VNIVERSITAT DE VALÈNCIA

Course Guide
44708 Instrumental techniques in organic chemistry

COURSE DATA

Data Subject	
Code	44708
Name	Instrumental techniques in organic chemistry
Cycle	Master's degree
ECTS Credits	4.0
Academic year	2023 - 2024

Study (s)

Degree	Center	Acad. year	Period
2226 - M.D. in Organic Chemistry	Faculty of Chemistry	1	Annual

Subject-matter

Degree	Subject-matter	Character
2226 - M.D. in Organic Chemistry	4 - Instrumental techniques in organic chemistry	Obligatory

Coordination

Name	Department
DEL POZO LOSADA, CARLOS	325 - Organic Chemistry

SUMMARY

There are three main types of applications of the instrumental technics commonly used: 1) Identification and quantification of the components of a mixture, known compounds whose structure, physical properties and response versus the analytical technique have already been described, like pesticides analysis in food or waste water. 2) Structure determination of an unknown compound, whose structure should be defined in an unambiguous way: like a new drug or a new natural compound from a plant. 3) The study and research of the technique itself, addressed to the development of new methods, in order to improve the sensibility of resolution of the technique or to the development of new techniques derived from it.

The physical bases of each technique render them less or more suitable to be used in the first two types of application. The focus of the matter is to wider the scope of the knowledge, on the three types of application, already attained by the student in the degree studies. An so, introduce the students into the more modern analytical techniques that allow the identification and determination of the structure of different products obtained in a process and/or separation of components of complex reaction mixtures. At the same time, the aim is to provide the students with the means to solve a problem in an efficient way. This objective will be approached from three points of view: nuclear magnetic resonance (NMR), mass

spectrometry (MS) and X-ray crystallography (XRC)

PREVIOUS KNOWLEDGE

Relationship to other subjects of the same degree

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

Other requirements

No restrictions have been established in relation with other matters in the study plan.

Other types of requisites:
Despite the fact that the spectroscopic technique are not directly related with the reactivity of the molecules, the understanding of the relations that the atoms establish, is fundamental for the correct interpretation of the spectral properties, and its spatial placement into sites of the molecule is only feasible when the basis of organic chemistry are mastered. Thus, previous knowledge

OUTCOMES

2226 - M.D. in Organic Chemistry

- Students should apply acquired knowledge to solve problems in unfamiliar contexts within their field of study, including multidisciplinary scenarios.
- Students should be able to integrate knowledge and address the complexity of making informed judgments based on incomplete or limited information, including reflections on the social and ethical responsibilities associated with the application of their knowledge and judgments.
- Students should communicate conclusions and underlying knowledge clearly and unambiguously to both specialized and non-specialized audiences.
- Students should demonstrate self-directed learning skills for continued academic growth.
- Students should possess and understand foundational knowledge that enables original thinking and research in the field.
- Use different presentation formats (oral, written, slide presentations, boards, etc.) to communicate knowledge, proposals and positions.
- Be able to access to information tools in other areas of knowledge and use them properly.
- Saber participar en debates y discusiones, dirigirlos y coordinarlos y ser capaces de resumirlos y extraer de ellos las conclusiones más relevantes y aceptadas por la mayoría.
- Poseer habilidades sociales, un buen nivel de comunicación oral y escrita, así como capacidad para trabajar en equipo y con personas de diferentes procedencias.

- Competencias de gestión tales como la capacidad para la planificación y gestión de tiempo y recursos, así como para dirigir y tomar decisiones.
- Ser capaces de valorar la necesidad de completar su formación científica, en lenguas, en informática, asistiendo a conferencias o cursos y/o realizando actividades complementarias, autoevaluando la aportación que la realización de estas actividades supone para su formación integral.
- Profundizar en los principios en los que se basan las técnicas físicas e instrumentales.
- Profundizar en las técnicas instrumentales de análisis cualitativo y cuantitativo de compuestos orgánicos: espectroscopía infrarroja, espectroscropia ultravioleta, resonancia magnética nuclear, espectrometría de masas y difracción de rayos X.
- Saber aprovechar los datos extraíbles de los diferentes tipos de espectros de moléculas orgánicas y transformarlos en información de tipo estructural.

LEARNING OUTCOMES

The main objective in this subject is to provide the students the tools to solve the structure of small and medium size molecules through the analysis of their spectroscopical data. Additionally, they will learn how to face a specific problem, being able to identify and perform the correct structural analysis strategy to find the solution in a very efficient way. They are also included indications of sample preparation and the use of the corresponding techniques (IR, UV, NMR, X-Ray, etc.) that will qualify students for their future work, being able to process their own spectra.

Therefore, the final aims that we treat to reach in this subject can be summarized in the following points:

- Knowledge of the fundamental principles where are supported physical and instrumental techniques. Capacity to understand the data extracted from the different type of spectra from organic molecules, and its transformation in structural information, including both the atomic conectivity and stereochemical relationships.

- Good knowledge of the language of modern NMR together with an ample perception of this technique as a structural elucidation tool, applicable in diverse areas, being able to determine in each case which NMR data are necessary to address the solution of an specific problem.

- Design, selection and/or development products and chemical processes efficiently (ODS 7) that minimize their impact in the environment (ODS 14 and 15), taking advantage of the alternative row materials and generating the minor amount of residues possible (ODS 11)

DESCRIPTION OF CONTENTS

1. Introduction to spectroscopy

Absorption of electromagnetic radiation by organic molecules. Types of spectroscopic techniques. Infra - Red Absorption and types of vibrations. Structural information. Ultraviolet and Visible Spectroscopy. Electronic excitation and chromophore groups. The Beer -Lambert law. Basics of Nuclear Magnetic Resonance. Nuclear relaxation. The chemical shift and its measure. Relative intensity of the signals.

2. 1H NMR

Structural factors influencing the chemical shift of the proton. Spin- spin coupling: Types of coupling and coupling systems. Coupling constants. heteronuclear coupling. Chemical and magnetic equivalences. Two-dimensional proton NMR techniques. Nuclear Overhauser Effect (NOE). Quantitative applications.

3. 13C NMR. NMR of other nuclei of interest.

Structural factors influencing the chemical shift of the carbon. Couplings of carbon with hydrogen and other nuclei. Two-dimensional NMR techniques in 13C NMR. Characteristics of the NMR signal for other common nuclei: 15N, 19F, 31P.

4. Mass Spectrometry

Fundamentals. Ionization methods applied to the analysis of organic and bioorganic molecules. Types of analyzers. Analytical techniques coupled to mass spectrometers. Tandem Mass Spectrometry (MS-MS). Applications and practical aspects of the technique.

5. X-ray diffraction

Crystal symmetry. Space groups. Diffraction and crystal structure. Resolution and refinement of crystal structures (single crystal). Determination of absolute configuration. Determination of crystal packing: molecular crystals. Determination of intermolecular interactions and supramolecular assembly in a solid state. Databases.

WORKLOAD

ACTIVITY	Hours	% To be attended
Theory classes	20,00	100
Seminars	20,00	100
Study and independent work	40,00	0
Preparation of evaluation activities	10,00	0
Resolution of case studies	10,00	0
TOTAL	**100,00**	

TEACHING METHODOLOGY

The students will have previous access to the didactical material related with the course.

On site activities: Theoretical lectures with active participation where the professor will select those key concepts which are the backbone of the theme, and basic for the comprehension of the whole matter; will give a global vision of the theme and key points, and will indicate the resources four a deeper preparation of the theme by the students.

Seminars: they will be devoted to the resolution, along with their exposition and debate, of the problems previously presented in the "aula virtual" or directly presented by the professor. They should provide the students the ability to recognize the fundamental concepts related to the structural analysis of organic compounds, be able to relate those concepts and used them in a predictive way. A previous work of the students, either individual or in groups, is required.

EVALUATION

The assessment of student learning will be based on :

1) The results of the written exam (with a weighting of 80%).

2) Corresponding to the quality of exposure and / or debate on the resolution of theoretical and practical problems (with a weighting of 10 %) results.

3) Continuous assessment (with a weighting of 10%).

In the student record shall include a rating of the module, not the sections that compose it. The rating module is obtained as a weighted average of the ratings issued by different teachers of the same, taking into account in this regard the credits that each imparts. In any case, the minimum passing grade is necessarily of 4.0 points in each section.

REFERENCES

Basic

- Hesse, M.; Meier, H. y Zeeh, B. Métodos espectroscópicos en Química Orgánica, 2ª edición, Editorial Síntesis, 2005.
- Duddeck, H.; Dietrich, W.; Toth, G. Elucidación estructural por RMN, Ed. Springer New York, 1989.
- Ekman R., Silberring J., Westman-Brinkmalm A., Kraj A. Mass spectrometry (Instrumentation,Interpretation, and Applications), John Wiley & Sons, 2009.

- Hammond, C. The basics of Crystallography and Diffraction, Oxford University Press (IUCr Texts in Crystallography, 12) 3rd Edition, 2009.
- Massa, W. Crystal structure determination, Springer-Verlag, 2004.

Additional

- Pretsch, E.; Clerc, T.; Seibl, J.; Simon, W. Tablas para la determinación estructural por métodos espectroscópicos, Ed. Springer, Barcelona, 1998.
- Claridge, T. D. W. High-Resolution NMR Techniques in Organic Chemistry, 2nd Edition, Pergamon: Amsterdam, 2009.
- Keeler, J. Understanding NMR Spectroscopy, Wiley: Chichester, 2005.
- Dass, C. Fundamentals of Contemporary Mass Spectrometry, John Wiley & Sons, 2007.
- The Cambridge Structural Database (CSD), Comprehensive of the published literature and highly curated, is an essential resource to scientists around the world.
- Mercury - Crystal Structure Visualisation, Exploration and Analysis Made Easy
- ChemBioOffice Ultra, PerkinElmer (CambridgeSoft). Amplia selección de aplicaciones y funcionalidades que permite estudiar, dibujar, formular, modelar y editar estructuras moleculares químicas y biológicas.