Subject Guide

SEPARATION PROCESSES

Academic year: 2019-2020
(Last update 18/05/2019)

MODULE	CONTENT	YEAR	TERM	CREDITS	TYPE
Complements of Formation	Separation processes	3rd	2nd	6 ECTS	Optative

LECTURERS	CONTACT INFORMATION
María Eugenia García Rubiño	Department of Physical Chemistry. Faculty of Pharmacy. Campus Universitario de Cartuja. 18071 – Granada. Telephone: 958243829 Email: rubino@ugr.es
	TUTORSHIPS
	María Eugenia García Rubiño (Room 194) • Tuesday and Thursday: 9:30–11:30 • Wednesday: 10:30–12:30

DEGREE WITHIN THE SUBJECT IS TAUGH	
Pharmacy	

PRERREQUISITES and/or RECOMMENDATIONS

Proper knowledge about:
- Instrumentals Techniques
- General Chemistry
- Basic Physics and Physical Chemistry
- Organic Chemistry
- Inorganic Chemistry
- Biochemistry

DETAILED SUBJECT SYLLABUS

THEORETICAL SYLLABUS

- UNIT 1. Introduction to chromatography. History. Concept of chromatography. Classification. Equilibrium distribution. Linear isotherms. Distribution parameters. Linear elution chromatography. Retention parameters. Migration.
- UNIT 2. Theories of chromatography. Theory of plates. Column efficiency. Kinetic theory. General equation. Differences between c. G. And c. L. Resolution. Retention time. Optimum efficiency conditions of the column. Gradient elution and temperature programming. Applications. The calibration method using standards. Standardization areas. Internal standard.
- UNIT 3. Plane chromatography. CP and CCF. How the separation is performed. Performance characteristics. Variables affecting the rf. Qualitative and quantitative determinations.
- UNIT 4. Gas chromatography. Gc retention volume, specific volume. Pharmaceutical applications. Qualitative interpretation of a chromatogram. Relative retention. Oster relationship. Kovats retention index.
- UNIT 5. Gas chromatography instrumentation. Carrier gas. Sample injection. Columns. Stationary phases. Thermal conductivity detectors, flame ionization, electron capture, atomic emission. Attachment with mass spectrometry.
- UNIT 6. HPLC instrumentation. Mobile phase. Pressure system. Columns. Column filling. Detectors: uv-v absorbance, fluorescence, electrochemical, refractive index, dispersion.
- UNIT 7. Liquid chromatography. Classification. Adsorption chromatography. Stationary phases. Separation mechanism. Mobile phase, eluent forte (0b).
- UNIT 8. Liquid-liquid or distribution chromatography. Normal phase chromatography. Reverse phase chromatography. Mobile phase polarity index. Mechanism. Applications. Ion pair chromatography.
- UNIT 9. Ion exchange chromatography. Types of exchange resins. Ion exchange mechanism. Suppressor column chromatography.
- UNIT 10. Exclusion chromatography. Mechanism. Parameters. Applications.
- UNIT 11. Other chromatographs. Affinity chromatography. Matrixes and ligands. Biospecific and nonspecific circumvention. Supercritical fluid chromatography. Properties of supercritical fluids. Instrumentation. Stationary and mobile phases. Pressure effect. Detectors. Comparison with other methods. Attachment to mass spectrometry: chemical ionization under atmospheric pressure. Electro-spray.
- UNIT 12. Electrophoresis. Electrokinetic phenomena. Zone electrophoresis. Factors affecting electrophoresis. Immunoelectrophoresis.
- UNIT 13. Polyacrylamide gel electrophoresis. Application to the separation of proteins. Non-denaturing conditions. Ferguson representation. Denaturing conditions. P.a.g.e.-sds. Estimation of molecular masses. Transfer membranes. Electrofocusing. Two-dimensional electrophoresis.
- UNIT 14. Agarose gel electrophoresis. Application to the separation of nucleic acids. Pulsed-field electrophoresis.
- UNIT 15. Capillary electrophoresis. Instrumentation. Migration and plate height in e.c. Electroosmotic flow characteristics. E.Cdetection zone. Capillary isoelectric focusing. Capillary electrochromatography. Capillary chromatography. Micellar electrokinetic.
- UNIT 16. Sedimentation. Sedimentation and ultracentrifugation. Transport under centrifugal forces. Lamm equation. Solutions to the lamm equation. Svedberg equations. Determination of molecular parameters. Multicomponent systems. Sedimentation equilibrium. Density gradient equilibrium.

LABORATORY SESSIONS AND SEMINARS
Seminars

- Problems solving.

Laboratory sessions:

- PRACTICE 1. Separation of DNA fragments by electrophoresis.
- PRACTICE 2. HPLC

- PRACTICE 3. Potentiometric determination of phosphate in a yeast extract powder by ion exchange chromatography.
- PRACTICE 4. Liquid gel exclusion chromatography.

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 (4ª Edición)
- "Técnicas de separación en Química Analítica". R. Cela, R.A. Lorenzo y M.C. Casais. Editorial Síntesis. 2002. (1ª Edición)
- "Tecnicas Analiticas de Separacion" M. Valcarcel , Ed. Reverte.
- "Técnicas de separación en química analítica". Rafael Cela, Rosa Antonia Lorenzo, Ma del Carmen Casais
- Ed. Síntesis, 2003
- "Análisis instrumental" Kenneth A. Rubinson, Judith F. Rubinson - 2001 Pearson Educación

ASSESSMENT. ASSESSMENT CRITERIA, CONTRIBUTION OF THE DIFFERENT ACTIVITIES ON THE FINAL MARK, ETC.)

Two different types of assessments will be considered:

A) Continous assessment. The final mark for those students included in this assessment, will comprises three parts:
1. Written exam about the contents of the subject programme.
It will consists of answering questions (types: tests, applications, theoretical...) and solving numerical problems. To pass this exam it will be mandatory to demonstrate a homogeneous knowledge of the subject. The contribution of this part to the final mark will be 65 %
2. To obtain a positive evaluation is necessary to assist to all practice sessions and present a lab-report with the description and resolution of the experiments realized, and to pass the practical exam carried out by a written and/or oral exercise.
Complete practice sessions and pass the exam will be prerequisites for approve the subject. The contribution of practical work to the final mark will be 10%
3. Other activities: oral presentation, lectures attendance, Kahoots, guided tour of the Centre of Scientific Instrumentation, general attitude during the course and participation in class and/or laboratory, will be evaluated and will contribute with 25% to the final mark.

B) One examination. The students who fulfil the requirements specified by the University of Granada and presented in time the corresponding solicitude (two weeks after their registration), shall have the right to make this class of examination. It consists of a just one exam. Exam will consists of answering all questions necessaries (types: tests, applications, theoretical, lab sessions…) and solving numerical problems. To pass this exam it will be mandatory to demonstrate a homogeneous and unambiguous knowledge of the subject.

FURTHER INFORMATION

- The teaching will be made exclusively in Spanish
- The attendance to practical sessions in laboratory is obligatory.
- The attendance to the lectures is highly recommended.