Universitat Autònoma de Barcelona

2023/2024

Structural Determination

Code: 102532
ECTS Credits: 6

Degree	Type	Year	Semester
2502444 Chemistry	OB	3	1

Contact

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Teaching groups languages

You can check it through this link. To consult the language you will need to enter the CODE of the subject. Please note that this information is provisional until 30 November 2023.

Teachers

Juan Pablo Bayon Rueda

Jordi Hernando Campos

Ona Illa Soler

Prerequisites

- All teaching, including teaching materials handed out to the students, will be in English. Therefore, good communication skills in English are necessary.
- It is recommended that only those students who passed the 2nd-year subject "Spectroscopy" ("Espectroscòpia") enroll in "Structural Determination".
- It is assumed that the student is familiar with the basic concepts of organic and inorganic chemistry. Therefore, the students are expected to have taken the 2nd-year subjects "Estructura i Reactivitat dels Compostos Orgànics" and "Química dels Elements".

Objectives and Contextualisation

This course aims at providing the students with basic tools for the analysis of the spectroscopic data of organic and inorganic molecular compounds, thus enabling the students to elucidate their structure. Various spectroscopic and spectrometric techniques will be considered (mass spectrometry and UV-vis, infrared and nuclear magnetic resonance spectroscopies), though most efforts will be devoted to the analysis of NMR data.

Specific goals of this subject are:

- Introduce the mass spectrometry technique and review basic concepts of UV-vis, IR and NMR spectroscopies covered by the 2nd-year subject "Spectroscopy".

- Introduce advanced concepts in NMR spectroscopy.
- Use this knowledge to undertake the analysis of mass, UV-vis, IR and 1D NMR spectra of organic and inorganic molecular compounds.
- Elucidate the structure of those compounds based on their spectroscopic data.

Competences

- "Interpret data obtained by means of experimental measures, including the use of IT tools; identify their meaning and relate the data with appropriate chemistry, physics or biology theories."
- Adapt to new situations.
- Apply knowledge of chemistry to problem solving of a quantitative or qualitative nature in familiar and professional fields.
- Be ethically committed.
- Communicate clearly in English.
- Handle standard instruments and material in analytic and synthetic chemical laboratories.
- Learn autonomously.
- Manage the organisation and planning of tasks.
- Manage, analyse and synthesise information.
- Obtain information, including by digital means.
- Propose creative ideas and solutions.
- Reason in a critical manner
- Recognise and analyse chemical problems and propose suitable answers or studies to resolve them.
- Resolve problems and make decisions.
- Show motivation for quality.
- Use IT to treat and present information.
- Use the English language properly in the field of chemistry.

Learning Outcomes

1. Adapt to new situations.
2. Analyse chemical problems and plan suitable answers or studies for their resolution on a spectroscopic level, using molecular models and bibliographic sources.
3. Be ethically committed.
4. Communicate clearly in English.
5. Evaluate the best spectroscopic methodology to solve a structural problem.
6. Evaluate the capacities of the information contained in online networks.
7. Examine spectroscopic databases and other related bibliographic data.
8. Innovate methods for adaptation to the interpretation of a specific molecular structure.
9. Interpret the data obtained from experimental measurements to express a chemical structure.
10. Interrelate databases and calculation programs to determine a structure.
11. Learn autonomously.
12. Manage the organisation and planning of tasks.
13. Manage, analyse and synthesise information.
14. Obtain information, including by digital means.
15. Propose creative ideas and solutions.
16. Reason in a critical manner
17. Recognise and analyse structural chemical problems in organic and inorganic compounds.
18. Recognise the English terminology in bibliographic databases and online information.
19. Resolve problems and make decisions.
20. Show motivation for quality.
21. Use IT to treat and present information.
22. Use spectroscopic methods [IR, UV-VIS, NMR (1 H, 13 C) and ME] to solve problems of a quantitative or qualitative nature in the field of intra- and intermolecular structure and relations.
23. Use the most common English chemistry terms.

Content

1. Introduction to Mass Spectrometry (MS)

Background and the experimental method. Spectral resolution. Isotope analysis. Fragmentation processes: homolytic and heterolytic bond cleavage. Fragmentation patterns associated to specific functional groups. Examples.

2. Basic concepts in Electronic (UV-Vis), Infrared (IR) and Nuclear Magnetic Resonance (NMR) Spectroscopies.

The experimental methods. UV-vis chromophores in organic molecules. IR absorptions of organic functional groups and interpretation of IR spectra. Functional group charts (IR). Basic aspects of NMR spectra: chemical shifts, spectral ranges and referencing.

3. ^{1}H NMR: the chemical shift.

Shielding mechanisms. Topical relationships and molecular symmetry. Other factors influencing the chemical shift: magnetic anisotropy, solvent effects. Correlations: hydrogens linked to carbon, hydrogens linked to other nuclei. Spectral simulations. Examples.

4. ^{1}H NMR: spin-spin coupling.

Basic concepts on spin-spin interaction, coupling constants and multiplicity patterns. The Karplus equation. Spin systems: the $\Delta\nu/J$ ratio, first and second order spectra. Heteronuclear couplings. Examples.

5. ^{1}H NMR: analysis of the spectra.

Time-dependent phenomena. Methods of analysis. Simplification of spectra: changing the magnetic field, spin decoupling, shift reagents. Cross-relaxation and the nuclear Overhauser effect (NOE). Introduction to 2D NMR spectroscopy. Examples.

6. ^{13}C NMR.

Overview. Recording methods (broad band, off-resonance, DEPT). Chemical shifts: additivity and spectral simulations. Spin-spin couplings. Analysis of the spectra. Examples

7. NMR of other nuclei.

^{1}H NMR in inorganic compounds, including metal complexes. ^{31}P NMR, ^{19}F NMR, ^{14}N and ^{15}N NMR. Metal complexes: multinuclear NMR.

8. Structural determination.

Combined application of the spectroscopic techniques. Examples.

Methodology

Two different types of activities will be developed in the classroom:

Theory Lectures

The lecturer will explain the contents of the course to the classroom using blackboard or multimedia material, which will be made available to the students in the "Moodle". After a set of lecture sessions taking place during the initial weeks to introduce basic concepts, the rest of the theory lectures will be based on a "problem-based learning" approach. Students will be required to solve spectroscopic exercises during these sessions, for which a mark will be given.

Problem-solving Sessions

A set of exercises will be made available to all students in the "Moodle" at the beginning of the course. Several of these will be discussed by a teaching assistant during the problem-solving sessions. Alternatively, students will be required to solve spectroscopic exercises during these sessions, for which a mark will be given.

Important Notes

Teaching, including all teaching and evaluation materials (e.g. slides, problems, exams), will be given in English. Students are encouraged to use English as well when answering evaluation materials or communicating to the professors. Despite this, the use of Catalan and Spanish will also be accepted in both cases.

Annotation: Within the schedule set by the centre or degree programme, 15 minutes of one class will be reserved for students to evaluate their lecturers and their courses or modules through questionnaires.

Activities

Title	Hours	ECTS	Learning Outcomes
Type: Directed			
Problem-solving Sessions	12	0.48	5, 4, 20, 7, 13, 8, 9, 10, 23, 3, 15, 16, 17, 19, 22
Theory Lectures	37	1.48	5, 6, 4, 20, 7, 8, 9, 10, 23, 3, 15, 16, 17, 19, 22
Type: Autonomous			
Personal study	43	1.72	11, 6, 20, 12, 13, 23, 3, 14, 16, 17, 18
Problem Solving	46	1.84	1, 11, 5, 6, 20, 7, 12, 13, 8, 10, 23, 3, 14, 15, 16, 17, 18, 19, 22, 21

Assessment

Students could choose between two different evaluation modes: continuous evaluation and one-step evaluation.

Continuous evaluation

The overall mark will be broken down as follows:

Problems solving (15%) + Midterm Exam 1 (35%) + Midterm Exam 2 (50%) = 100%

The evaluation of students will comprise the following items:

Problem solving: A number of short exercises will be periodically handed out to the students. A mark will be given to each exercise. The weighted average mark of the exercises will account for 15% of the overall mark of the course.

Written exams: Two exams will be held during the course and they will account for 35% and 50% of the overall grade of the course, respectively.

To pass the subject, students must fulfil both of the following requisites:

A) The weighted average mark of the two exams must be at least 5/10.

B) The overall mark (problems + Midterm Exam 1 + Midterm Exam 2) should be at least 5/10.

In the case that these conditions are not met, a retake exam is also scheduled and will be compulsory for those with a weighted average mark for the two midterm exams lower than 5/10. Those with a passing grade but who wish to improve their mark may also take the final exam. Only those students that have taken both exams during the course are eligible to take the retake exam.

For those students taking the retake exam, the overall mark will be computed as follows:

Problems solving (15%) + Retake Exam (85%) = 100%

The formula will apply to all students who have taken the retake exam, regardless of whether the new mark is higher or lower than the original.

To pass the subject, students must fulfil both of the following requisites:

A) The grade of the retake exam must be at least 5/10.

B) The overall grade (problems + retake exam) should be at least 5/10

One-step evaluation

The overall mark will be directly obtained from a single final exam to be held at the end of the semester. If the mark of this exam is lower than 5/10, students should take a retake exam. Those with a passing grade but who wish to improve their mark may also take the retake exam. In both cases, the mark from the retake exam will replace that previously obtained for the single final exam. Only those students that have taken tha pevious exam will be eligible to take the retake exam.

To pass the subject, students must fulfil the following requisite:

A) The mark of the single final exam (or of the retake exam if necessary) must be at least 5/10.

Students taking less than 1/3 of the evaluation items will be graded as "no avaluable".

Assessment Activities

Title	Weighting	Hours	ECTS	Learning Outcomes
Exams	85%	8	0.32	1, 11, 5, 4, 20, 7, 12, 13, 8, 9, 10, 23, 3, 14, 15, 16, 17, 18, 19, 22
Problem Solving	15%	4	0.16	1, 2, 11, 5, 6, 4, 20, 7, 12, 13, 8, 9, 10, 23, 3, 14, 15, 16, 17, 18, 19, 22, 21

Bibliography

a) Text books

- D.H. Williams, I. Fleming, *Spectroscopic Methods in Organic Chemistry*, McGraw-Hill, London, 2007.
- R. Silverstein, F.X. Webster, D.J. Kiemle, *Spectrometric Identification of Organic Compounds*, Wiley, New York, 2005.
- P. Crews, J. Rodriguez, M. Jaspars, *Organic Structure Analysis*, Oxford University Press, New York, 2009.
- Simpson, Jeffrey H.*Organic Structure Determination Using 2-D NMR Spectroscopy - A Problem-Based Approach (2nd Edition).* Elsevier, 2012. e-book: link

b) Problems

- L.D. Field, S. Sternhell, J.R. Kalman, *Organic Structures from Spectra*, Wiley, Chichester, 2008.
- J. R. Pedro, G. Blay, 200 Problemas de Determinación Estructural de Compuestos Orgánicos, Vision Libros, Madrid 2010. ISBN 978-84-9983-993-6
- A. Randazo, Guía Práctica de Interpretación de Espectros de RMN, Loghia Publishing, Nápoles 2018. ISBN 978-88-95122-44-1
- Simpson, Jeffrey H.*Organic Structure Determination Using 2-D NMR Spectroscopy - A Problem-Based Approach (2nd Edition).* Elsevier, 2012. e-book: link

c) Tables

- E. Pretsch, P. Bühlmann, C. Affolter, A. Herrera, R. Martínez, *Determinación estructural de compuestos orgánicos*, Springer, Barcelona, 2002.
- E. Pretsch, P. Bühlmann, M. Badertscher, *Structure Determination of Organic Compounds*, Springer, Berlin, 2020. e-book: https://csuc-uab.primo.exlibrisgroup.com/permalink/34CSUC_UAB/1c3utr0/cdi_askewsholts_vlebooks_9

Software

Does not apply