Universidad de Alcalá

COURSE GUIDE

ANALYTICAL TECHNIQUES

(Aprobada en CD el 18-06-2018)

DEGREE IN PHARMACY
University of Alcalá

Academic Year 2018/2019
2nd Year - Annual

COURSE DESCRIPTION

Name of the course:	**Analytical Techniques**
Code:	**570011**
Degree:	**DEGREE IN PHARMACY**
Department and Area of Knowledge:	**Analytical Chemistry, Physical Chemistry and Chemical Engineering**
Type of course:	**Compulsory**
ECTS: 12 ECTS (9 theory + 3 experimental)	
Course and period	**Second / Annual**
Instructor:	Dra. María Castro Puyana Dra. Ana María Díez Pascual Dra. Beatriz Jurado Sánchez
Coordinator:	Dra. Merichel Plaza del Moral **Dr. Miguel Ángel López Gil**
Schedule for Tutorials:	**Appointment with the instructor**
Language of Instruction:	**English**

1. PRESENTATION

This subject is designed to introduce students to the systematic approach of the analytical process and to the evaluation of analytical information with scientific criteria. In brief, this is a scientific discipline that develops and applies methods, instruments, and strategies to obtain information on the composition of nature of matter in space and time. Learning of this course will allow students to: i) know main tools for reliable chemical information, ii) use equilibrium constants to calculate concentrations and activities of chemical species in solution, iii) know and apply the fundamentals of volumetric and gravimetric analysis, iv) know and apply the different instrumental techniques used in quantitative and qualitative analysis of chemical species of pharmaceutical interest.

This course focuses on formulating questions, selecting and studying analytical techniques, understanding the analytical problem, and developing critical reasoning for the provision of analytical information. This matter is an essential element in the pharmacist's professional training covering clinical, industrial and environmental aspects.

Prerequisites and Recommendations (if applicable)

2. COMPETENCES AND LEARNING OUTCOMES

General Competences (Orden CIN/2137/2008, 3 de julio) and Learning Outcomes

1. Understand the principles and procedures for the analytical determination of compounds: analytical techniques applied to the analysis of water, food and environment.

2. Select the techniques and the procedures for the design, the implementation and the evaluation of reagents, methods and analytical techniques.

3. Know and apply the main techniques for investigating chemical structures including spectroscopy.

4. Identify, design, collect, analyse and produce active ingredients, drugs and other products and materials of health interest.

5. Learn about the origin, nature, design, acquisition analysis and control of medicines and health products.

Specific competences:

1. Understand and identify the importance of the stages of General Analytical Process.

2. Knowing the chemical equilibria in solution and their application in volumetric and gravimetric analyses.

3. Understand and use technical and bibliographic information related to analytical chemistry.

4. Ability to estimate the reliability of analytical results and developing factual report writing skills.

5. Ability to communicate ideas and express oral and written correctly.

6. Developing skills for teamwork.

7. Development of independent learning skills.

8. Initiation in the ability of argumentation supported by textbooks and other references given in the course.

9. Initiation and self-critical ability.

3. CONTENTS

Lectures:

THEMATIC UNIT I - INTRODUCTION AND BACKGROUND OF ANALYTICAL TECHNIQUES.

Chapter 1.- Introduction to Analytical Chemistry. Objectives of Analytical Chemistry. Analytical Chemistry in today's society. The analytical problem. The vocabulary of Analytical Chemistry. Classification criteria of Analytical Chemistry. Sources of Information in Analytical Chemistry.

Chapter 2.- The Analytical Process. General steps of the analytical process: sampling, previous operations, measurement and signal transduction, data acquisition and processing. Errors in Analytical Chemistry. Analytical properties.

Chapter 3.- The measurement in Analytical Chemistry: standards and traceability. Types of standards: basic chemicals and chemical-analytical. General properties and use of different standards.

Chapter 4.- General characteristics and objectives of qualitative analysis. Analytical properties in qualitative analysis. General characteristics and objectives of quantitative analysis. Quantification methodologies. Resolution of practical problems.

THEMATIC UNIT II – TITRIMETRIC AND GRAVIMETRIC ANALYSIS.

Chapter 5.- Introduction to titrimetric techniques. - Overview of titrimetry. Equivalence points and end points. Titration errors. Titration curves.

Chapter 6.- Acid-base titration. Titration curves of strong acids and bases and weak monoprotic acids and bases. Titration curves of polyprotic acids and bases. Titration of mixtures. Factors affecting the titration curves. Acid-base indicators. Acidic and basic titrants. Buffer solutions: buffer capacity and handling. Titration in nonaqueous media. Applications.

Chapter 7.- Complexation titrations. EDTA complexes of metal ions. EDTA titration curves. Conditional formation constant. Factors affecting the titration curves. Metallochromic indicators. EDTA titration methods. Applications.

Chapter 8.- Redox titrations. Overview. Calculating the titration curves. Factors affecting the titration curves. Redox indicators. Adjusting the titrant's oxidation state: auxiliary reagents. Applications with oxidizing and reducing titrants.

Chapter 9.- Precipitation titrations. - Titration curves. End point detection: Mohr, Volhard and Fajans methods. Applications.

Chapter 10.- Gravimetric methods. Overview. Types of gravmetric methods. Precipitation gravimetry: theory and practice. Formation and properties of precipitates. Homogeneous precipitation. Quantitative applications.

THEMATIC UNIT III – OPTICAL METHODS.

Chapter 11.- Overview. Transduction: concept and types. Classification of instrumental techniques. Signal and noise. Calibration in instrumental analysis.

Chapter 12.- Overview of spectroscopy. Electromagnetic radiation: wave and particle properties. The electromagnetic spectrum. Photons as signal source. Spectroscopy based on absorption. Spectroscopy based on emission. Instrumentation: basic components of spectroscopic instruments.

Chapter 13.- UV/Vis molecular absorption spectroscopy. Transmittance and absorbance. Absorbance and concentration: Beer's law. Beer's law and multicomponent samples. Limitations of Beer's law. Instrument designs for molecular UV/Vis absorption. Quantitative and qualitative applications.

Chapter. 14.- Infrared molecular absorption spectroscopy. Molecular vibrations. General approach to IR spectrum analysis. Instrument designs and components for infrared absorption. Applications.

Chapter 15.- Molecular emission spectroscopy. Overview of luminescent techniques. Photoluminescence: fluorescence and phosphorescence. Excitation and emission spectra. Instrumentation. Applications. Chemo- and bioluminescence.

Chapter 16.- Atomic absorption spectroscopy. Overview. Atomic spectra. Instrumentation: spectral line sources, flame atomizers, electrothermal atomizers. Interferences. Applications.

Chapter 17.- Atomic emission spectroscopy. Overview. Flame emission spectroscopy and Inductively Coupled Plasma (ICP). Instrumentation and applications.

THEMATIC UNIT IV – ELECTROANALYTICAL TECHNIQUES.

Chapter 18.- Overview of electrochemical methods. Key concepts for electrochemistry. Electrochemical cells. Electrolysis. Classification of electroanalytical techniques. Instrumentation. Electrodes: Classification. Reference electrodes. Indicator electrodes.

Chapter 19.- Potentiometric techniques. Direct potentiometry. Ion selective electrodes (ISEs). Measurement of pH. Gas sensing electrodes. Quantitative applications.

Chapter 20.- Voltammetric methods: fundamentals and key processes. Voltammetric techniques: Polarography, Hydrodynamic voltammetry, Cyclic voltammetry, Stripping voltammetry. Quantitative applications. Amperometry.

THEMATIC UNIT V – ANALYTICAL SEPARATIONS.

Chapter 21.- Overview of analytical separations. Chromatographic separation. Characteristic values of a chromatogram. Chromatographic theory. Classification of chromatographic techniques.

Chapter 22.- High Performance Liquid Chromatography (HPLC). Basic instrumentation and components: mobile phase, column, pumping system, injector and detector. Separation modes. Applications.

Chapter 23.- Gas chromatography (GC). Basic instrumentation and components: mobile phase, column, pumping system, injector and detector. Chromatographic process control. Derivatisation. Applications.

Chapter 24.- Electrophoretic techniques. Fundamentals of electrophoresis. Classical electrophoresis. Gel electrophoresis. Capillary electrophoresis. Applications.

THEMATIC UNIT VI – OTHER TECHNIQUES.

Chapter 25.- Turbidimetry and nephelometry: fundamentals, instrumentation and applications. Refractometry: instrumentation and applications. Polarimetry: fundamentals, instrumentation and applications. Spectropolarimetry. Circular dichroism. Applications.

Chapter 26.- Mass spectrometry (MS). Basic principles. Instrumentation. Coupled techniques. Applications.

Chapter 27.- Thermal analysis. Overview. Thermogravimetry. Differential thermal analysis. Differential scanning calorimetry. Instrumentation. Applications

Laboratory:

1. Preparation of a solution of acetate buffer and calculation of its buffer capacity.
2. Complexometric titrations. Water Hardness. EDTA titration of Ca^{2+} and Mg^{2+} in natural waters. Solving a mixture of zinc and nickel with sample separation (ion exchange resin) and EDTA titration.
3. Back redox titration. Analysis and control of vitamin C in tablets and raw material.
4. Precipitation and potentiometric titration. Analysis of Cl^- ions in soil.
5. Determination of iron in a synthetic sample. Gravimetric determination.
6. Solving a mixture by UV/Vis spectrophotometric analysis. Application of Beer's Law.
7. Determination of Na^+ and K^+ in natural waters and serum. Flame photometry.
8. Solving mixtures of acids through conductimetric titrations.
9. Determination of paracetamol in pharmaceuticals products. Cyclic voltammetry.
10. HPLC: simulation and key parameters for optimal separation. Analysis of pills containing caffeine, acetylsalicylic acid and paracetamol.
11. Determination of sulphates in natural waters. Turbidimetric analysis.

Seminars:

Seminars 1-2. - Exercises and case studies on chapters 2-4
Seminars 3-7. - Exercises and case studies on chapters 5-10.
Seminars. 8-11 - Exercises and case studies on chapters 11-17.
Seminars 12-13. - Exercises and case studies on chapters 18-20.
Seminars 14-15.- Exercises and case studies on chapters 21-24.
Seminar 16.- Exercises and case studies on chapters 25-27.

3.1. Organization of the course

Thematic Unit	Topics	Hours
Part I. INTRODUCTION AND BACKGROUND OF ANALYTICAL TECHNIQUES	• Chapters 1-4 • Seminars 1-2	• 9 hours (lectures) • 2 hours (seminars)

Part II. TITRIMETRIC AND GRAVIMETRIC ANALYSIS	• Chapters 5-10 • Seminars 3-7 • Practical work 1-5	• 15 hours (lectures) • 5 hours (seminars) • 18 hours (laboratory)
Part III. OPTICAL METHODS	• Chapters 11-17 • Seminars 8-11 • Practical work 6-7	• 13 hours (lectures) • 4 hours (seminars) • 6 hours (laboratory)
Part IV. ELECTRO-ANALYTICAL TECHNIQUES	• Chapters 18-20 • Seminars 12-13 • Practical work 8-9	• 5 hours (lectures) • 2 hours (seminars) • 6 hours (laboratory)
Part V. ANALYTICAL SEPARATIONS	• Chapters 21-24 • Seminars 14-15 • Practical work 10	• 9 hours (lectures) • 2 hours (seminars) • 3 hours (laboratory)
Part VI. OTHER TECHNIQUES	• Chapters 25-27 • Seminar 16 • Practical work 11	• 5 hours (lectures) • 1 hours (seminars) • 3 hours (laboratory)

4. TEACHING-LEARNING METHODOLOGIES. TRAINING ACTIVITIES

4.1. Distribution of the teaching (Number of hours)

Number of classroom hours:	• Number of lecture hours: 56 • Number of hours in seminars: 16 • Number of hours in laboratory: 36

	• Group Tutorials: 9
Number of hours of independent study:	• Number of hours of laboratory-related, independent work: 30 • Number of hours of independent study: 143 • Self-assessment tests and / or evaluation through the virtual platform: 10
Total	300 (12 ECTS)

4.2. Methodological strategies

Classroom	Small group (S): Two kinds of activities will be undertaken in classroom: lectures and seminars. During lectures, the teacher will present each chapter describing main items. During seminars, a particular analytical problem or practical case will be exposed and discussed. Active participation of students will be deeply encouraged Printed material: textbooks and specific material provided by the instructor will set the basis for the content of this course. Videos, demos, and tutorials accessible in the web. All materials discussed and presented will be available in the online course management platform.
Independent work	Students will analyse and study each chapter of the course using all available means of peer-reviewed information. Preferably, students will elaborate and deliver summaries and notes that can be discussed with the instructor

5. ASSESSMENT

PROCEDURES FOR EVALUATION

In each academic year the student has the right to have two calls, one ordinary and one extraordinary. The ordinary call will be based on continuous evaluation, except in those cases contemplated in the regulations for the evaluation of the UAH and students will have access to the final evaluation. To benefit from this final evaluation procedure, the student will have to request it in writing to Dean or Center Director in the first two weeks of the subject, explaining the reasons why the continuous assessment system will continue. In case there are students who for justified reasons have not formalized their registration on the date of the start of the course or the period of delivery of the subject, the term indicated is a computation from its incorporation to the degree.

Regular call:

Continuous assessment

The continuous assessment will follow the regulations for the evaluation of the UAH. Attendance to classes, seminars and tutoring is mandatory and only a maximum of 20% of absents will be allowed. The active participation of students in all face-to-face activities and work carried out, as well as the skills developed during the practical lessons, will be evaluated. The students must demonstrate a minimum level in the acquisition of the corresponding competences so that their global qualification is obtained.

The theoretical knowledge of the subject will be assessed through four (4) written partial tests. The ordinary call may be approved when the weighted average of all of them is greater than 5.0. To apply such average, it will be essential to have obtained a grade higher than 4.0 in each of the tests. Students will have an additional test, within the ordinary call, in which they will be able to recover a maximum of two partial tests.

The practical contents of the subject will be assessed through two partial tests of a practical nature in the laboratory.

Participate in the continuous evaluation supposes to consume the ordinary call. Continuous assessment students who wish to appear as not presented in this call must communicate it in writing at the Department's secretariat within the established deadline (towards the middle of the subject).

In case of not participation in the ordinary call, students will have the right to perform a final exam in the extraordinary call.

Final assessment

Exceptionally, students who have not opted for continuous assessment and are registered in the Dean's Office, will perform a global examination consisting of questions, problems and / or practical exercises that allow assessing the acquisition of the skills included in the teaching guide.

Extraordinary call:

There will be a global exam consisting of questions, problems and/or practical exercises that allow assessing the acquisition of the skills included in the teaching guide. This test will consist of two blocks: block I, corresponding to thematic units I and II; block II, corresponding to the thematic units III, IV and V. The students will be able to present themselves, independently, to one or both blocks to recover the part not exceeded in the ordinary call. Students who have completed the practices and have failed must pass a specific test to pass the subject in this call.

ASSESSMENT CRITERIA

1. Attendance and participation in seminars.
2. Assimilation and understanding of course's content.
3. Ability to apply acquired knowledge.
4. Integration and communication of knowledge.
5. Interpretation of results and resolution of questions and problems.
6. Time management for planning activities and laboratory experiments.

RATING CRITERIA

Numerical ratings (%) of activities

To pass the course will require:

Continuous assessment. Normal and extraordinary calls: theoretical content (75%) and practical work (25%). Furthermore, the percentage of the theoretical content includes, 70% of theoretical questions and 30% of resolution of practical cases (numerical exercises).

Final assessment. Normal and extraordinary calls: theoretical content (75%) and practical work (25%). Due to the different competences pursued with theoretical content and practical work and lacking information from continuous assessment, students will have to demonstrate sufficient knowledge and abilities within each part, separately.

6. BIBLIOGRAPHY

[1] Quantitative Chemical Analysis, Daniel C. Harris. Eighth edition, 2010. W.H. Freeman & Company. New York. BAF543.062HAR

[2] Analytical Chemistry 2.0 (eText), David Harvey.

[3] Fundamentals of Analytical Chemistry. Douglas A. Skoog, Donald M. West, F. James Holler, Stanley R. Crouch. Eighth edition, 2004. Thomson – Brooks/Cole. BAF543SKO