PERSONA CIÈNCIA EMPRESA

COURSE: IDENTIFICATION AND STRUCTURAL CHARACTERIZATION

SUBJECT: Identification and Structural Characterization
MODULE: Technological
PROGRAM: University Master's Degree in Analytical Chemistry

GENERAL CHARACTERISTICS*

Type: ☐ Basic training, ☑ Obligatory, ☐ Elective

☐ Master's thesis work, ☐ Practicum

Duration: Semester **Semester/s**: 1
Number of credits ECTS: 5
Language/s: Spanish, Catalan, English

DESCRIPTION

SHORT DESCRIPTION AND JUSTIFICATION (of the sense of the course in relation to the studies. Between 100 and 200 words.)

This course gives to the students the necessary knowledge and skills to understand, select and use the NMR experiments appropriate to stablish the structure of chemical compounds. Furthermore, it gives knowledge of X-ray diffraction and thermal analysis techniques suitable for the characterization of solid substances. Organic molecules figure prominently in the course, but the techniques are also useful for advanced work and research with other kinds of compounds, such as biomolecules. An emphasis will be specially on the utility of the main techniques, and also in experimental aspects, such as sample preparation. Similarly, some related aspects (equipment economic cost, methods and security) will be included.

COMPETENCES (of the course in relation to the preassigned skills of the subject.)

Basic competences
CB6 - Have and understand knowledge which provides the ground or opportunity to be innovative in the development and/or application of ideas, often in a research context
CB7 - Apply their knowledge and their ability to solve problems in new or unfamiliar environments within broader (or multidisciplinary) contexts related to their field of study
CB8 - Integrate knowledge and deal with the complexity of formulating judgments based on information which, being incomplete or limited, includes reflections on social and ethical responsibilities related to the application of their knowledge and judgments
CB9 - Communicate conclusions, and the reasons that sustain them, to specialized and non-specialized audiences in a clear and unambiguous way.

Specific competences
E1 - Demonstrate advanced knowledge of NMR, X-ray diffraction and thermal analysis for designing, applying and interpreting analytical methods.
E2 - Ability to interpret the results obtained with NMR, X-ray diffraction and thermal analysis to identify and determine the structure of chemical compounds

* Estas características no deben ser modificadas sin la aprobación de los órganos responsables de las estructuras académicas de nivel superior (materia, módulo y/o plan de estudios).

PERSONA CIÈNCIA EMPRESA

COURSE: IDENTIFICATION AND STRUCTURAL CHARACTERIZATION

SUBJECT: Identification and Structural Characterization
MODULE: Technological
PROGRAM: University Master's Degree in Analytical Chemistry

Transversal competences
T3 - Ability to assess the impact of the use of chemistry in the sustainable development of the society

PREREQUISITES* (modules, subjects, courses or knowledge necessary to follow the course. Courses that should be previously taken can be figured here.)

Students of this Master coming from Chemistry degrees do not need any additional Training Complement. Students coming from other degrees should previously course subjects that include the basics of Organic Chemistry and Structural Determination (including IR, NMR, and MS).

CONTENTS (as a list of the sections that form the program of the course, to second-level detail.)

1. ORGANIC ELEMENTAL ANALYSIS.
   1.1. Basis.
   1.2. Applications.
2. ADVANCED NMR.
   2.1. Historical perspective and experiment compendium.
   2.2. The atomic nucleus.
   2.3. Nuclear magnetism.
   2.4. Resonance.
   2.5. Energy levels and spectra.
3. EXPERIMENTAL ASPECTS OF NMR
   3.1. The spectrometer.
   3.2. Sample preparation.
   3.3. Spectra acquisition.
4. PROCESSING OF NMR SPECTRA.
   4.1. 1D-NMR spectra.
   4.2. 2D-NMR spectra.
5. ^{1}H-NMR SPECTRA.
   5.1. The experiment.
   5.2. Spectral parameters.
   5.3. Exercises.
6. ^{13}C-RMN SPECTRA.
   6.1. ^{13}C-NMR experiments.
   6.2. Spectral parameters.

* Estas características no deben ser modificadas sin la aprobación de los órganos responsables de las estructuras académicas de nivel superior (materia, módulo y/o plan de estudios).

6.3. The APT experiment.
6.4. The DEPT experiment.
6.5. Exercises.

7. 1D-RMN SPECTRA OF OTHER ½-SPIN NUCLEI.
   7.1. ^{19}F-NMR spectra.
   7.2. ^{31}P-NMR spectra.
   7.3. ^{15}N-NMR spectra.
8. THE SECOND DIMENSION IN NMR.
   8.1. Data acquisition and processing.
   8.2. 2D-NMR spectra.
9. 2D-NMR SPECTRA WITH HOMONUCLEAR SCALAR CORRELATION.
   9.1. COSY spectra.
   9.2. TOCSY spectra.
   9.3. INADEQUATE spectra.
10. 2D-NMR SPECTRA WITH HETERONUCLEAR SCALAR CORRELATION.
   10.1. HSQC and HMQC spectra.
   10.2. HMBC spectra.
   10.3. HETCOR spectra.
11. 2D-NMR SPECTRA WITH CORRELATION THROUGH SPACE.
   11.1. Relaxation and NOE.
   11.2. Experiments of steady-state NOE.
   11.3. Experiments of transient NOE.
12. DYNAMIC NMR.
   12.1. Chemical exchange and NMR spectra.
   12.2. Molecular motion: DOSY spectra.
   12.3. Applications.
13. COMBINED PROBLEMS FO SPECTROSCOPY.
   13.1. Problems of 2D-RMN spectra.
   13.2. Combined problems of spectroscopy.
14. THE CRYSTALLINE STATE.
   14.1. Crystals, unit cells and crystalline systems.
   14.2. Crystals and symmetry.
15. X-RAY DIFFRACTION.
   15.1. The X-rays.
   15.2. Preparation of single crystals.
   15.3. Assembly and cell determination.
   15.4. Determination of the crystalline structure.
   15.5. Diffraction of powders.
   15.6. Analytic utility.

COURSE: IDENTIFICATION AND STRUCTURAL CHARACTERIZATION

SUBJECT: Identification and Structural Characterization
MODULE: Technological
PROGRAM: University Master's Degree in Analytical Chemistry

16. ANÁLISIS TÉRMICO
    16.1. Introduction.
    16.2. Thermogravimetry.
    16.3. Differential Thermal Analysis.
    16.4. Differential Scanning Calorimetry.

METHODOLOGY

TRAINING ACTIVITIES* (Fill the la table connecting activities, workload, in ECTS credits, and skills.)

Training activities	Hours / ECTS credits	Competences
Sessions of exposition of concepts	31 / 1.15	E1, E2, T3, CB6, CB7
Sessions solving exercises, problems and cases	4 / 0.15	E1, E2, T3, CB6, CB7
Seminars	2 / 0.07	E1, E2, T3, CB7, CB8, CB9
Practical work / laboratory	-	
Presentations	4 / 0.15	E1, E2, T3, CB8, CB9
Activities of personal study of the students	90 / 3.33	E1, E2, T3, CB6, CB7, CB8
Evaluation activities (exams, monitoring controls…)	4 / 0.15	E1, E2, T3, CB9
TOTAL	**135 / 5**	

TEACHING METHODOLOGY (justifying the didactic methods used in relation to the skills and contents of the course. Between 100 and 200 words.)

In the **sessions of presentation of concepts**, the concepts included in the program are presented using *classic techniques*: *chalk-blackboard* and *projections*.

Sessions of solution of exercises: Several collections of exercises are available, which are given progressively to the students throughout the semester. The exercises try helping the student to understand, deepen and relate the concepts studied in the sessions of presentation of concepts. Exercises of the different topics are solved in the classroom, with an emphasis on exercises that present major difficulties for the students.

* Estas características no deben ser modificadas sin la aprobación de los órganos responsables de las estructuras académicas de nivel superior (materia, módulo y/o plan de estudios).

PERSONA CIÈNCIA EMPRESA

COURSE: IDENTIFICATION AND STRUCTURAL CHARACTERIZATION

SUBJECT: Identification and Structural Characterization
MODULE: Technological
PROGRAM: University Master's Degree in Analytical Chemistry

Seminars: They tackle a particular topic, or a part of the course, in order to strengthen it or, simply, to solve doubts appeared during the study of the subject, without the time limitations typical of normal classes.

Presentations: Oral presentation to the teacher and, eventually, to other students, of a practical work or project ordered to students.

Individual or group consultations in the professor office: Students can consult the teacher individually or in groups of two to five persons.

Evaluation activities: Students answer one or more written control exams during the course, and a final written exam at the end of the course.

EVALUATION

ASSESSMENT METHODS* (Fill the table relating evaluation methods, skills and weights in the mark of the course.)

Evaluation methods	Weight	Competences
Final Exam	48%	E1, E2, T3 CB6, CB7, CB8
Monitoring activities	24%	E1, E2, T3 CB6, CB7
Projects and presentations	24%	E1, E2, T3 CB8, CB9
Participation	4%	T3

LEARNING OUTCOMES (Explication of the realizations of the student that allow the evaluation of skills, relating them to skills and evaluation methods.)

The student have to demonstrate his/her knowledge of the application of NMR to the identification and structural determination of the structure of chemical compounds (CB6, CB7, CB8, E1, E2, T3). Final exam, follow-up activities, Works and presentations, and Participation.

The student have to demonstrate his/her knowledge of the application of X-ray diffraction to the identification and structural determination of chemical compounds (CB6, CB7, CB8, E1, E2, T3). Final exam, Follow-up activities, Works and presentations, and Participation.

* Estas características no deben ser modificadas sin la aprobación de los órganos responsables de las estructuras académicas de nivel superior (materia, módulo y/o plan de estudios).

PERSONA CIÈNCIA EMPRESA

COURSE: IDENTIFICATION AND STRUCTURAL CHARACTERIZATION

SUBJECT: Identification and Structural Characterization
MODULE: Technological
PROGRAM: University Master's Degree in Analytical Chemistry

The student have to demonstrate his/her knowledge of the application of thermal analysis to the structural determination of chemical compounds (CB6, CB7, CB8, E1, E2, T3). Final exam, Follow-up activities, Works and presentations, and Participation.

The student have to know interpret the results from NMR, X-ray diffraction and thermal analysis for the identification and structural determination of chemical compounds (CB6, CB7, CB8, E1, E2, T3). Final exam, Follow-up activities, Works and presentations, and Participation.

The student have to demonstrate his/her knowledge of the main chemical properties of the most important chemical products, and the repercussions following of their incorrect use (CB9, T3). Final exam, Follow-up activities, Works and presentations, and Participation.

QUALIFICATION (Explanation of the system of calculation of the mark of the course.)

The grade of this course is obtained:

Final exam:	48%
Follow-up activities:	24%
Works and presentations:	24%
Participation:	4%

The **monitoring activities** will evaluate in writing the knowledge that the students achieve throughout the course.

The **projects and presentations** include jobs proposed during the course.

PERSONA CIÈNCIA EMPRESA

COURSE: IDENTIFICATION AND STRUCTURAL CHARACTERIZATION

SUBJECT: Identification and Structural Characterization
MODULE: Technological
PROGRAM: University Master's Degree in Analytical Chemistry

ASSESSMENT OF THE COMPETENCES (Define expressions to calculate each skill from the corresponding evaluation activities.)

Competences	Evaluation methods	Observations
Knowledge and understanding that provide a basis or opportunity for originality in developing and/or applying ideas, often in a research context (**CB6**).	Final exam Follow-up activities	Questions of theory.
That the students can apply their knowledge and their ability to solve problems in new or unfamiliar environments within broader (or multidisciplinary) contexts related to their field of study (**CB7**).	Final exam Follow-up activities	Questions of problems.
That the students are able to integrate knowledge and handle complexity, and formulate judgments based on information that was incomplete or limited, includes reflections on social and ethical responsibilities linked to the application of their knowledge and judgments (**CB8**).	Final exam Works and presentations	Questions of interpretation and justification of results.
That the students can communicate their conclusions, and the knowledge and rationale underpinning these, to specialist and non-specialist audiences, clearly and unambiguously (**CB9**).	Works and presentations	Specific activities in classroom, including seminars.
Posses advanced knowledge of NMR, X-ray diffraction and thermal analysis for application in drug research (**E1**).	Final exam Follow-up activities Works and presentations	Questions on structural analysis from different chemical sectors. Specific activities in classroom, including seminars.
Ability to interpret the results obtained with NMR, X-ray diffraction and thermal analysis to identify and determine the structure of chemical compounds (**E2**).	Final exam Follow-up activities Works and presentations	Questions of theory. Specific activities in classroom, including seminars.
Ability to asses the impact of the use of chemistry in sustainable development of society (**T3**).	Final exam Follow-up activities Works and presentations Participation	Questions of theory. Specific activities in classroom, including seminars.

The grade of each competence will be calculated applying the percentages indicated in the section "Qualifiquation" for each of the mark methods specified in the table, followed by normalization to 100%.

PERSONA CIÈNCIA EMPRESA

Universitat Ramon Llull

COURSE: IDENTIFICATION AND STRUCTURAL CHARACTERIZATION

SUBJECT: Identification and Structural Characterization
MODULE: Technological
PROGRAM: University Master's Degree in Analytical Chemistry

BIBLIOGRAFY (recommended and accessible to the student.)

- R.M. Silverstein, F.X. Webster & D.J. Kiemle, *Spectrometric Identification of Organic Compounds*, 7th edition, John Wiley & Sons, USA, 2005.
- M. Hesse, H. Meier & B. Zeeh, *Métodos Espectroscópicos en Química Orgánica*, 2ª edición ampliada y actualizada, Ed. Síntesis, Madrid, 2005.
- H. Friebolin, *Basic One- and Two-Dimensional NMR Spectroscopy, 4th Completely Revised and Updated Edition*, Wiley-VCH, Weinheim, 2005.
- J.K.M. Sanders & B.K. Hunter, *Modern NMR Spectroscopy. A Guide for Chemists*, Oxford University Press, Oxford, 1994.
- T.D.W. Claridge, *High-Resolution NMR Techniques in Organic Chemistry*, Pergamon, Oxford, 1999.
- J. Keeler, *Understanding NMR Spectroscopy*, John Wiley & Sons, Chichester, 2005.
- S.A. Richards & J.C. Hollerton, *Essential Practical NMR for Organic Chemistry*, John Wiley & Sons, Chichester, 2011.
- E. Breitmaier, *Structure Elucidation by NMR in Organic Chemistry*, John Wiley & Sons, Chichester, 1995.
- E. Pretsch, T. Clerc, J. Seibl & W. Simon, *Tablas para la Determinación Estructural por Métodos Espectroscópicos*, Springer-Verlag Ibérica, Barcelona, 1998.
- W. Clegg, *Crystal Structure Determination* (Oxford Chemistry Primers, 60), Oxford University Press, Oxford, 1998.
- C. Pico, M.L. López & M.L. Veiga, *Cristaloquímica de Materiales*, Ed. Síntesis, Madrid, 2007.
- R.A. Meyers (ed.), *Encyclopedia of Analytical Chemistry. Applications, Theory and Instrumentation*, John Wiley & Sons, Chichester, 2000.

PERSONA CIÈNCIA EMPRESA

COURSE: IDENTIFICATION AND STRUCTURAL CHARACTERIZATION

SUBJECT: Identification and Structural Characterization
MODULE: Technological
PROGRAM: University Master's Degree in Analytical Chemistry

DOCUMENT HISTORY

PREVIOUS CHANGES (Indicate date and author/s, the more recent first)
05-September-2016, Dr. Xavier Batllori
04-September-2014, Dr. Xavier Batllori

LAST REVISION (Indicate date and author/s.)
25-October-2017, Dr. Xavier Batllori