60454 - Fundamental methodologies in synthesis

Información del Plan Docente

Academic Year	2017/18
Faculty / School	100 - Facultad de Ciencias
Degree	543 - Master's in Molecular Chemistry and Homogeneous Catalysis
ECTS	2.0
Year	1
Semester	First semester
Subject Type	Optional
Module	---

1.General information

1.1.Introduction

Fundamantal Methodologies in Synthesis is an experimental subject that aims to empower students to the preparation and characterization of molecular structures by developing practical working skills in the laboratory, within the context of the Master's Degree in *Molecular Chemistry and Homogeneous Catalysis*.

In order to acomplish this goal, the synthesis of several organic and inorganic compounds requiring the use of synthetic techniques and usual working methods in research laboratories is proposed. Experimental sessions will be complemented by the development of a report containing the synthetic procedures performed and characterization of the products obtained.

Thus, the subject is constituted as a very important complement to theoretical training acquired in other subjects of the Master and the development of Master's Thesis.

1.2.Recommendations to take this course

Fundamantal Methodologies in Synthesis is a course aimed at students with different levels of experience working in a chemical laboratory chemical synthesis and catalysis. It is recommended to all students who wish to improve their skills in a chemical research laboratory.

1.3.Context and importance of this course in the degree

Fundamantal Methodologies in Synthesis is a 2 ECTS subject which is included in the module entitled Structural Characterization. It is a practical subject aimed to help the student expand his/her experimental skills in chemical synthesis. The training will empower him/her for performing advanced work in a chemical research laboratory and also for the development of the practical work of both the subjects included into the basic module of the Master, and also of the Master's Thesis.

1.4.Activities and key dates

The course will be held throughout the first half of the academic year. Practical sessions will take place in the teaching laboratories of the Sciences Faculty. The practical work will be carried out during the October to December period.

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The information about schedules, calendars and exams is available at the websites of the Sciences Faculty, https://ciencias.unizar.es/calendario-y-horarios, and the Master, http://masterqmch.unizar.es.

2.Learning goals

2.1.Learning goals

In order to succesfully pass this subject, the student must show to be capable of:

Handling wisely the basic equipment and techniques which are of common use in a research laboratory.

Knowing the Safety rules on a research laboratory.

Develop observation skills and decision-making abilities.

Applying synthetic strategies for the preparation of chemical compounds.

Relating theoretical concepts with the observed experimental facts.

Preparing rigorous reports, demonstrating ability to interpret and analyze the results.

2.2.Importance of learning goals

The set of knowledge and skills acquired during the course will provide the student tools and resources that will help him/her to develop his/her work in the chemical laboratory, either for the completion of a doctoral thesis or joining the chemical industry. Such knowledge and skills will also be useful for the realization of Master's Thesis.

3.Aims of the course and competences

3.1.Aims of the course

This course aims to offer students the opportunity to expand their experimental skills in chemical synthesis, regardless of previous experience. The student will conduct synthetic procedures using some of the techniques and methodologies commonly used in research laboratories. As a result the student will improve their capacity for critical analysis of the procedures used and data collected, as well as his/her ability to present scientific results. All this will result in a significant benefit towards his/her incorporation in chemical research laboratories.

3.2.Competences

To apply protocols, procedures and advanced synthetic techniques.

To get information on the degree of risk, toxicity and environmental implications of any chemicals, in order to handle them safely and responsibly.

To expand and use the vocabulary and specific terminology within the framework of Organic , Inorganic, Organometallic Chemistry and Catalysis.

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Capacity to design, plan and carry out synthetic processes of new organic, inorganic or organometallic molecules, of scientific, industrial and technological interest.

To be able to select and use independently different instrumental and structural determination techniques.

To investigate, understand and interpret the mechanisms of chemical reactions.

Capacity to understand, evaluate and interpret research findings in Molecular Chemistry and Catalysis, and relate them to the theoretical knowledge.

4.Assessment (1st and 2nd call)

4.1.Assessment tasks (description of tasks, marking system and assessment criteria)

Continuous assessment of this subject will be based based on the following criteria:

1.- Evaluation of experimental skill (quality of work performed and the results obtained), 40%.

2.- Laboratory Notebook, 10%.

3.- Laboratory report, 50%.

Students who have not passed this type of evaluation or want to improve their score may be submitted to an overall test in which they will have to demonstrate having achieved the expected learning outcomes. The number of official examination calls per registration and their use will be subjected to the statements of the *Regulation of Permanence in Master Studies* and the *Regulation of the Learning Assessment* (http://www.unizar.es/ice/images/stories/calidad/Reglamento%20Evaluacion.pdf). The latest document will also regulate the general design and scoring criteria of the assessment activities, as well as the exam schedules and timetable for the post-examination review.

5.Methodology, learning tasks, syllabus and resources

5.1.Methodological overview

The methodology followed in this course is oriented towards achievement of the learning objectives. A wide range of teaching and learning tasks are implemented, such as:

1. Laboratory sessions: the synthesis of a series of organic and inorganic products requiring the use of standard synthetic methodologies.

2. A report: the preparation of an experimental work containing the synthetic performed procedures, the characterization of the prepared products, and interpretation of the data.

5.2.Learning tasks

The course includes the following learning tasks:

- Laboratory sessions (2 ECTS). This course includes 9 working sessions (3 hour each) in the laboratory. Given the experimental nature of this course, these sessions may be extended to 30 hours if necessary.
- Individual or reduced group tutorials.

5.3.Syllabus

The course, **Fundamantal Methodologies in Synthesis**, includes 9 working sessions (3 hour each) in the laboratory. It will address the following topics:

Topic 1. Synthesis of a 4-arylidene oxazolone compound.

- The synthesis and purification of an oxazolone derivative will be carried out. The isolated compound will be characterized by IR and NMR spectroscopies.

Topic 2. Titration of an organomagnesium compound.

- A tetrahydrofuran solution of phenylmagnesium bromide will be titrated, by reaction with a phenylhydrazone under a protected atmosphere.

Topic 3. Synthesis of N-Boc diphenylprolinol.

- Bulk N-Boc diphenylprolinol will be prepared under inert atmosphere. The product will be subsequently purified by column chromatography and characterized by polarimetry and NMR spectroscopy.

Topic 4. Synthesis of Nickel complexes.

- Two Nickel compounds will be synthesized under strict oxygen- and water-free atmosphere. Firstly the sandwich, 20-valence electron, Ni(C5H5)2 derivative (nickelocene) will be prepared, and its magnetic moment will be experimentally determined. Secondly, a half-sandwich derivative stabilized by a N-heterocyclic ligand will be isolated and characterized by NMR spectroscopy.

Topic 5. Synthesis of molybdenum complexes.

- Two common molybdenum starting materials will be synthesized under strict oxygen- and water-free conditions. Firstly, the monohydride, 18-valence electron derivative MoH(C5H5)(CO)3 will be prepared, isolated and characterized by NMR spectroscopy. Secondly, the highly unstable, paramagnetic, 15-valence electron derivative Mo(C5H5)Cl4 will be isolated and subsequently analyzed by EPR spectroscopy.

5.4.Course planning and calendar

The laboratory sessions will be carried out preferably during the period between October and December. Work sessions and assessment tests will be communicated in advance.

Further information concerning the timetable, classroom, assessment dates and other details regarding this course, will be provided on the first day of class or please refer to the Faculty of Science website (https://ciencias.unizar.es).

Students will be provided with diverse teaching material either at reprography or through the University's virtual platform https://moodle2.unizar.es/add.

5.5.Bibliography and recommended resources

BB Armarego, W. L. F. Purification of laboratory chemicals [recurso electrónico] / Wilfred L.F. Armarego, Christina Li Lin Chai. - 6th ed. 6th ed.

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BB Arnáiz García, Francisco Javier. Síntesis de compuestos inorgánicos y organometálicos: una guía para el laboratorio / Francisco J. Arnáiz [Burgos]: El autor, 2011

BB Csák, Aurelio G. Técnicas experimentales en síntesis orgánica / Aurelio G. Csák, Mª Angeles Martínez Grau. 2ª ed. corr. y amp. Madrid: Síntesis, 2012

BB Synthesis of organometallic compounds: a practical guide / edited by Sanshiro Komiya Chichester [etc]: John Wiley & Sons, cop. 1997

BC Armarego, W. L. F. Purification of laboratory chemicals / W. L. F. Armarego and D. D Perrin. - 5th ed. Amsterdam [etc.]: Butterworth-Heinemann, 2003

BC Crabtree, Robert H. The organometallic chemistry of the transition metals / Robert H. Crabtree. - 4th ed. Hoboken, N.J.: Wiley-Interscience, cop. 2005

BC Elschenbroich, Christoph. Organometallics / Christoph Elschenbroich; translated by Jose Oliveira and Christoph Elschenbroich. - 3rd completely rev. and extended ed. Weinheim [etc.]: Wiley-VCH, cop. 2006

BC Hartwig, John F. Organotransition metal chemistry: from bonding to catalysis / John F. Hartwig Sausalito, Ca.: University Science Books, 2010

BC Rodríguez Yunta, María Josefa. Curso experimental en química orgánica / Mª Josefa Rodríguez Yunta, Fernando Gómez Contreras Madrid: Síntesis, D.L. 2008

URLs LIST:

Reception manual on risk prevention. Safety regulations in Chemistry laboratories. Elena Atrián Blasco, Vanesa Fernández Moreira. Instituto de Síntesis Química y Catálisis Homogénea, 2013.
http://82.223.223.103:8080/CEQMA/download/ManualAcogidaISQCH.pdf?id=listadoDocumentos