VNIVERSITAT DE VALÈNCIA

Course Guide
34064 Chemical Analysis

COURSE DATA

Data Subject	
Code	34064
Name	Chemical Analysis
Cycle	Grade
ECTS Credits	9.0
Academic year	2019 - 2020

Study (s)

Degree	Center	Acad. year	Period
1201 - Degree in Pharmacy	Faculty of Pharmacy and Food Sciences	2	Annual
1211 - D.D. in Pharmacy-Human Nutrition and Dietetics	Faculty of Pharmacy and Food Sciences	2	Annual

Subject-matter

Degree	Subject-matter	Character
1201 - Degree in Pharmacy	4 - Chemical analysis	Obligatory
1211 - D.D. in Pharmacy-Human Nutrition and Dietetics	1 - Asignaturas obligatorias del PDG Farmacia-Nutrición Humana y Dietética	Obligatory

Coordination

Name	Department
MOLINER MARTINEZ, YOLANDA	310 - Analytical Chemistry

SUMMARY

Chemical Analysis is a compulsory core subject of 9 ECTS credits to be taught in the second course of the degree in Pharmacy. Attending to the proficiencies that a pharmacist has to develop, chemical analysis can be considered as a needed discipline, essential for the proper development in their professional practice. This subject introduces and develops the essential knowledge needed for the identification and determination of chemical compounds in matrices of pharmaceutical interest. The program consists of 12 thematic units divided into three blocks and a thematic unit that includes a series of laboratory practices involving the application of some of the analytical methods included in the program.

In the first block general objectives and work in chemical analysis are discussed. Steps of the analytical process are described and finally referring to statistical treatment of analytical results.
Then, work methods and applications of volumetric and gravimetric methods commonly known methods of analysis are studied.
The program ends with 6 lessons that are dedicated to the description of different instrumental methods of analysis: optical methods, electroanalytical methods and chromatographic methods.
For each basis and necessary instrumentation, way of working and usefulness for analysis of substances of interest in the pharmaceutical field it indicated.

PREVIOUS KNOWLEDGE

Relationship to other subjects of the same degree

There are no specified enrollment restrictions with other subjects of the curriculum.

Other requirements

In order to successfully address the subject, is essential that the student gathers a number of previous knowledge and skills:

- Nomenclature and chemical formulation.
- Adjusting of chemical reactions.
- Chemical equilibrium in solution.
- Stoichiometric calculations.
- Basic mathematical calculations (solving equations, operations with logarithms, systems of equations ...)
- Use of the scientific calculator for performing mathematical operations and least squares regression.

OUTCOMES

1201 - Degree in Pharmacy

- To possess and to understand the knowledge in the different areas of study included in the formation of the pharmacist.
- To apply this knowledge to the professional world, contributing to the development of Human Rights, democratic principles, principles of equality between women and men, solidarity, protection of the environment and promotion of a culture of peace with Gender perspective.
- To know how interpret, value and communicate relevant data in the different aspects of pharmaceutical activity, making use of information and communication technologies.
- Skill to communicate ideas, analyze problems and solve them with a critical mind, achieving team-working abilities and assuming leadership whenever required.

- Development of skills to update their knowledge and undertake further studies, including pharmaceutical specialization, scientific research and technological development, and teaching.
- Ability to collect and transmit information in English with a level of competence similar to the B1 of the Council of Europe.
- Identify, design, obtain, analyze, control and produce drugs and medicines and other products and raw materials of health interest for human or veterinary use.
- Module: Chemistry - Ability to select appropriate techniques and procedures in the design, application and evaluation of reagents, methods and analytical techniques.
- Develop hygienic-sanitary analyzes, especially those related to food and the environment.
- Identify and understand the importance of each stage of the analytical process.
- Understand the importance of quality control in the analytical laboratory, as well as the statistical procedures and tools necessary to carry out this control.
- Establish the classification of the main analytical methods, understand their fundamentals and know how to select their use according to the purpose of the analysis.
- Properly employ the working methodologies of the techniques used in practical laboratory sessions and know how to prepare and submit an analytical report.

LEARNING OUTCOMES

After completing this subject, students should be able to:

- Identify and describe each stage in the analytical process.
- Apply basic statistic basis for evaluating the quality of analytical results.
- Apply the procedures and statistical tools needed to carry out quality control in the analytical laboratory.
- Establish the classification of the main instrumental techniques for analysis.
- Apply properly different calibration methods for quantification in instrumental analysis.
- Define and calculate the most important figures of merit in the instrumental methods.
- Enumerate the main separation techniques and understand their basis and objectives.
- Define and explain concepts related to the volumetric and gravimetric methods and expose their main applications of health interest.
- Apply an adequate methodology for performing calculations in volumetric and gravimetric applications.

- Enumerate and explain the main electrochemical techniques and their applications.

- Describe the basis, experimental methodology and the main applications of the various techniques of molecular spectrometry and analytical characteristics of them.

- Describe the basis, experimental methodology and the main applications of the different techniques of atomic spectrometry and analytical characteristics of them.

- Enumerate and expose the basis of the different chromatographic methods and how to interpret adequately the information provided by chromatograms.

- Define the concept of hybrid (coupled) instruments and their importance for the elucidation and analysis of samples.

- Describe the most important coupled systems based on gas chromatography and liquid chromatography.

- Detail the highlights of automation in the analytical laboratory.

- Explain the basis and the main applications of chemical sensors.

- Work properly in an analytical laboratory.

- Use adequate working methods of the techniques used in practice laboratory sessions.

Develop and submit an analytical report based on data obtained in the laboratory, after performing the calculations and the appropriate statistical treatment.

DESCRIPTION OF CONTENTS

1. INTRODUCTION TO ANALYTICAL CHEMISTRY

Concept and structure. Types and levels of information. Stages of the analytical process. Classification of analytical techniques. Importance of qualitative and quantitative analysis in the pharmaceutical field.

2. SAMPLING, STORAGE, TRANSPORT AND PREPARATION OF MATERIALS

Importance of the processes of sampling and treatment of materials. Sampling. Sampling plan. Implementation of the sampling plan. Previous treatments of the sample. Filtration and centrifugation. Initial dissolution. Deproteinization. Extractive separation techniques. Other isolation and preconcentration techniques.

3. EVALUATION OF DATA, CALIBRATION AND VALIDATION OF ANALYTICAL METHODS

Errors in chemical analysis. Precision and accuracy. Rejection of discordant results. Presentation of analytical results. Concept of calibration. Linear calibration. Analytical figures of merit: sensitivity, detection and quantification limits, dynamic range. Standard addition method. Internal standard method. Concept validation. Hypothesis testing. Validation of accuracy. Validation of accuracy.

4. VOLUMETRIC AND GRAVIMETRIC ANALYSIS

Introduction to volumetric methods. Fundamentals of gravimetric methods. Precipitation mechanisms. Basic operations of gravimetric analysis. Calculations. Combustion analysis. Some applications of pharmaceutical interest.

5. ACID-BASE TITRATIONS AND BUFFER SOLUTIONS

Acid-base equilibrium. Titrating strong acids and strong bases. Titrating weak acid, weak bases and polyprotic systems. Buffer solutions: concept, limitations and utilities.

6. OTHER TITRATIONS. CONCEPT OF SIDE REACTION

Equilibrium of complex formation and precipitation: concepts of side reaction and conditional constant. Complexation and precipitation titrations. Equilibrium and redox titration.

7. ELECTROCHEMICAL ANALYSIS

Electrochemical cells. Electrode potentials. Potentiometry. Voltammetry. Instrumentation. Analytical methodology. Analytical features and performance. Some applications of pharmaceutical interest in qualitative and quantitative analysis. Electrochemical sensors.

8. ANALYTICAL SPECTROMETRY

Fundamentals. Instrumentation. Analytical methodology. Analytical features and performance. Some applications of pharmaceutical interest in qualitative and quantitative analysis in molecular and atomic spectrometry. Optical sensors.

9. INTRODUCTION TO CHROMATOGRAPHIC ANALYSIS AND COUPLED METHODS

Concept and classification of chromatographic techniques. Chromatographic modes. Main parameters in chromatography. Theory of the chromatographic separations. Coupled methods.

10. GAS CHROMATOGRAPHY

Fundamentals. Components in a gas chromatograph. Columns and stationary phases. Detectors. Effect of temperature. Analytical methodology. Some applications of pharmaceutical interest in qualitative and quantitative analysis. GC-MS analysis.

11. LIQUID CHROMATOGHRAPHY

Fundamentals. Classification. Thin Layer Chromatography. Column chromatography. Components in a liquid chromatograph. Analytical methodology. Some applications of pharmaceutical interest in qualitative and quantitative analysis. LC-MS analysis.

12. ELECTROPHORESIS

Fundamentals. Classification. Basic parameters. Capillary and gel electrophoresis. Methodology. Applications of pharmaceutical interest.

13. LABORATORY SESSIONS

SESSION 1.- Determination of total hardness of water by complexometric titration
SESSION 2.- potentiometric determination of fluoride in toothpaste
SESSION 3.- Colorimetric determination of N-acetyl-L-cysteine with Fe (III) and 1,10-phenanthroline in pharmaceutical preparations
SESSION 4.- Determination of calcium content in tablets by atomic absorption spectrometry
SESSION 5.- Quality control of pharmaceutical preparations: determination of paracetamol, aspirin and caffeine by HPLC

WORKLOAD

ACTIVITY	Hours	% To be attended
Theory classes	40,00	100
Laboratory practices	25,00	100
Seminars	15,00	100
Tutorials	4,00	100
Preparing lectures	62,50	0
Preparation of practical classes and problem	70,00	0
TOTAL	**216,50**	

TEACHING METHODOLOGY

The subject is structured considering five types of activities for its development: lectures, problems seminars, laboratory sessions, tutorial sessions and workshops.

Lectures and problems seminars. Since the subject has a very practical nature, lectures and problem classes are alternated throughout the course, up to a total of forty hours of lectures (20 hours / semester). The time spent on theory and problems will vary depending on the needs.

Lectures. In the lectures, the instructor will offer an overview of the topic, emphasizing in the key concepts for understanding, and he/she will answer incidental doubts or questions.

Problems seminars. They have the aim of applying the knowledge acquired the lectures by solving questions and problems. The instructor will resolve for the whole class some selected problems; the students will work other new examples in small groups. Additional problems will be proposed to the students, to be resolved individually and they will be discussed with the instructor along individual evaluation meetings.

Tutorial sessions. Students will assist in small groups, participating in 4 sessions along the course. In them, the teacher will give to the student advices on all the elements of the learning process, in terms of global strategies and specific issues. Also, students will present the results they have obtained on the additional problems and the questions set by the teacher, and will discuss them on the blackboard.

Laboratory sessions. Prior to attending the lab, the student must have studied the script of each practice, review all the theoretical concepts involved, answer a series of previous questions, and prepare a flow diagram of the work to do. In the lab, the teacher will emphasize the most important points on the current session and will supervise the experiments. Once completed the experimental work, the student will perform the relevant calculations and will process statistically the gathered data, using spreadsheets and software available in the lab PCs. During the last laboratory session, the students will be evaluated by an oral exam on some issues discussed during the practical sessions. Finally, the student will prepare a detailed report showing the analytical results in all experiments performed.

Workshops. Throughout the course there will be workshops on various aspects of depth on the subject. The teacher will provide the necessary materials and propose a series of activities to promote learning.

EVALUATION

The evaluation of students learning will take into account the different aspects outlined in the methodology section of this guide and will be performed by the teacher in a continuous way. For this purpose, the subject is structured in three parts: theoretical sessions, laboratory sessions and other activities.

The score or the theoretical part weighs 65% in the final mark. In this section the acquired knowledge will be evaluated through two written exams along the academic year, the first one at the end of the first semester and the second one coinciding with the June general exams. These written exams will consist of conceptual questions and problems that allow the student to demonstrate his/her degree of assimilation of fundamental concepts. They may also include more general topics where the student will have to show his/her ability to synthesize and expose. The minimum qualification of each exam to proceed to a mean score will be 4. In the case of obtaining a score below 4 on the first exam, the June session will consist of a final test of all the program. The minimum score that may be achieved in the theoretical part to proceed to an average with the other activities of the subject is 4.5. Students that do not achieve the minimum score in the theoretical part but have a score above 5 in one of both parts (first or second semester) could have their mark preserved for the second opportunity of that academic year, but NOT for following courses.

To evaluate laboratory sessions, where attendance is compulsory, the student will have to present a report gathering the analytical results of all practices. In addition, during the last laboratory session, there will be an oral exam on issues discussed during their development. 20% of the total score will correspond to the laboratory report, 30% to the exam and 50% to the quality of the results (in terms of precision and accuracy). The overall qualification will rate 20% of the final score of the subject. In case of not passing the subject, the mark obtained in the laboratory part could be preserved during the two following academic years.

15% of the global score will come from activities developed in any of the sections of the learning process. It will consider issues such as: active participation in the tutorial sessions, preparation and presentation of the proposed activities, class attendance, reasoned and clear participation in the discussions, progresses in the adequate use of the chemical terminology, critical spirit and capability to collaborate with the rest of the group. In case of not passing the subject, the mark obtained in activities will NOT be preserved for following years.

FIRST GENERAL EXAM OPPORTUNITY

The final course score is calculated averaging the specific scores in theory, laboratory sessions and other activities by the following expression:

Final Rating = THEORY x 0.65 + PRACTICAL x 0.20 + ACTIVITIES x 0.15

This expression only applies in case of obtaining a minimum score of 4.5 out of 10 in each of the parts. To pass the subject, the student must obtain a qualification of 5 out of 10. In the case of the final rating being smaller than 5.0 points, or not having obtained a minimum score of 4.5 to balance underscored parts, the subject will be considered failed.

Note:

The student may request signing a document to be evaluated only by a written exam. In this case, the exam will consist of three sections. One of them will be identical to the exam resolved by the rest of the students; it will take place simultaneously to their exam, and will contribute in 65% to the overall qualification. The second part will consist of a series of questions assessing the skills that the other students have demonstrated to have through the implementation of activities in seminars and tutorials (15%). Finally, the third part will consist of a practical examination in the laboratory (20%).

SECOND GENERAL EXAM OPPORTUNITY

In the second round of qualification, the same criteria used in the first round are applied. Students who did not pass in the first opportunity one or more of the three parts, should have to be examined of the pendant part(s).

Those students who do not show the theory exam (June and July) but who have participated and have noted in some / s of educational activities (partial review, seminars practices, tutorials) were qualified as not presented in the first call Thriller course as in the second.

REFERENCES

Basic

- QUÍMICA ANALÍTICA. D.A. Skoog, D.M. West , F.J. Holler y S.R. Crouch, 8ª edición, Thomson, 2005.
- ANÁLISIS QUÍMICO CUANTITATIVO. D.C. Harris, 3ª edición, Reverté, 2007.
- QUÍMICA ANALÍTICA MODERNA. D. Harvey, McGraw-Hill Interamericana, 2002.
- PRINCIPIOS DE QUÍMICA ANALÍTICA. M. Valcárcel, Springer, 1999.
- Analytical Chemistry 2.0: http://acad.depauw.edu/harvey_web/eText%20Project/AnalyticalChemistry2.0.html

Additional

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- APROXIMACIÓ A LANÀLISIS QUANTITATIVA MITJANÇANT LA RESOLUCIÓ DE PROBLEMES. C. Gómez Benito, S. Torres Cartas, S. Meseguer Lloret, C. Cháfer Pericás, Y. Martín Biosca, editorial UPV, 2009.
- QUÍMICA ANALÍTICA CONTEMPORÁNEA. J.F. Rubinson y K.A. Rubinson, Prentice Hall, 1999.
- TOMA Y TRATAMIENTO DE MUESTRAS. C. Cámara (ed.), P. Fernández, A. Martín Esteban, C. Pérez Conde y M. Vidal., Síntesis, 2002.
- PROBLEMAS RESUELTOS DE QUÍMICA ANALÍTICA. P. Yánez-Sedeño, J.M. Pingarrón y F.J.M de Villena, Síntesis, 2003.

ADDENDUM COVID-19

This addendum will only be activated if the health situation requires so and with the prior agreement of the Governing Council

1. Contenidos

La asignatura Análisis Químico pretende que el estudiante adquiera los conocimientos básicos necesarios para la identificación y análisis de compuestos químicos en todo tipo de muestras de interés farmacéutico. Se ofrece en el segundo curso del Grado en Farmacia a lo largo de los dos cuatrimestres del año académico. Globalmente, la asignatura consta de 40 horas de clases de teoría (20 horas por cuatrimestre), 25 horas de clases prácticas de laboratorio, 15 horas de sesiones de seminarios (10 horas en el primer cuatrimestre y 5 horas en el segundo) y 4 horas de sesiones de tutorías (2 horas en cada cuatrimestre), todas ellas presenciales, que se complementan con el trabajo no presencial de los estudiantes relativo a la preparación de las clases de teoría, de problemas y clases prácticas. Esta docencia se imparte a cinco grupos de estudiantes y diecisiete subgrupos de prácticas.

En la fecha de interrupción de la docencia presencial, todos los grupos de teoría habían completado un 75% de las clases, habiendo impartido aproximadamente un tercio de la materia asignada al segundo cuatrimestre, así como dos seminarios. Por lo tanto, al interrumpirse las actividades en las instalaciones de la Universidad no se han podido impartir

10 clases de teoría y tres seminarios. Respecto a los subgrupos de prácticas, únicamente dos de los diecisiete no habían comenzado las sesiones de laboratorio.

La importancia que tienen los contenidos impartidos en el segundo cuatrimestre en los laboratorios farmacéuticos, junto con el hecho de que no hay ninguna otra asignatura en el Grado en Farmacia que los recoja, ha determinado que se continúe con el temario inicialmente previsto en la guía docente hasta completarlo en su totalidad, sin dejar fuera ningún tema. Los contenidos de los temas que quedaban por impartir son adaptables a la docencia no presencial. Respecto a las prácticas de laboratorio, la voluntad de los profesores ha sido proporcionar la máxima formación posible relacionada con el contenido de las mismas

2. Volumen de trabajo y planificación temporal de la docencia

La docencia presencial en la asignatura de Análisis Químico constaba de los siguientes apartados:

1. Clases de teoría

2. Seminarios

3. Tutorías

4. Clases de laboratorio

Los tres primeros apartados se han mantenido en el paso a la docencia no presencial. A cada semana se la ha asignado un tema. En el aula virtual se han cargado las cuestiones básicas que debían aprender los estudiantes y materiales docentes. La idea es poder repasar la materia antes del fin de curso, siendo conscientes de las dificultades que plantea la ausencia de presencialidad.

3. Metodología docente

Se ha recomendado a los profesores que realicen las actividades programadas de la misma forma que cuando las realizaban de forma presencial. Los apuntes de los temas de teoría en Power Point u otros formatos se han seguido dejando en aula virtual, apoyados con documentos de Power Point locutados. Este material ofrece una visión global del tema e incide en aquellos conceptos clave para la comprensión del mismo. Asimismo, se han proporcionado guías de apoyo para resolución de problemas, y cuando ha sido posible, se han habilitado sesiones de chat o videoconferencia para aclarar las dudas que hayan podido surgir. En otros casos, se ha planteado a través de aula virtual una serie de temas cada semana para que los estudiantes enviaran su respuesta, abriendo la posibilidad a la consulta de otras fuentes que deben ser citadas. Con esta metodología se pretende que los estudiantes adquieran los conceptos básicos, que localicen fuentes fiables en la red y contrasten las opiniones.

Estas herramientas también se utilizarán para las sesiones de tutorías a realizar en el mes de mayo. En las clases de prácticas, los estudiantes disponen de guiones, cuestiones y datos primarios de laboratorio para elaborar los informes de las prácticas que estaban previstas, siguiendo la programación correspondiente.

Durante el tiempo de docencia no presencial, los profesores siguen corrigiendo las actividades de tutorías y seminarios entregadas por los estudiantes e informes de prácticas.

4. Evaluación

En la asignatura de Análisis Químico tiene amplio peso en la nota el bloque de teoría. La nota final se calcula teniendo en cuenta el peso de cada bloque en la calificación final:

1. Teoría: 65 %. Se realizan dos pruebas escritas a lo largo del curso, una primera prueba parcial al finalizar el primer cuatrimestre y una segunda que coincide con la primera convocatoria de la asignatura. La nota mínima para compensar entre ambos exámenes es de 4. En el caso de obtener una calificación inferior a 4 en la primera prueba, la convocatoria de junio consiste en una prueba final de toda la asignatura. La nota mínima que se ha de obtener en el bloque de teoría para poder promediar con las otras

actividades de la asignatura es de 4,5.

2. Clases de laboratorio: 20 %. Los informes de prácticas constituyen un 20% de la nota, un 30% el examen y un 50% los resultados obtenidos.

3. Actividades: 15 %. Realizadas en clase, seminarios o tutorías.

El peso de estos porcentajes para calcular la nota final se aplica en el caso de haber obtenido una nota mínima de 4,5 puntos sobre 10 en cada una de las partes. Para aprobar la asignatura es necesario obtener una calificación final de 5 puntos sobre 10. En caso de obtener una calificación final inferior a 5 puntos, o de no haber obtenido la nota mínima de 4,5 para compensar alguna de las partes, no se supera la asignatura.

Con el paso a la docencia no presencial, se han mantenido estos porcentajes. El examen de primera convocatoria se realizará en la fecha prevista, pero de forma online a través de aula virtual. La principal diferencia radica en que las preguntas conceptuales y de desarrollo se sustituyen por un cuestionario tipo test. Los problemas también se preguntarán con cuestiones que seguirán este formato. Previo a la fecha del examen se informará a los estudiantes del tipo de examen a fin de que se puedan detectar posibles problemas de accesibilidad y poder proponer una solución adecuada. En caso de que se produzca un fallo en la conexión durante la realización del examen, el estudiante reportará dicha incidencia enviando una foto de la pantalla donde quede reflejada la hora y la fecha de la misma. En tal caso, se arbitrará una solución alternativa para que dichos estudiantes realicen el examen.

Por otra parte, las cuestiones propuestas en aula virtual contestadas por los estudiantes se evaluarán de 0 a 10 con el fin de tener una nota complementaria a la nota del examen, en caso de que proceda. En el caso de las clases de laboratorio, el profesor calificará las contestaciones a las cuestiones planteadas a través de aula virtual y los informes de prácticas entregados.

5. Bibliografía

La bibliografía empleada se halla reseñada en la guía docente, por lo que el paso de clases presenciales a no presenciales no ha supuesto ningún cambio. Debido al cierre de la Biblioteca del Campus y la imposibilidad de los desplazamientos, los alumnos que no disponen de los libros, pueden acceder a ellos online a través del Servicio de Bibliotecas.