Subject Guide

1. Information about the subject

SUBJECT Experimentation in Organic Chem		nistry II	CODE	GQUIMI01-4-004
EDUCATIONAL OFFER	CENTER	Facultad de Química		
TYPE	Compulsory	N° TOTAL CREDITS	6.0	
PERIOD	First Semester	LANGUAGE	Spanish	
COORI	EMAIL			
Rodríguez Iglesias Felix	frodriguez@uniovi.es			
LE	EMAIL			
Aguilar Huergo Enrique	eah@uniovi.es	(English Group)		
BRIEVA COLLADO MARI	rbrieva@uniovi.es	vi.es		
Rodríguez Iglesias Felix	frodriguez@uniovi.es	(English Group)		
Cabal Naves Carmen Mai	pcabal@uniovi.es	(English Group)		
Ballesteros Gimeno Alfred	abg@uniovi.es			
LOPEZ GARCIA LUIS AN	lalg@uniovi.es			

2. Context

The course *Experimentation in Organic Chemistry II (EOQII)* is the second of the two experimental courses of the *Organic Chemistry subject* within the *fundamental module* of the Bachelor's Degree in Chemistry. This course is closely related to the course *Experimentation in Organic Chemistry I (EOQI)* and to the theoretical courses of the subject *Organic Chemistry: Organic Chemistry I* and *II*, which are taught in the second and third years of the degree. It is necessary to master those previous courses in order to obtain an adequate use of this course.

In *EOQII* students will consolidate the competences acquired during *EOQI* related to the work in the laboratory of Organic Chemistry. All the basic laboratory operations that students had occasion to know during *EQOI* will be routinely used along the course. Students will also be familiarized with new laboratory techniques of greater complexity and accuracy. In addition, the chemical transformations that take place in this laboratory involve chemical reactions of greater experimental difficulty.

On the other hand, after the previous courses students taking *EOQII* should have a basic knowledge of the spectroscopic methods of structural determination. For this reason, this fundamental aspect of the work in an organic chemistry laboratory will be deeply developed in *EOQII*. In this sense, the structural determination of the synthesized compounds will be carried out by standard spectroscopic techniques.

The teaching activities for each course instructor are included in the teaching plan of the Department of Organic and Inorganic Chemistry for the academic year 2016-2017, and are detailed below:

- Coordinators: Alfredo Ballesteros Gimeno (course coordinator)/Enrique Aguilar Huergo (coordinator of English groups)
- Lab Practices (LP's): LP1-I (Enrique Aguilar Huergo, Carmen Mª Paz Cabal Naves); LP2-I (Félix Rodríguez Iglesias).
- Classroom Practices (CP): The instructors who teach each one of the groups of Laboratory Practices (LP), detailed above, will teach the corresponding Classroom Practice (CP) to each group of students.

3. Requirements

The prerequisites established for all the courses of the *fundamental module* are to have passed the basic *subject Chemistry*.

Additionally, to be enrolled in *EOQII* the students must have passed the courses *Organic Chemistry II* and *EOQI*, which are programmed during the third year of the degree; moreover, a well-consolidated knowledge of them is an essential requirement to get a good benefit of *EOQII*.

4. Competencies and learning results

The main goal of the course *EOQII* is to contribute to develop in the student the competences included in the *Organic Chemistry* subject of the *fundamental module*, which are presented in the Memory of the Degree in Chemistry in the section CG (general competences) and CE (specific competences), (see pages 52 to 54 in the Memory).

Below are detailed the general and specific competences that the student must have achieved by taking successfully the course EOQII:

General Competences:

- CG-1 Rationalization and analysis abilities
- CG-2 Being capable of effectively solving specific problems
- CG-4 Planning and organizing abilities
- CG-5 Decision making abilities
- CG-6 Manage information properly
- CG-7 Use of a foreign language (English)
- CG-9 Self-learning abilities
- CG-12 Have environmental conscience
- CG-17 Develop critical thinking
- CG-18 Work in teams
- CG-20 Acquire or possess basic skills in ICT (Information and Communication Technologies)

Specific Competences:

- CE-1 Acquire the fundamentals of chemical terminology, nomenclature, conventions and units
- CE-2 Relate macroscopic properties to those of individual atoms and molecules
- CE-5 Describe the types of chemical reactions and their main characteristics
- CE-6 Apply the principles and procedures used in the chemical analysis for the determination, identification and characterization of chemical compounds
- CE-7 Apply the principles of thermodynamics and its applications in Chemistry
- CE-8 Understand the kinetics of the chemical change, including catalysis and reaction mechanisms.
- CE-10 Recognize the nature and behavior of the functional groups in organic molecules and the main routes of synthesis in Organic Chemistry
- CE-11 Deduce the properties of organic and organometallic compounds

- CE-19 Demonstrate knowledge and understanding of the essential facts, principles and theories related to chemistry
- CE-20 Solve quantitative and qualitative problems, according to previously developed models
- CE-22 Acquire skills to evaluate, interpret and analyze chemical information
- CE-24 Data processing and computing, related to chemical data and information
- CE-25 Safe manipulation of chemical reagents, labware and instruments
- CE-26 Performing standard synthetic and analytical lab procedures
- CE-27 Monitoring, by observation and measurement, chemical properties, changes or events, compiling relevant information
- CE-28 Planning, designing and performing practical research from the problem-discovery stage to assessment and evaluation of results
- CE-29 Employing standard instrumentation for identification, quantification, separation and structural determination applied to several disciplines
- CE-30 Interpretation of data coming from observations and lab measurements in terms of significance and theoretical support
- CE-31 Measuring the risks in the employment of chemical substances and procedures in the laboratory.
- CE-32 Correct use of the inductive and deductive methods in the environment of Chemistry
- CE-33 Recognize and value chemical processes in daily life.

Learning Outcomes

The proposed goals should be transformed into the following **learning outcomes**:

- 1. To consult and use scientific information effectively.
- 2. To recognize and analyze new problems in the field of Organic Chemistry and plan strategies to solve them.
- 3. To prepare and present a report correctly both orally and in writing (experimental procedure).
- 4. To manage computer programs in the field of Organic Chemistry.
- 5. To demonstrate knowledge and understanding of the facts, concepts, principles and theories related to Organic Chemistry and its application to solve problems.
- 6. To relate the fundamentals of analytical, spectroscopic and structural research techniques with their applications.
- 7. To recognize and assess risks in the use of chemicals and laboratory procedures, which involves safe handling of chemicals and standard chemical instrumentation.
- 8. To perform laboratory, analytical and synthetic practices, with scientific rigor in the measurement, in the operational procedure and in obtaining data.
- 9. To know and use, with safety and respect to the environment, laboratory techniques for the analysis, synthesis and characterization of chemical substances, including the necessary calculations and expressing the results in an appropriate manner.
- 10. To follow-up a chemical reaction by observing and measuring chemical properties, collecting the appropriate information and relating it to the theoretical concepts on which it is based.

5. Contents

This course is carried out in a laboratory of experimentation in Organic Chemistry, and puts special emphasis on the techniques, methods of synthesis and characterization of organic compounds. In view of this latter point, emphasis will be placed on the structural determination of organic compounds by spectroscopic methods. Therefore, a series of experiments, which are detailed below, will be carried out, covering different experimental techniques and types of reactions. The experiments have been classified thematically and their ordering is not related to the temporal order in which they will be carried out, which may be variable.

It is possible that not all students have time to complete all of the proposed experiments, although everyone will have to make a minimum number of them.

There is no doubt that an important part of the work of an organic chemist requires the use of different spectroscopic techniques, especially infrared (IR) spectroscopy and nuclear magnetic resonance (NMR). In this sense, it is intended that the students carry out a detailed analysis of the available spectroscopic information for each compound obtained in each of the experiments performed. To this end, a part of the course is devoted to the use of spectroscopic techniques in the structural elucidation of organic compounds.

On the other hand, the students will be trained in the work under an inert atmosphere, to carry out chemical reactions with reagents sensitive to humidity and / or oxygen.

The course is divided in the following parts:

Part A. Structural Determination.

Application of Infrared (IR) and Nuclear Magnetic Resonance (NMR) spectroscopic techniques to the structural determination of organic compounds: Preparation of samples, acquisition and interpretation of spectra.

They will be used in all the experiments proposed in Part B.

Part B. Synthesis.

- Synthesis of 3-iodo-4-phenyl-quinoline.
- Synthesis of 3,5-bis(ethoxycarbonyl)-2,4-dimethylpyrrole by the Knorr procedure.
- Generation of dichlorocarbene and its addition to styrene. Synthesis of (2,2-dichlorocyclopropyl) benceno.
- Enamines: Acetylation of cyclohexanone through its pyrrolidine-derived enamine.
- Alpha-methylenation of aldehydes.
- Wadsworth-Emmons reaction: Synthesis of *E,E*-1,4-diphenyl-1,3-butadiene.
- Preparation and employment of a Grignard reagent.
- Chemoselectivity in the reduction of 3-nitroacetophenone.
- Employment of protective groups in peptide synthesis.

6. Methodology and working plan

As an experimental course, it will consist mostly of the students work in the laboratory, where they will acquire the competences and skills of the course.

Attendance at experimental sessions is mandatory.

In general, the experiments will be performed individually by the students, using the lab scripts as a reference.

In accordance with the Procedure for the elaboration of the plan for the organization of regulated teaching adapted to RD 1393/2007 (approved in the Governing Council of the University of Oviedo on 04/29/2012), the maximum number of students per group is 10.

The course consists of 18 sessions of three hours devoted mostly to the work of students. During the initial session the general rules of Safety in the Laboratory, good practices, hygiene and environmental respect will be remembered and the laboratory will be presented before starting the experimental work. The rest of the sessions will follow the next pattern. The teacher will assign each student an experiment well in advance. The student must prepare the experiment using the material available in the Virtual Campus and consulting the appropriate bibliography. Experimentation will not begin until the student is fully aware of the basis of the experiment he/she is about to perform. Most experiments are multi-step processes, which last for more than one session. Once the experiment is

concluded, the student must present the results of his/her experiment, the characterization data of the products obtained and the laboratory notebook, where all the observations must be recorded in a rigorous way.

The Program has included a number of experiments that could exceed the time available in this course but it will not be necessary for all students to complete all the proposed practices.

As part of the continuous evaluation, one or two very brief written tests may be carried out during the lab period. Also, each of the students may have to explain briefly, in front of the rest of the group, some aspects of the experiments (expositions to the group). On the other hand, the instructor will carry out the continuous monitoring of the activity of the students during the lab period.

All the material needed for the course (digital presentations, lab scripts, bibliography, preliminary questions) will be provided through the Virtual Campus.

According to the recommendations of the University of Oviedo regarding the teaching organization, the distribution of the different types of activities is indicated in Table 2. On the other hand, the proposed temporal distribution (schedule) for the different activities is shown in Table 3.

Table 2. Distribution of teaching activities in the EOQ II course.

MODA	ALITIES	Hours	%	Total	
In- class	Lectures				
	Classroom practices/Seminars/Workshops	6.0	6.0 4		
	Group Tutorials			60	
	Laboratory Sessions	50	33.3		
	Evaluation Sessions	4	2.7		
Out-	Team work				
of- class	Personal work	90	60	90	
	Total	150			

Table 3. Schedule of teaching activities in the EOQ II course

			IN-CLASS WORK							OUT-OF-CLASS WORK		
Topics	Tota l Hou rs		Classroo m practice s/ Seminar s/ Worksho ps	Laborato ry Sessions	Group Tutori als	Other activiti es	Evaluati on Sessions	Tot al	Tea m wor k	Person al work	Tot al	
Experiments 1-2	24		0.5	9				9.5		14.5	14.5	
Experiment 3	16.5		0.5	6				6.5		10	10	

Experiment 4	20,5	0.5	9			9.5	11	11
Experiments 5-6	27,5	1	12			13	14.5	14.5
Experiments 7-8	27.5	1	12			13	14.5	14.5
Structural Determinatio n	30	2.5	2			4.5	25.5	25.5
Evaluation Sessions	4				4	4		
Total	150	6.0	50		4	60	90	90

7. Evaluation of the student's learning results

Regular Call

The evaluation of this course will be done by a combination of 1) *Continuous Assessment* and 2) *Examination*. The contribution of each part and the proposed criteria and evaluation tools are shown in Table 4.

Table 4. Assessment for the course EOQ II.

Part		Criteria	Evaluation Tools	Weight
1) Continuous	a) Lab activity	Attitude and interest in the lab, participation, preparation of the experiments and results of the experiments will be assessed.	Results of the experiments, interviews with the instructor, group discussions during lab period and written tests.	40 %
Assessment	The proper description of the experiments and the		Review of lab notebook.	10 %
2) Examination		Solve problems and issues related to lab practices and techniques	Written exam after the end of lab period.	50 %

The final mark of the course will be the result of the arithmetic mean of the marks obtained in parts 1) *Continuous Assessment* and 2) *Examination*.

To pass the subject the student must have obtained at least a 3.5-point mark in each of these two parts.

The note in the *Continuous Assessment* part will be the result of the evaluation of: *Student Lab Activity* (40%) and *Lab Notebook* (10%).

At the same time, the *Student Lab Activity* mark will be obtained from: (a) one or two written tests, (b) group presentations, and (c) instructor's personal notes regarding the student's work and reasoning ability in the lab.

Lab Notebook: It will be written in the lab itself and it will be assessed considering the order and clarity of its content, as well as the reproducibility of its experiments.

Final Written Exam: It will consist of several exercises and questions that will be related to the experiments carried out in the lab and to general aspects of them.

The evaluation system described above will be valid for the regular call of January.

Extraordinary Calls

The final mark in May and June Extraordinary Calls will be obtained in both a practical (test score: 50%) and a written exam (test score: 50%). A minimum mark of 3,5 (out of 10) points in each one of those two exams will be required to pass the course.

Those students who had obtained a mark higher than 3.5 points in section 1) Continuous Assessment in the ordinary exam, may avoid the Practical examination of the extraordinary call. In this case, the mark of the Practical Examination of the extraordinary call will be that obtained by the student in section 1) Continuous Assessment in the ordinary call.

Those students who had obtained a mark higher than 3.5 points in section 2) Examination in the ordinary exam, may avoid the written exam of the extraordinary call. In this case, the mark of the Written Exam of the extraordinary call will be that obtained by the student in section 2) Examination in the regular call.

8. Resources, bibliography and complementary documentation

All didactic material needed (lab scrips, FIDs and copies of NMR spectra) will be available in Virtual Campus. In addition, the following Bibliography is proposed, especially the first two titles:

- Recommended textbooks:
 - M. A. Martínez Grau. Técnicas experimentales en síntesis orgánica, 2ª Ed. Síntesis. 2012.
 - L. M. Harwood, C. J. Moody, J. M. Percy, Experimental organic chemistry: standard and microscale, Blackwell Science. 1999.
- Other textbooks for additional consulting:
 - J. C. Gilbert, S. F. Martin, Experimental Organic Chemistry: A Miniscale and Microscale Approach, Cengage Learning. 2010.
 - L. F. Tietze, T. Eicher, U. Diederischen, A. Speicher, Reactions and Syntheses in the Organic Chemistry Laboratory, Wiley-VCH.
 2007.
 - A. I. Vogel, B. S. Furniss, A. J. Hannaford, P. W. G. Smith, A. R. Tatchell, *Vogel's Textbook of Practical Organic Chemistry*, Longman Group UK. 5th Edition. **1999**.
 - K. P. Vollhardt, N. E. Schore, Organic Chemistry: Structure and Function, 7th Ed., Freeman and Company. 2014.
- Links of interest:
 - Organic Syntheses: http://www.orgsyn.org/
 - Journal of Chemical Education: http://pubs.acs.org/journal/jceda8