To apply the knowledge acquired in the study of chemical equilibria in solution to the resolution of quantitative analytical problems using gravimetric and titration methods.
- **CE6-MFQA2:** To propose a titration or gravimetric analytical technique suitable for the quantification of an analyte.
- **CE7-MFQA1:** To apply basic concepts of Chemometrics as a tool to solve analytical, metrology and quality management problems.

■ **GENERIC:**

- **CT1-MF1:** To prepare and write analytical reports of a scientific and technical nature.
- **CT2-MF1:** To work as a team.
- **CT3-MF1:** To demonstrate critical thinking and self-criticism.
- **CT5-MF1:** To manage quality chemical information, bibliography, specialize databases and accessible resources from Analytical Chemistry.
- **CT6-MF1:** To assess the importance of Analytical Chemistry in the industrial, environmental, and social context.
- **CT7-MF1:** To use the tools and computer packages for the treatment of experimental results.
- **CT11-MF1:** To develop self-learning capacity.
- **CT12-MF2:** To develop sensitivity to environmental issues related to Analytical Chemistry.

VI.- LEARNING OUTCOMES:

Once this course passed, the student must be able to:

- Describe the importance of Analytical Chemistry and the role of the analytical chemist in solving problems of social, economic, and scientific-technical interest.
- Explain the basic operations of the Analytical Process.
- Apply the fundamentals of ionic equilibria in solution to qualitative and quantitative analysis.
- Apply the concepts of accuracy, precision, sensitivity, and selectivity.
- Describe the basic sample treatment techniques and select the most appropriate one according to the different nature of the samples and/or the compounds to be determined.
- Calculate the pH and concentrations of species in the equilibrium of polyprotic systems.
- Explain the fundamentals of titrations, acid-base titration curves and end-point indicators.

- Describe the most important applications of acid-base titrations.
- Explain the fundamentals of complexes formation equilibria.
- Define and apply the conditional constants of complexes formation.
- Describe the most important applications of complexometric titrations.
- Describe the importance of precipitation in determination and separation methods in Analytical Chemistry.
- Apply the concepts of solubility product and conditional solubility.
- Explain the principles of gravimetries and their different types.
- Describe the most important gravimetric applications.
- Assess the stability of species in solution from the point of view of their redox reactions.
- Calculate the conditional normal potential of a semi-reaction.
- Describe the main titration reagents used in volumetric redox methods.
- Describe the most relevant applications of redox titrations.
- Properly use sample digestion/dissolution methods applying the necessary safety measures.
- Use appropriate titration methods for the determination of different species.
- Properly use of gravimetric methods of analysis.
- Calculate the concentration of analytes in the samples.
- Perform statistical studies of the results obtained in the analysis to assess their precision and accuracy.
- Interpret the results obtained in titration and gravimetric analyses.
- Prepare analytical reports of the results obtained.

VII. – WORKING HOURS DISTRIBUTED BY ACTIVITY

Activity	Attendance (hours)	Self-study (hours)	Credits (hours)
Lectures	42	58	4
Seminars / Problem classes	20	20	1,6
Tutorials / Guided work	8	12	0,8
Laboratory	18	13,5	1,26
Laboratory seminars	4	6	0.4
Written assignments and exams preparation	6	17,5	0,94
Total	**98**	**127**	**9**

VIII.- METHODOLOGY

The contents of the course are presented face-to-face to students divided in three types of classes:

Lectures will be given to the whole group and the student will be introduced to the fundamental contents of the subject. At the beginning of each unit, the contents and the main objectives will be clearly stated.

Exercises and questions that illustrate the contents developed in the lectures will be solved in **seminars and problem classes**. A list of problems/exercises to solve will be given to the students periodically before the class. Some of these exercises will be collected by the lecturer for assessment.

As a complement to the **self-study** carried out by the student, and to promote the development of teamwork, the elaboration and presentation of a *written assignment* or some other alternative activity about the contents of the course will be proposed as a **guided activity**. To make it easier for students to follow the face-to-face classes, they will be provided with the teaching material used by the lecturer, either in photocopies or in the Virtual Campus. The explanation of each of the topics will be done using the blackboard and PowerPoint presentations.

Seminars will be given to the whole group. In them, numerical problems will be explained in which the topics developed in the theory classes will be applied. Students will periodically be provided with a list of problems/exercises so that they can try to solve them before class. Different methods may be used for student assessment in seminar classes, such as the following:

- propose to the student the solution of some of the problems in class, discussing the procedure followed, the result obtained and its meaning.
- discuss the results obtained by different students working in small groups.
- periodically collect exercises or tests.
- other procedures as deemed appropriate by the teachers.

The **laboratories** will be taught in 3-hour sessions in which students will apply the analytical knowledge acquired through classroom activities and their personal work to the determination of some species of industrial, environmental, etc. interest.

In the **guided work**, students will have to solve proposed theoretical and practical problems.

The lecturer will program **tutorials** about diverse activities that allow him/her to identify the strengths and weaknesses in the daily work of the students. Tutorials will also be available for students who individually wish to solve doubts that arise during the study.

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:

- Robert A. Kellner; Jean-Michel Mermet; Matthias Otto; Miguel Varcárcel; H. Michael Widmer. *Analytical Chemistry. A Modern Approach to Analytical Science*", 2nd ed., Ed. Wiley-VCH, 2004.

- Skoog, D. A.; Skoog, D. A. *Analytical Chemistry: An Introduction*, 7th ed.; Saunders Golden Sunburst Series; Saunders College Publishing, A Division of Harcourt College: Fort Worth, 2000.
- Harris, D. C. *Quantitative Chemical Analysis*, 7th ed.; W.H. Freeman: New York, 2007.

■ **COMPLEMENTARY:**

- Douglas A. Skoog; Donald M. West; F. James Holler; Stanley R. Crouch. *Fundamentos de Química Analítica*, 8ª ed., Ed. Thomson, 2004.
- Harris, D. C.; Berenguer Murcia, Á.; Berenguer Murcia, Á.; Berenguer Navarro, V.; Berenguer Navarro, V. *Análisis Químico Cuantitativo*, 3ª ed. (6ª ed. original), reimp.; Reverté: Barcelona etc, 2013.
- Silva, M.; Barbosa, J.: *"Equilibrios iónicos y sus aplicaciones analíticas",* 1ª ed., Ed. Síntesis, 2002.
- Yáñez-Sedeño, P.; Pingarrón, J. M.; Manuel de Villena, F. J.: "*Problemas resueltos de Química Analítica*", 1ª ed., Ed. Síntesis, 2003.
- Yáñez-Sedeño, P.; Pingarrón, J. M.; González Cortés, A.: "*300 Problemas resueltos de Química Analítica*", 1ª ed., Ed. Síntesis, 2022.
- As an additional complementary bibliography, students will be provided with specific bibliography for the preparation of the guided work.

X.- ASSESSMENT PROCEDURE

The student's academic performance and the final mark of the course will be computed in a weighted manner, considering the following percentages, which will be maintained in all the exams.

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.

■ **WRITTEN EXAMS:** **65 %**

Ordinary June exam: There will be two mid-term exams and a final exam. The mid-term exams passed will be released for the final exam in June. Students who pass both mid-term exams are not required to take the final exam. The compensation between mid-term exams will require a minimum score of 4 out of 10 in the mid-term exam not passed and an average of 5 out of 10 between both exams. In the final exam, students who have not passed one of the mid-term exams may choose, on the same day of the final exam, between taking the exam for the failed mid-term or for the whole subject. In the June final exam, whether the student takes only one midterm or the whole subject, it will be an essential requirement, to average with the remaining activities, to obtain a minimum score of 4.5 out of 10. Those who wish to take the final exam in June to obtain a higher score must do so with all the subject matter of the course. The exams will consist of questions on the application of concepts learnt during the course and related practical questions.

The grades of the activities planned for the evaluation of the course (mid-term exams, laboratories, tutorials, problem sets, etc.) will be communicated to the students sufficiently in advance of the final exam, so that they can plan their studies accordingly.

In particular, the scores of the mid-term exams will be communicated within a maximum period of 20 days, except in the case of the second mid-term exam, where the period may be shorter to adapt to the final exam.

In any case, the minimum period of seven days between the publication of the scores and the date of the final exam of the course will be respected.

Assessed competencies:

CG1-MF1, CG2-MF1, CG5-MF1, CG6-MF1, CG7-MF1, CG8-MF1.
CE4-MFQA, CE5-MFQA, CE6-MFQA, CE7-MFQA.
CT3-MF1, CT5-MF1, CT6-MF1, CT7-MF1, CT12-MF2.

■ GUIDED WORK: 15 %

Guided work carried out by the student will count for a maximum of 15 % of the final score. The student's skills in solving the proposed theoretical and practical problems, the student's participation and evaluation in the scheduled group tutorials and individual tutorials, and his or her participation in the theory and seminar classes will be graded.
Assessed competencies:

CG1-MF1, CG2-MF1, CG5-MF1, CG6-MF1, CG7-MF1, CG8-MF1.
CE5-MFQA, CE6-MFQA, CE7-MFQA.
CT1-MF1, CT2-MF1, CT3-MF1, CT5-MF1, CT6-MF1, CT7-MF1.

■ LABORATORY: 20 %

Attendance at the laboratory and its seminars is **compulsory**. An unexcused absence in the laboratory may be sufficient reason to fail the course. Group changes will only be made with certificates justifying the change.

At the end of the practical sessions in the laboratory, a report on the work carried out will be handed in and a written exam will be taken that covers some aspects of the fundamentals, working methods and numerical calculations of the practical sessions. The exam, the report and the active participation of the student will contribute a maximum of 20 % to the final score (10 % will correspond to the active participation in the laboratory and the report and the other 10 % to the exam score). Both parts will be compensable only if a score equal to or higher than 4.0 is achieved in each of them. Failure to achieve an overall laboratory grade of 4 (out of 10) will be grounds for 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.
If an overall laboratory grade of 5 or higher is achieved, the grade obtained will be retained for the following academic year, and it will not be necessary, in the event of having to repeat the subject, to retake the laboratory.

Assessed competencies:

CG9-MF1, CG10-MF1, CG10-MF2, CG11-MF1, CG12-MF1, CG13-MF1.
CE7-MFQA.
CT1-MF1, CT2-MF1, CT3-MF1, CT5-MF1, CT6-MF1, CT7-MF1, CT12-MF2.

EXTRAORDINARY JULY EXAM

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

Students who have passed the laboratory in June will not have to take the laboratory in July. Likewise, students who have passed the theoretical part of the course in June and have failed the laboratory will only be examined in the laboratory in July.

The written theory exam accounts for 65 % of the final score, and a score of 4.5 out of 10 is required to be averaged with the remaining activities.

Students who have failed the laboratory, if they have made the required attendance during the practical period, will be entitled to a final theoretical and/or practical exam.

ACTIVITY SCHEDULE

SECTION	ACTIVITY	HOURS	START	END
1. Analytical Chemistry and the Analytical Process	Lectures	4	1st week	2nd week
	Problem solving classes /Tutorials	1		
2. Introduction to Sample Pretreatment	Problem solving classes/ /Tutorials	4	2nd week	3rd week
3. Acid-Base Equilibria for Polyprotic Species **4. Applications of Acid-Base Titrations**	Lectures	10	4th week	9th week
	Problem solving classes/ /Tutorials	8		
5. Complexes in Analytical Chemistry **6. Applications of EDTA Titrations**	Lectures	9	10th week	15th week
	Problem solving classes/ /Tutorials	6		
7. Precipitation Equilibria and Applications: Precipitation Titrations and Gravimetric Analyses	Lectures	7	16th week	21st week
	Problem solving classes/ /Tutorials	4		
8. Redox Equilibria and Applications of Redox Titrations	Lectures	10	22nd week	30th week
	Problem solving classes/ /Tutorials	7		
OTHER ACTIVITIES*				
Solving proposed theoretical and practical problems.				
Written exams: two mid-term and one final theory exam and two final laboratory exams on dates determined by the Faculty.				

* The laboratories planning is published on the Faculty's website.

SUMMARY OF ACTIVITIES

Teaching activity	Associated competencies	Lecturer activity	Student activity	Assessment procedure	P	NP	Total	C
Lectures	CG1-MF1, CG2-MF1, CG5-MF1, CG6-MF1, CG7-MF1, CG8-MF1. CE4-MFQA, CE5-MFQA, CE6-MFQA, CE7-MFQA. CT3-MF1, CT5-MF1, CT6-MF1, CT7-MF1, CT12-MF2.	Presentation of theoretical concepts. Raising of questions. Questions to students during the class.	Note-taking. Participation in the questions asked by the teacher. Formulation of questions and doubts.	Evaluation of active participation in relation to theoretical concepts and class attendance.	42	58	100	
Problem solving classes/ Seminars		Presentation of theoretical concepts. Raising of questions. Questions to students during the class.	Note-taking. Participation in the questions asked by the teacher. Formulation of questions and doubts.	Evaluation of active participation in relation to the resolution of practical and numerical exercises and class attendance.	20	20	40	
Guided work	CG1-MF1, CG2-MF1, CG5-MF1, CG6-MF1, CG7-MF1, CG8-MF1. CE5-MFQA, CE6-MFQA, CE7-MFQA. CT1-MF1, CT2-MF1, CT3-MF1, CT5-MF1, CT6-MF1, CT7-MF1.	Preparation and proposal of problems and exercises. Critical evaluation of them.	Theoretical and practical problem solving.	Assessment of the work carried out.	-	17,5	17,5	15%
Tutorials	CG1-MF1, CG2-MF1, CG5-MF1, CG6-MF1, CG7-MF1, CG8-MF1. CE4-MFQA, CE5-MFQA, CE6-MFQA, CE7-MFQA. CT3-MF1, CT5-MF1, CT6-MF1, CT7-MF1, CT12-MF2.	Helping students to direct their study with explanations and bibliographical recommendations. Raising questions to be analyzed as a group.	Consultation with the teacher on the conceptual and methodological difficulties encountered when studying the course. Resolution of the raised questions.	Evaluation of the answers given by the student to the teacher's questions. Assessment of the competence demonstrated in learning the subject.	8	12	20	

Teaching activity	Associated competences	Lecturer activity	Student activity	Assessment procedure	P	NP	Total	C
Laboratory	CG9-MF1, CG10-MF1, CG10-MF2, CG11-MF1, CG12-MF1, CG13-MF1. CE7-MFQA. CT1-MF1, CT2-MF1, CT3-MF1, CT5-MF1, CT6-MF1, CT7-MF1, CT12-MF2.	Helping the student to carry out the internship with explanations and methodological recommendations.	Carrying out of the proposed practices and presentation of results and reports.	Attendance and evaluation of the answers to the practical problems posed.	18	13,5	31,5	20%
Seminars associated with the Laboratory	CG9-MF1, CG10-MF1, CG10-MF2, CG11-MF1, CG12-MF1, CG13-MF1. CE7-MFQA. CT1-MF1, CT2-MF1, CT3-MF1, CT5-MF1, CT6-MF1, CT7-MF1, CT12-MF2.	Solving practical problems related to the laboratory teaching. Final exam preparation.	Solving practical problems related to the laboratory teaching. Final exam completion.	Attendance and evaluation of the answers to the problems in relation to the practical exercises carried out. Final exam.	4	6	10	
Exams	CG1-MF1, CG2-MF1, CG5-MF1, CG6-MF1, CG7-MF1, CG8-MF1. CE4-MFQA, CE5-MFQA, CE6-MFQA, CE7-MFQA. CT3-MF1, CT5-MF1, CT6-MF1, CT7-MF1, CT12-MF2.	Proposal, invigilation and correction of the theory exam and seminars. Evaluation of the student.	Preparation and performance in the theory exams.	Evaluation of the exam	6	--	6	65%
P: In-class; NP: Self-study; C: Evaluation								

If the development of the academic year 2021-22 is affected by measures leading to non-attendance, the Course Guide will be adapted for its transition to online teaching and assessment, adopting measures like those included in the Addenda of the subjects of the Degree of the academic year 2019-20.