SUBJECT GUIDE(∞)

INSTRUMENTAL TECHNIQUES

Curso 2020-2021

(Fecha última actualización: 07/07/2017)
(Fecha de aprobación en Consejo de Departamento: 30/06/2020)

MODULE	CONTENT	YEAR	TERM	CREDITS	**TYPE**
Chemistry	Instrumental Techniques	2st	2st	6	Compulsory

LECTURER(S) Luis Crovetto Gonzalez Mª Isabel Martínez Puentedura, Manuel Jiménez Durán	Department of Physical Chemistry. Faculty of Pharmacy. University of Granada Campus Universitario de Cartuja. 18071 -Granada (Spain) Telf.:+34 958-243823. luiscrovetto@ugr.es, 958-249473 martinez@ugr.es, 958-243824 mjduran@ugr.es, 958 246 212
	TIMETABLE FOR TUTORIALS OR LINK TO WEBSITE http://fisicoquimica.ugr.es/pages/docencia/curso_2021/_doc/horariotutorias2021
DEGREE WITHIN WHICH THE SUBJECT IS TAUGHT	
Pharmacy Degree	

PREREQUISITES and/or RECOMMENDATIONS)

Proper knowledge about:
- Maths
- General Chemistry
- Basic Physics and Physical Chemistry

BRIEF ACCOUNT OF THE SUBJECT PROGRAMME (ACCORDING TO THE DEGREE

Study of the most employed Instrumental Techniques in Pharmaceutical laboratory and Pharmaceutical research. The study will be theoretical and practical, and it will be applied to the resolution and interpretation of problems.

OBJECTIVES (EXPRESSED IN TERMS OF EXPECTED RESULTS OF THE TEACHING PROGRAMME)

- To show the importance of the Instrumental Techniques in the pharmaceutical field.
- To show the most employed techniques for the identification and quantification of pharmaceutical products.

- • To give the physicochemical principles which the Techniques are based on.
- • Description of the basic components of the employed instruments.
- • Running of the instruments.
- • Basis of the employed methodology.
- • To select the most suitable technique for the analysis and control of pharmaceuticals, and water, food and environmental analysis.
- • To know and apply the main techniques for research, from the point of view of their basis and from the instrumentation.

DETAILED SUBJECT SYLLABUS

Theoretical syllabus:

Lesson 1. Concept, interest and classification of the Instrumental Techniques.
Concept of the Instrumental Techniques in Pharmaceutical Sciences. Advantages and disadvantages of the instrumental methods. Pharmaceutical interest of Instrumental Techniques. Classification of Instrumental Techniques. Choice of a Technique.

Lesson 2. Introduction to Spectroscopy
General concepts. Nature and properties of the electromagnetic radiation: Photoelectric effect. Energy levels of atoms and molecules. Regions of the electromagnetic spectrum. Selection rules.

Lesson 3. Components of the instruments for Optical Spectroscopy
Set up and components of the instruments employed for Optical Spectroscopy. Radiation sources (continuous and discontinuous). Wavelength selectors (monochromators and filters). Detectors of radiation.

Lesson 4. Absorption of light
Lamber-Beer law about radiation absorption. Limitations and deviations of Lamber-Beer law. Absorbance and Transmittance range of minimum error.

Lesson 5. Atomic Spectroscopy
5.1. Introduction to Atomic Spectroscopy: Atomic spectra and selection rules. Effect of temperature in the Atomic Spectra. Atomization of the sample. Introduction of the sample.
5.2. Atomic Absorption Spectroscopy: Radiation sources. Flame atomization. Electrotermic atomization. Types of spectrophotometers. Interferences.
5.3. Atomic Emission Spectroscopy: Atomization instruments. Plasma techniques. Types of spectrophotometers. Applications. Flame emission spectroscopy. Clinic applications of Flame emission spectroscopy. Comparison between both atomic techniques.

Lesson 6. Vibrational or Infrared Spectroscopy
Infrared region of the electromagnetic spectrum. Vibration of diatomic molecules and potential

energy curve. Mechanisms of IR radiation absorption. Selection rules. Vibrational Spectra and strength constant in diatomic molecules. Anarmonicity. Vibration of polyatomic molecules. Instrumentation in IR spectroscopy. Applications of the IR spectroscopy: Identification of substances.

Lesson 7. Electronic Spectroscopy: Ultraviolet-visible
Electronic spectra: Vibrational structure of the electronic bands. Franck-Condon Principle. Dissociation Energy. Main electronic transitions in polyatomic molecules. Chromophore and auxochrome groups. Instrumentation. Applications of the Molecular Absorption Spectroscopy UV-visible.

Lesson 8. Fluorescence Spectroscopy
Theoretical basis of the fluorescence Spectroscopy. Molecular relaxation processes form molecules in excited state: Radiative and non-radiative processes. Types of spectra. Factors affecting the emission intensity. Kavanagh Law. Instrumentation. Quenching. Stern-Volmer equation. Energy transfer processes between molecules. Applications.

Lesson 9. Nuclear Magnetic Resonance (NMR) Spectroscopy
General concepts. Physicochemical basis of NMR. Proton- NMR. Chemical shift. Multiplicity: spin-spin coupling. Instrumentation. Applications. Interpretation of spectra.

Lesson 10. Mass Spectrometry
Physicochemical basis. Types of mass spectra. Components of the instrumentation. Introduction of the sample. Ionization techniques (EI, CI, DI, FI, MALDI, FAB, APcI and ESI). Mass analyzers (Magnetic sector, Quadruplex, TOF, ion trap, FTMS). Detectors. Information from a mass spectrum. Applications. Interpretation of spectra.

Lesson 11. Chromatography techniques
Concept of chromatography. Classification: according to physicochemical basis, phase combination and instrument. General methodologies. Cromatrographic theories. Cromatographic parameters.

Practical Sessions in the laboratory:

Session 1. Construction of spectrophotometric absorption graphic. Calculation of molar extintion coefficients. Spectrophotometric determination of a mixture of B_2 and B_{12}vitamins.

Session 2. Study of the dynamic quenching of lucigenin by chloride ions.

Session 3. Separation of pharmaceuticals by chromatography.

Session 4. Obtaining and interpretation of the infrared spectrum of acetilsalicitic acid.

READING

READING

Basics:

- Principios de Análisis Instrumental. (7ª Edición) Skoog-Holler-Crouch . Cengage Learning Técnico. 2019 .
- Análisis químico e instrumental moderno. Frederic Walton Harnold; Jorge Reyes. Ed. Reverté 2005. ISBN: 8429175199 ISBN-13: 9788429175196
- Espectroscopia Atómica y Molecular. J. Zúñiga Román. Pearson Educación.
- Fundamentos de Espectroscopía Molecular. C.N. Banwell. Ediciones del Castillo.
- Análisis Instrumental. K.A. Rubinson- J.F. Rubinson. Editorial Prentice Hall.
- Métodos Instrumentales de Análisis. H.H. Willard y col. Grupo Editorial Iberoamérica.
- Técnicas Instrumentales de Análisis en Bioquímica. Gavilanes Franco, José Gregorio. Editorial Síntesis.1999.
- Métodos Ópticos de Análisis. E. Olsen. Editorial Reverté, 1995.
- 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
- Process Analytical Technology: Spectroscopic Tools and Implementation Strategies for the Chemical and Pharmaceutical Industries. Ed. Katherine A. Bakeev. John Wiley & Sons, 2010.
- Métodos espectroscópicos en Química Orgánica Hesse, Manfred · Meier, Herbert · Zeeh, Bernd.ISBN: 9788477385226. Ed. Síntesis 2005
- Técnicas espectroscópicas en química analítica . Ríos Castro, Ángel ; Cruz Moreno Bondi, María , Simonet Suau, Bartolomé M. (coords.)Ed. Síntesis 2012

Monographs:

- Pharmaceutical and Medical Applications of Near-Infrared Spectroscopy, Second Edition. Emil W. Ciurczak, Benoit Igne. CRC Press, 2014
- Espectroscopia. Requena, Federico M. Pearson Publications Company (2005)
- Espectroscopía Infrarroja. Robert Conley. Editorial Alambra.
- Espectroscopia ultravioleta y visible. C.N. Rao. Ed. Alambra.
- Fluorescente Spectroscopy. A.J. Pesce. Marcel Dekker. New Cork.

- Espectroscopia de Resonancia Magnética. F. J. López. Addison Wesley Iberoamericana S.A.
- Introducción a la cromatografía. Abbott y Andrews, Exedra, Ed. Alhambra.
- Cromatografía en papel y placa delgada, J.A. Domínguez, OEA.
- Fundamentos de la cromatografía de gases. J. M. Storch de García, Exedra, Ed. Alhambra.
- Cromatografía de gases. I, Dabrio, Ed. Alhambra.
- Técnicas Instrumentales Fisicoquímicas. S. Senent. Publicaciones UNED
- Química Física (Vol. 1). M. Díaz Peña, A. Roig Muntaner. Editorial Alhambra. Química Física. P. Atkins. (8ª Ed). Editorial Médica Panamericana.
- Química Física. A. Requena. Prentice Hall. Prentice Hall.
- Fisicoquímica: Problemas y Soluciones. L. Labowitz. Editorial Paraninfo.
- Fisicoquímica. (Vol. 2). Ira N. Levine. 5ª Ed. Editorial Mc. Graw Hill.

- Química Física. J. Morcillo Rubio. 2ª Ed. Publicaciones UNED.
- Aplicación de las Nuevas Tecnologías a la Enseñanza Práctica de Técnicas Instrumentales. Proyecto de Innovación Docente 07-02-08 subvencionado por la UGR Ref. MVGPI2007
- Aplicación de las Nuevas Tecnologías a la Enseñanza Práctica de Técnicas Instrumentales (2ª parte). Proyecto de Innovación Docente con Código Nº 08-22 subvencionado por la UGR Ref. MVG/PI2008

Others:

https://www.chemedx.org/page/activity

- http://nautilus.fis.uc.pt/wwwfi/hipertextos/espectro/hiper_espectro.html
- https://www.coursehero.com/file/13810368/7-Beers-Law-and-Its-Implicationsppt/
- http://www.chm.davidson.edu/ChemistryApplets/spectrophotometry/BeersLaw.html
- http://www.sc.ehu.es/sbweb/fisica/cuantica/fotoelectrico/fotoelectrico.htm
- Prism Applet - Refraction and Dispersion
- WebSpectra - Problems in NMR and IR Spectroscopy
- Espectroscopía IR:
- IR Absorption Spectrometers
- IR Helper
- Vibración de una molécula diatómica
- Vibraciones de las moléculas diatómicas
- Espectroscopía de Fluorescencia:
- http://teaching.shu.ac.uk/hwb/chemistry/tutorials/molspec/lumin3.htm

RECOMMENDED INTERNET LINKS

General Spectroscopy:
http://jchemed.chem.wisc.edu/JCESoft/Issues/Series_B/9B1/prog3-9B1.html
http://nautilus.fis.uc.pt/wwwfi/hipertextos/espectro/hiper_espectro.html
https://www.coursehero.com/file/13810368/7-Beers-Law-and-Its-Implicationsppt/
http://www.chm.davidson.edu/ChemistryApplets/spectrophotometry/BeersLaw.html
http://www.sc.ehu.es/sbweb/fisica/cuantica/fotoelectrico/fotoelectrico.htm
Prism Applet - Refraction and Dispersion
WebSpectra - Problems in NMR and IR Spectroscopy
IR Spectroscopy:
IR Absorption Spectrometers
IR Helper
Vibración de una molécula diatómica
Vibraciones de las moléculas diatómicas
Fluorescence Spectroscopy:
http://teaching.shu.ac.uk/hwb/chemistry/tutorials/molspec/lumin3.htm
RMN Spectroscopy:
Basics of NMR
http—www.pharma.ethz.ch-people-oliver.zerbe-Vorlesung-NMR.pdf
http://teaching.shu.ac.uk/hwb/chemistry/tutorials/molspec/nmr1.htm
http://www.ch.ic.ac.uk/local/organic/nmr.html
Mass Spectrometry:
http://www.astbury.leeds.ac.uk/facil/MStut/mstutorial.htm
Chromatography:
http://www.sci.sdsu.edu/TFrey/Bio750/Chromatography.html

http://teaching.shu.ac.uk/hwb/chemistry/tutorials/chrom/gaschrm.htm
http://caspar.bgsu.edu/~courses/HPLC/HPLCManual.html
http://www.instrumentalchemistry.com/index.htm

TEACHING METHODOLOGY

- Master classes for theroy teaching
- Practical seminars regarding application problems of each lesson
- Practical lessons in the laboratory
- Quiz and test from online platforms for continuous evaluation

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

- Written exams about theoretical concepts. Percentage of the final mark: 50-55%.
  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.

- Written exams about mathematic resolution of application problems. Percentage of the final mark: 25-30%.
  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.

- Practical lessons. 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%

- Continuous evaluation and attending to theoretical classes. 10%.

2. Extraordinary call and single final evaluation

- Written exams about theoretical concepts. Percentage of the final mark: 55-60%.
  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.

- Written exams about mathematic resolution of application problems. Percentage of the final mark: 30-35%.
  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.
- Practical lessons. 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 (According to Official Academic Organization Plan)	TOOLS FOR TUTORIALS (Indicate which digital tools will be used for tutorials)
https://fisicoquimica.ugr.es/pages/docencia/curso_2021/_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 cannot 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).	
Ordinary 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 assesment 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 assesment 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 (According to Official Academic Organization Plan)	TOOLS FOR TUTORIALS (Indicate which digital tools will be used for tutorials)
https://fisicoquimica.ugr.es/pages/docencia/curso_2021/_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)	
Ordinary assessment session	
• Written exams about theoretical concepts. . Percentage of the final mark: 42-58%. Individual exams by PRADO platform. • Written exams about theoretical concepts. . Percentage of the final mark: 22 - 38%. Individual exams by PRADO platform • Practical lessons assessment. Percentage of the final mark: 10%, divided in two tasks: Laboratory book 30% Exam about practical contents : 70% • Continuous assessment. Percentage of the final mark: 10-20%.	
Extraordinary assessment session	
• Written exams about theoretical concepts. . Percentage of the final mark: 55-60%. • Individual exams by PRADO platform. • Written exams about theoretical concepts. . Percentage of the final mark: 30 - 35%.	

Individual exams by PRADO platform • Practical lessons assessment. Percentage of the final mark: 10%,
Single final assessment
• Written exams about theoretical concepts. . Percentage of the final mark: 55-60%. Individual exams by PRADO platform. • Written exams about theoretical concepts. . Percentage of the final mark: 30 - 35%. Individual exams by PRADO platform • Practical lessons assessment. 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 virtual written exam in the PRADO platform. Those students that have not attended 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 imposed by the health authorities' determination due to the evolution of the pandemic caused by COVID-19.