COURSE GUIDE FOR PROCESOS DE SEPARACIÓN

Academic year 20-21 (Date last update: 30/06/2020) (Date approved in Department Council: 30/06/2020)

MODULE	SUBJECT MATTER	YEAR	SEMESTER	CREDITS	ТҮРЕ
Bridging courses	Separation processes	3st	1st	6	Optional subject
TEACHING STAFF ⁽¹⁾			ADDRESS, TELEPHONE NUMBER, EMAIL, ETC. DIRECCIÓN COMPLETA DE CONTACTO PARA TUTORÍAS (Dirección postal, teléfono, correo electrónico, etc.) Departamento de Fisicoquímica. Facultad de Farmacia. Campus Universitario de Cartuja. 18071-Granada.		
- Mª Eugenia García Rubiño - Javier Valverde Pozo			DEPT Fisicoquímica Facult y of Pharmacy. Office number: 193 and lab 213. Email: <u>rubino@ugr.es</u> ; <u>javalverde@ugr.es</u>		
			TIMETABLE FOR TUTORIALS OR LINK TO WEBSITE		
			http://fisicoquimica.ugr.es/pages/docencia/cur so 2021/ doc/horariotutorias2021		
BELONGS TO UNDERGRADUATE DEGREE PROGRAMME			AND ALSO TO OTHER UNDERGRADUATE DEGREE PROGRAMMES		
Degree in Pharmacy					
PREREQUISITES O	R RECOMMENDATIONS (where app	licable)	1		

Basic knowledges Biochemistry and Inorganic Chemistry are strongly recommended. •

BRIEF DESCRIPTION OF CONTENT (ACCORDING TO OFFICIAL VALIDATION REPORT)



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¹ Consult any updates in Acceso Identificado > Aplicaciones > Ordenación Docente (∞) This course guide should be filled in according to UGR regulations on assessment of student learning: (http://secretariageneral.ugr.es/pages/normativa/fichasugr/ncg7121/!)

The subject lays foundations in the key areas of: gas chromatography, liquid chromatography, molecular exclusion chromatographies, affinity and supercritical fluids, electrophoresis, centrifugation, sedimentation and thin layer chromatographic separation, with application in Pharmacy courses.

GENERAL AND SPECIFIC COMPETENCES

• CG1 - Identify, design, obtain, analyze, control and produce drugs and medicines, as well as other products and raw materials of health interest for human or veterinary use.

• CG10 - Design, apply and evaluate clinical analytical reagents, methods and techniques, knowing the basic fundamentals of clinical analysis and the characteristics and contents of laboratory diagnostic findings.

• CG15 - Recognize one's own limitations and the need to maintain and update professional competence, with particular emphasis on self-learning new knowledge based on available scientific evidence.

• CT2 - Ability to use ICTs with ease.

• CE49 - Know the analytical techniques related to laboratory diagnosis, toxic, food and environment.

OBJECTIVES (EXPRESSED AS EXPECTED LEARNING OUTCOMES)

- Knowledge of the different Separation Methods of interest in Pharmacy.
- Study of the different physico-chemical processes that govern these processes.
- Analysis and determination of the parameters involved.
- Knowledge of the necessary instrumentation.
- Interpretation of results in each methodology.
- Select the most suitable technique for the analysis and control of medicines, sanitary products, water, food and environmental analysis.

DETAILED SYLLABUS

THEORY:

Theoretical syllabus:

- TOPIC 1.- Introduction to chromatography. (4 hours)

History. Concept of chromatography. Classification. Distribution equilibria. Linear isotherms. Distribution parameters. Linear elution chromatography. Retention parameters. Migration.

- TOPIC 2.- Chromatography theories. (5 hours)

Plate theory. Column efficiency. Kinetic theory. General equation. Differences between C.G. and C.L. Resolution. Retention time. Optimal column efficiency conditions. Gradient elution and temperature programming. Applications. Calibration method with standards. Normalization of areas. Internal pattern.

- TOPIC 3.- Flat Chromatography. (1hour)

CP and CCF. How separation is performed. Efficacy characteristics. Variables that affect Rf. Qualitative and quantitative determinations.

- TOPIC 4.- Instrumentation of gas chromatography. (3 hours)

Carrier gas. Sample injection. Columns. Stationary phases. Thermal conductivity detectors, flame ionization detectors, electron capture detectors, atomic emission detectors. Couplings with mass spectrometry.

- TOPIC 5.- Gas chromatography. (3 hours)

CG retention volumes, specific volume. Applications to pharmaceuticals. Qualitative interpretation of a



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chromatogram. Relative retention. Oster ratio. Kovats retention rate.

- TOPIC 6.- HPLC instrumentation. (2 hours)

Mobile phase. Pressure system. Columns. Column filling. Detectors: UV-V absorbance, fluorescence, electrochemical, refractive index, dispersion.

- TOPIC 7.- Liquid chromatography. (2 hours)

Classification. Adsorption chromatography. Stationary phases. Separation mechanism. Mobile phase, eluent force ($\epsilon 0$).

- TEMA 8. - Liquid-liquid or delivery chromatography. (1 hour)

Normal phase chromatography. Reverse phase chromatography. Mobile phase polarity index. Mechanism. Applications. Chromatography of ion pairs.

- TEMA 9. - Ion exchange chromatography. (2 hours)

Types of exchange resins. Ion exchange mechanism. Suppressor column chromatography.

- TOPIC 10. - Exclusion chromatography. (1 hour)

Mechanism. Parameters. Applications.

- TOPIC 11. - Other chromatographies. (3 hours)

Affinity chromatography. Matrices. Ligand coupling. Biospecific and non-specific elution. Applications. Supercritical fluid chromatography. Properties of supercritical fluids. Instrumentation. Stationary and mobile phases. Pressure effect. Detectors. Comparison with other methods. Couplings with mass spectrometry.

- TOPIC 12. - Electrophoresis. (2 hours)

Electrokinetic phenomena. Zone electrophoresis. Factors that affect electrophoresis. Immunoelectrophoresis.

- TOPIC 13. - Electrophoresis in polyacrylamide gels. (3 hours)

Application to protein separation. Non-denaturant conditions. Ferguson representation. Denaturant conditions. P.A.G.E.-SDS. Molecular mass estimation. Membrane transfer. Electrofocus. Two-dimensional electrophoresis.

- TOPIC 14. - Electrophoresis in agarose gels. (1 hour)

Application to nucleic acid separation. Pulsating field electrophoresis.

- TOPIC 15. - Capillary electrophoresis. (2 hours)

Instrumentation. Migration and plate height in capillary electrophoresis. Electroosmotic flow characteristics. Detection in zone capillary electrophoresis. Capillary isoelectrofocus. Capillary electrochromatography. Capillary chromatography. Micellar electrokinetic.

- TOPIC 16. - Sedimentation. (3 hours)

Ultracentrifugation and Sedimentation. Transport under centrifugal forces. Lamm equation. Lamm equation solutions. Svedberg equations. Determination of molecular parameters. Multicomponent systems. Sedimentation equilibrium. Density gradient equilibria.

PRACTICE: Seminars/Workshops



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At the end of each lesson, there will be a seminar with practical problems.

Laboratory work

- Session 1. - Separation of DNA fragments by electrophoresis.

DNA fragments are separated and their molecular weight is calculated by calibration to known molecular weight standards.

- Session 2. - Separation of analgesics by HPLC.

A reverse phase HPLC separation of a mixture of four analgesics is performed, with the main chromatographic parameters calculated.

- Session 3. - Potentiometric determination of phosphate in a powdered yeast with ion exchange chromatography extraction.

The percentage of phosphate in a commercial yeast is quantified by potentiometric titration of the purified phosphate using ion exchange chromatography.

- Session 4. - Gel exclusion liquid chromatography.

Two proteins and a polysaccharide are separated by gel exclusion chromatography, each of which is identified by spectrophotometry.

BASIC BIBLIOGRAPHY

- "Principios de Análisis Instrumental". (6ª Edición) Skoog-Holler. S.A. Ediciones Paraninfo, 2009.

-"Fundamentos de Química Analítica". Douglas A. Skoog, Donald M. West y F. James Holler. Editorial Reverté. 1997 (Cuarta Edición).

-"Técnicas de separación en Química Analítica". R. Cela, R.A. Lorenzo y M.C. Casais. Editorial Síntesis. 2002. (Primera Edición).

-"Tecnicas Analiticas de Separacion". M. Valcarcel, Ed. Reverte.

-"Técnicas de separación en química analítica." Rafael Cela, Rosa Antonia Lorenzo, Mª del Carmen Casais-Editorial Síntesis, 2003.

-"Análisis instrumental". Kenneth A. Rubinson, Judith F. Rubinson - 2001 Pearson Educación.

ADDITIONAL BIBLIOGRAPHY

-"Fundamentos de Análisis Instrumental". D.A. Skoog y J. Levy, Editorial Interamericana McGraw Hill, (Cuarta Edición).

-"Métodos y técnicas instrumentales modernas". Francis Rouessac y Annick Rouessac, Editorial McGraw Hill.

-"Curso de análisis farmacéutico". Connor. Editorial Reverte.

-"Fisicoquímica para farmacia y biología". P. Sanz Pedrero. Ediciones Científicas y Técnicas S.A. (Masson y Salvat Medicina).

RECOMMENDED LINKS

- https://www.youtube.com/watch?v=CKu-zpK9NJM
- https://www.youtube.com/watch?v=oIFoRQG_cis
- https://www.youtube.com/watch?v=6vKLT5mQoBM



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- https://www.youtube.com/watch?v=TD0-2lkvfgU
- https://www.youtube.com/watch?v=Xt-8mEfsksA
- https://www.youtube.com/watch?v=mCFFxiDuiDA
- https://www.youtube.com/watch?v=GjpSvKHMPB0&feature=youtu.be

• https://www.mediatheque.lindau-nobel.org/videos/33807/1958-die-nobelstiftung-einige-gedankenueber-ihre-tradition-und-ihr-wirken/meeting-1958

TEACHING METHODOLOGY

- **Theoretical sessions**. These sessions are held in the form of a master lesson through face-to-face exhibitions where the theoretical contents of the subject will be taught and discussed. Use will be made of the audiovisual media available in the classrooms of the Faculty of Pharmacy. The materials of the subjects, such as figures, diagrams and summaries, will be made available to students through the PRADO platform, as well as web links to pages of interest to deepen the study of the subject.
- **Practical sessions of laboratory**. In these sessions the student must carry out an experimental work as an application of the theoretical classes and has for this purpose a notebook of practices where the realization of the experimental work is described and the obtained results must be collected. At all times the teacher will be supervising the performance of the work.
- **Problem sessions**. Lists of problems will be published corresponding to the topics explained, which the student has to carry out in advance and which will be resolved in the corresponding seminars.
- **Performance of work**. Students must perform the work recommended by the teacher and they will be supervised by the teacher. The exposition of these will be done according to the criterion of the teacher.
- **Conduct of other ongoing evaluation activities**. There will be continuous evaluation activities such as solving numerical problems, self-evaluation tests through the platforms PRADO, Kahoot, active participation in forums of the platform PRADO, etc, as well as guided tours to the Scientific Instrumentation Center (headquarters in the Faculty of Pharmacy and headquarters in Fuentenueva).

ASSESSMENT (ASSESSMENT INSTRUMENTS, CRITERIA AND PERCENTAGE VALUEOF FINAL OVERALL MARK, ETC.)

All the evaluation processes will be carried out according to the normative of the University of Granada.

1. Ordinary call

- <u>Written exams about theoretical concepts</u>. Percentage of the final mark: 55%.
 - There will be a mid-course exam and a final exam.

The qualification considered in this section will be the mark obtained in the final exam. If the subject has been passed by overcoming both mid-course and final exam, the qualification will be the average between them.

• <u>Written exams about mathematic resolution of application problems</u>. Percentage of the final mark: 15%. There will be a mid-course exam and a final exam.

The qualification considered in this section will be the mark obtained in the final exam. If the subject has been passed by overcoming both mid-course and final exam, the qualification will be the average between them.

- <u>Practical lessons.</u> Percentage of the final mark: 10%.
 - It is compulsory to attend to all practical lessons. The global mark is divided in two tasks:
 - Laboratory book 30%
 - Exam about practical contents : 70%



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• Continuous evaluation and attending to theoretical classes. 20%.

2. Extraordinary call and single final evaluation

Written exams about theoretical concepts. Percentage of the final mark: 60%.

There will be a mid-course exam and a final exam.

The qualification considered in this section will be the mark obtained in the final exam. If the subject has been passed by overcoming both mid-course and final exam, the qualification will be the average between them.

• <u>Written exams about mathematic resolution of application problems.</u> Percentage of the final mark: 30%. There will be a mid-course exam and a final exam.

The qualification considered in this section will be the mark obtained in the final exam. If the subject has been passed by overcoming both mid-course and final exam, the qualification will be the average between them.

• <u>Practical lessons.</u> Percentage of the final mark: 10%.

DESCRIPTION OF THE EXERCISES WHICH WILL CONSTITUTE SINGLE FINAL ASSESSMENT AS ESTABLISHED IN UGR REGULATIONS

• This description is reported in the assessment section

SCENARIO A (ON-CAMPUS AND REMOTE TEACHING AND LEARNING COMBINED)

TUTORIALS

TIMETABLE	TOOLS FOR TUTORIALS
(According to Official Academic Organization Plan)	(Indicate which digital tools will be used for tutorials)
https://fisicoquimica.ugr.es/pages/docencia/curso_202 1/_doc/horariotutorias2021	There will be both on-site and virtual tutorials. Proposed telematic media are: - Forums in virtual platform (PRADO) - e-mail

MEASURES TAKEN TO ADAPT TEACHING METHODOLOGY

- **Theory lessons**: Teaching methodology will be the same proposed in the previous section about "teaching methodology". In this case, the master classes will be broadcasted in real time to those students that can not attend if the number of people in the classes is limited by the health situation.
- **Practical lessons**: Due to the limited capacity of the facilities of the faculty there will be two days of practical lessons in the lab and two days of virtual lessons, concerning to the theoretical aspects of the two on-site tasks.
- **Continuous evaluation** .In these cases methodology will be the same proposed in the previous section about "teaching methodology" and there will be preferentially developed on-site. In case there will be capacity limitation virtual media will be used (Google Meet, PRADO Kahoot, etc).

EVALUATION ADAPTATION MEASURES (Instruments, criteria and percentages on final rating)

Ordinary assessment session



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• Adaptative measures have only been proposed for the practical lessons. The written examns of this section will be in virtual through the PRADO platform. The percentages are those described in the assessment section

Extraordinary assessment session

• Adaptative measures have only been proposed for the practical lessons. The written examns of this section will be in virtual through the PRADO platform. The percentages are those described in the assessment section.

For those students that have attended to the practical lessons and have not passed the exam there will be a virutal written exam in the PRADO platform.

Those students that have not atended to the practical lessons will have to pass a practical exam in the laboratory. The evaluation will be carried out by the teachers in charge of the practical lessons.

Single final assessment

• There is no adaptative measures in this type of assessment

SCENARIO B (ONCAMPUS ACTIVITY SUSPENDED)

TUTORIALS

TIMETABLE	TOOLS FOR TUTORIALS		
(According to Official Academic Organization Plan)	(Indicate which digital tools will be used for tutorials)		
https://fisicoquimica.ugr.es/pages/docencia/curso_202 1/_doc/horariotutorias2021	There will exclusively virtual tutorials. Proposed telematic media are: - Forums in virtual platform (PRADO) - Google meet - email communication		

MEASURES TAKEN TO ADAPT TEACHING METHODOLOGY

- Theory:
 - Synchrony classes through Google-Meet.
 - Recorded videos of the theoretical lessons.
- Practial lessons:

• All the practical lessons will be virtual and attending is compulsory by Google-Meet videoconference.

• Seminars:

• The seminars related with the application problems will be taught by Google Meet. Students will provide the solutions by PRADO or email.

- Continuous assessment:
 - Quiz, test and games through PRADO, SWAD and Kahoot.

MEASURES TAKEN TO ADAPT ASSESSMENT (Instruments, criteria and percentage of final overall mark)



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Ordinary assessment session

- <u>Written exams about theoretical concepts.</u>. Percentage of the final mark: 55%. Individual exams by PRADO platform.
- <u>Written exams about theoretical concepts.</u> Percentage of the final mark: 15%. Individual exams by PRADO platform
- <u>Practical lessons assessment</u>. Percentage of the final mark: 10%, divided in two tasks: Laboratory book 30% Exam about practical contents : 70%
- <u>Continuous assessment</u>. Percentage of the final mark: 20%.

Extraordinary assessment session

- <u>Written exams about theoretical concepts</u>. Percentage of the final mark: 60%.
 - Individual exams by PRADO platform.
- <u>Written exams about theoretical concepts</u>. Percentage of the final mark: 30%.
 Individual exams by PRADO platform.
- <u>Practical lessons assessment</u>. Percentage of the final mark: 10%.

Single final assessment

- <u>Written exams about theoretical concepts.</u>. Percentage of the final mark: 60%. Individual exams by PRADO platform.
- <u>Written exams about theoretical concepts.</u>. Percentage of the final mark: 30%. Individual exams by PRADO platform
- <u>Practical lessons assessment</u>. Percentage of the final mark: 10%.
 For those students that have attended to the practical lessons and have not passed the exam there will be a virutal written exam in the PRADO platform.

Those students that have not atended to the practical lessons will have to pass a practical exam in the laboratory. The evaluation will be carried out by the teachers in charge of the practical lessons

ADDITIONAL INFORMATION (if necessary)

The move to scenario A to scenario B will be impossed by the health authorities determination due to the evolution of the pandemic caused by COVID-19.



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