Bachelor´s Degree in Chemistry

Subject Guide

1. Information about the subject

SUBJECT	Experimentation in Inorganic Chemistry II		**CODE**	GQUIMI01-4-003
EDUCATIONAL OFFER	Bachelor´s Degree in Chemistry	**CENTER**	Facultad de Química	
TYPE	Compulsory	**Nº TOTAL CREDITS**	6.0	
PERIOD	Second Semester	**LANGUAGE**	Spanish	
COORDINATORS/ES		**EMAIL**		
CABEZA DE MARCO JAVIER ANGEL		jac@uniovi.es		
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2. Context

Experimentation in Inorganic Chemistry II (*EIC-II*) is a course integrated within the general subject *Inorganic Chemistry* of the *Fundamental Module* of the Bachelor's Degree in Chemistry offered by the University of Oviedo. It is an experimental course that is taught in the second semester of the fourth academic year.

The contents of this experimental subject are closely related to the syllabus of the theoretical subject *Chemistry of the Transition Elements*. The student knowledge on bonding, structure, synthesis, reactivity and characterization aspects associated with compounds and materials derived from transition metals will be experimentally consolidated in *EIC-II*, interrelating theoretical an experimental approaches.

EIC-II will be fully taught in English by Prof. Dr. Javier A. Cabeza de Marco and Dr. Pablo García Álvarez, and in Spanish by Prof. Dr. Marilín Vivanco Fernández, Prof. Dr. Victorio Cadierno Menéndez, Prof. Dr. Pascale Crochet, and Prof. Dr. Sergio E. García Garrido, all of them belonging to the Department of Organic and Inorganic Chemistry of the University of Oviedo.

3. Requirements

To take this course, it is mandatory for the student to have passed the courses (a) *General Chemistry,* (b) *Laboratory Operations and Information Technology Tools* (both within the basic module *General Chemistry*) and (c) *Chemistry of the Transition Elements.*

It is highly recommended to have previously taken the third-year subject *Experimentation in Inorganic Chemistry I* (*EIC-I*) as well as all other experimental subjects of the second and third years of the degree, since the students of *EIC-II* will make use of experimental skills that should have been previously acquired in these courses.

4. Competencies and learning results

The students that satisfactorily pass *EIC-II* are expected to:

General competences

- Acquire synthetic and analytical abilities (CG-1).
- Solve problems in a effective way (CG-2).
- Acquire planning and organizing abilities (CG-4).
- Make decisions (CG-5).
- Handle information with efficiency (CG-6).
- Speak and write correctly in English (CG-8)
- Acquire self-learning abilities (CG-9).

- Acquire sensibility towards environmental issues (CG-12).
- Acquire critical thinking (CG-17).
- Acquire group-working abilities (CG-18).

Specific knowledge competences

- Handle with confidence the chemical nomenclature, conventions and units (CE-1).
- Recognize the periodical properties and trends of the chemical elements (CE-3).
- Describe the main types of chemical reactions and their properties (CE-5).
- Apply the principles and procedures of chemical analysis to characterize inorganic compounds (CE-6).
- Understand the kinetics of the chemical change, including reaction mechanisms (CE-8).

Specific skills competences

- Evaluate, interpret and prepare chemical information (CE-22).
- Handle reagents, instruments and chemical devices in a safe way (CE-25).
- Perform standard synthetic and analytical laboratory procedures (CE-26).
- Acquire the appropriate information to observe, measure and monitorize chemical properties, events or changes (CE-27).
- Use standard instrumentation to identify, quantify, isolate and structurally characterize coordination and organometallic compounds (CE-29).
- Interpret experimental observations and measurements on the basis of their theoretical fundations (CE-30).
- Evaluate the risks associated with the use chemical products and laboratory procedures (CE-31).
- Use the inductive and deductive methods in the domain of Chemistry (CE-32).

Learning outcomes

- Know and understand the chemical properties of the elements and of their most important derivatives.
- Plan the own experimental work and the associated data collection.
- Handle original bibliographic sources.
- Choose appropriate laboratory techniques and experimental conditions to prepare and purify inorganic products (inert atmospheres, high and low reaction temperatures, chromatographic separations, etc.).
- Use the appropriate analytical techniques and interpret their results to characterize inorganic compounds (IR, RMN (1H and 31P), V-UV, magnetism, electric conductivity, thermal analysis, powder X-ray diffraction).
- Identify and evaluate the safety issues associated to the use of chemicals and laboratory procedures. Perform the experiments in a safe way.

- Perform the different laboratory procedures with scientific rigor.
- Work in the laboratory being well-organized and tidy.
- Prepare a report (oral and written) using the adequate chemical language.

5. Contents

EIC-II includes the synthesis of coordination complexes, organometallic complexes and inorganic solid. Some of these preparations require the use vacuum and inert atmosphere techniques. The student will familiarize with solution and solid-state synthetic methods as well as with purification methods (recrystallization, chromatography). The use of instrumental analytical techniques for compound characterization is a key part of this subject. The contents of this course have been grouped in four units:

I. Introduction

Overall organization and contents of the course. Identification of materials and equipment (synthetic and analytical). Safety rules in the laboratory and waste management. Safe procedures and laboratory practices.

II. Synthesis and characterization of coordination complexes

Experiment 1. Synthesis of [Co(H2O)(NH3)5]Cl3, [CoCl(NH3)5]Cl2, [Co(k*O*-NO2)(NH3)5]Cl2 and [Co(k*N*-NO2)(NH3)5]Cl2. Characterization by their electrical conductivity in solution as well as by their IR and V-UV spectra.

References: *Experimental Methods in Inorganic Chemistry*, J. Tanaka, S. L. Suib; Prentice Hall, 1999. *Inorganic Experiments*, 3rd ed., J. Derek Woolins; Wiley-VCH, 2010. G. M. Williams, J. Olmsted III, A. P. Breksa III, *J. Chem. Ed.* **1989**, *66*, 1043.

Experiment 2. Synthesis of two nickel(II) complexes having different structures: [Ni(NCS)2(PPh3)2] and [NiCl2(PPh3)2]. Characterization using magnetic susceptibility measurements and IR spectroscopy.

Reference: *Inorganic Experiments*, 3rd ed., J. Derek Woolins; Wiley-VCH, 2010.

Experiment 3. Synthesis of the binuclear complex [Cr2(m-O2CCH3)4(H2O)2] and determination of its magnetic susceptibility.

References: *The Synthesis and Characterization of Inorganic Compounds*, W. L. Jolly; Prentice Hall, 1970, p 442. *Practical Inorganic Chemistry. Preparations*, *Reactions and Instrumental Methods*, G. Pass, H. Sutcliffe; Chapman and Hall, 1974.

III. Synthesis and characterization of organometallic complexes

Experiment 4. Stepwise synthesis of the ylidic silver complex [Ag{CH(PPh3)C(O)CH3}2][NO3] from triphenylphosphane. The use of solution electrical conductivity measurements as well as IR and RMN (1H y 31P{1H}) spectroscopies to characterize it and its intermediate products.

Reference: J. Vicente, M. T. Chicote, I. Saura-Llamas, *J. Chem. Ed.* **1993**, *70*, 163.

Experiment 5. Synthesis of the manganese(I) complexes *fac*-[MnBr(CO)3(k2*P*,*P*-dppm)] and *cis,mer*-[MnBr(CO)2(k2*P*,*P*-dppm){P(OPh)3}]. Characterization using solution IR as well as 1H and 31P{1H} NMR spectroscopies.

Reference: G. A. Carriedo, *J. Chem. Ed.* **1988**, *65*, 1020.

Experiment 6. Synthesis of [Fe(h5-C5H5)(h5-C5H4COCH3)] by acetylation of ferrocene. Purification by column chromatography and characterization by IR and 1H NMR spectroscopies.

References: *Técnica y Síntesis en Química Inorgánica*, R. J. Angelici; Reverté, 1979. *Microscale Inorganic Chemistry: A Comprehensive Laboratory Experience*, Z. Szafran, R. M. Pike, M. M. Singh; John Wiley, 1991.

IV. Synthesis and characterization of solids

Experiment 7. Synthesis of the hydrated group 2 metal oxalates MC2O4·nH2O (M = Ca, Sr, Ba). Characterization by thermogravimetric analysis.

Reference: *Microscale Inorganic Chemistry. A Comprehensive Laboratory Experience*, Z. Szafran, R. M. Pike, M. M. Singh; John Wiley, 1991.

Experiment 8. Synthesis of the mixed oxide CaMnO3. Characterization by powder X-ray diffraction.

Reference: *Inorganic Experiments*, 3rd ed., J. Derek Woolins; Wiley-VCH, 2010.

6. Methodology and working plan

In the first session, the instructor will comment on the main features associated with the contents of the subject, will identify and describe the glassware and the equipment to be used, and will highlight key aspects of laboratory safety rules and potential hazards associated with the toxicity and handling of chemicals.

Each student will carry out the experiments individually, which will be performed according to the following procedural guidelines:

1. Prior to each laboratory session, the student should have read and understood the experiment guide and should master the appropriate theoretical background.

2. The instructor will discuss the theoretical background of the experiment, the practical procedure and all safety precautions that should be taken.

3. The student should balance all chemical equations and precisely calculate molar amounts and proportions of the used reagents.

4. The student should assemble the reaction equipment when necessary.

5. The student should carry out the experiment following its practical guide and calculate the yields of all reaction products.

6. The student should perform the appropriate analytical experiments and/or should collect the data they provide in order to characterize the products.

7. The student should analyze all of the experimental observations and analytical data.

8. During and after each experiment, the student should make sure that all used materials, equipment and working area is clean and well organized.

9. A group meeting will be carried out after each experiment to discuss the experimental results and their associated theoretical basis.

All experiments will be carried out under the supervision of the laboratory instructor, who will give additional explanations to individual students or to small groups when necessary, and will take care of the proper implementation of the procedures and the safety rules. Furthermore, the instructor will frequently ask the students questions related to the experiment in order to check their level of understanding and capacity to adequately perform it.

Each student must fill in a notebook where he/she will describe the experimental procedures, report a detailed description and interpretation of the obtained results, comment on the appropriate analytical data, and also provide answers to any question formulated by the instructor.

Hourly assignment of the student work:

		ON-SITE WORK				**OUT-SIDE WORK**		
Unit	***Total hours***	*Seminars*	*Laboratory Experiments*	*Evaluation sesions*	***Total***	*Group work*	*Individual work*	**Total**
Unit I		1			1			
Unit II		1	22		23		42	42

Unit III		2	22		24		38	38
Unit IV		1	8		9		10	10
Total	**150**	5	52	3	**60**		90	**90**

WORK TYPE		**Hours**	**%**	**Total %**
On-site	Seminars	5	3.3	**40**
	Laboratory experiments	52	34.7	
	Evaluation sessions	3	2	
Out-site	Group work			**60**
	Individual work	90	60	
	Total	**150**		

7. Evaluation of the student's learning results

Attendance to all laboratory sessions is mandatory. However, in particular cases, which should be documentary justified, the student will be allowed to skip a small number of laboratory hours or sessions.

In the ordinary call, the final mark will arise from a summation of three components:

(a) His/her work in the laboratory (35 %)

- Preliminary preparation of the experiments, discussions between instructor and student, participative attitude of the student.
- Compliance of security rules and correct disposal of residues. Order and cleanness during the experiment execution.
- Student skills performing the experiments. Quality of the experimental results.
- Appropriate use of analytical techniques and correct interpretation of their results.

(b) His/her laboratory notebook (15%)

After completing the laboratory sessions, the student will hand in his/her laboratory notebook. Its evaluation will take into account:

- The correctness of the experiment descriptions, which will be accompanied by balanced chemical equations and all of the necessary calculations.
- The clarity and adequate organization of its contents.
- The discussion of the experimental results.
- The interpretation of the analytical results.
- The ability to structure and systematize.
- The appropriate use of bibliographical resources.

(c) A written test (50 %)

It will be scheduled at the end of the semester. It will consist of questions and exercises that will allow an evaluation of the student knowledge about the theoretical foundations and the preparative and analytical aspects of the syllabus experiments.

To pass this course in the ordinary call, the student should get marks of at least of 4 out of 10 in the laboratory work (lab work and notebook) and in the written test, and a final mark of at least 5 out of 10.

In the extraordinary call, the student will carry out (a) a hands-on laboratory test (closely related to one of the syllabus experiments) and will hand out a written report on it, and (b) a written test (analogous to that of the ordinary call). Both tests will be evaluated with the same criteria as those of the ordinary call. Each of these tests will contribute 50% to the final mark. To pass the subject in this call, the student should get marks of at least 4 out of 10 in the experimental (+ report) and written tests and a final mark of at least 5 out 10.

8. Resources, bibliography and complementary documentation

Resources

The laboratory is equipped with the appropriate materials and instrumentation to carry out the programmed experiments. The laboratory materials include glassware, tubing, heating stirrers, heating mantles, balances, Dewar glasses, laboratory ovens, muffle furnace, vacuum-inert gas lines and fume cupboards. The laboratory is also equipped with ice, liquid nitrogen an inert gas (nitrogen) installations. The on-site instrumentation includes a melting point determination system, magnetic balances (2), IR (2) and UV-V (2) spectrophotometers and a solution conductivity device. Additional analytical equipment is accessed through the University of Oviedo Scientific-Technical Services: NMR spectrometers, thermogravimetric analysis and powder X-ray diffraction.

Bibliography and complementary documentation

The student will be provided, through the University of Oviedo Virtual Campus, with a manual that contains a detailed guide of each experiment, security rules and information on chemical toxicity.

In addition to the specific bibliographic data associated to each experiment (see above in the syllabus section), the following complementary bibliography is also recommended:

- *Seguridad en el Laboratorio de Química*, F. J. García Alonso; Universidad de Oviedo, 2011.

- *CRC Handbook of Chemistry and Physics*, 91st ed., D. R. Lide, W. M. M. Haynes (Ed.); CRC Press, 2011.

- *Infrared and Raman Spectra of Inorganic and Coordination Compounds*; 6th ed., K. Nakamoto; Wiley, 2009.

- *Practical Inorganic Chemistry*, G. Marr, B. W. Rockett; van Nostrand Reinhold Co., 1972.

- *Inorganic Experiments*; 3rd ed., J. Derek Woolins; Wiley-VCH, 2010.

- *Integrated Approach to Coordination Chemistry*, R. A. Marusak, K. Doan, S. D. Cummings; Wiley-Interscience, 2007.

- *Experimental Methods in Inorganic Chemistry*, J. Tanaka, S. L. Suib; Prentice Hall, 1999.

- *Microscale Inorganic Chemistry. A Comprehensive Laboratory Experience*, Z. Szafran, R. M. Pike, M. M. Singh; John Wiley, 1991.

- *The Synthesis and Characterization of Inorganic Compounds*, W. L. Jolly; Prentice Hall, 1970.

- *Practical Inorganic Chemistry. Preparations*, *Reactions and Instrumental Methods*, G. Pass, H. Sutcliffe; Chapman and Hall, 1974.

- *Técnica y Síntesis en Química Inorgánica*, R. J. Angelici; Reverté, 1979.

The following textbooks can also be useful:

- *Chemistry of the Elements*, 2nd ed., N. N. Greenwood, A. Earnshaw; Elsevier, 1998,

- *Advanced Inorganic Chemistr*y, 6th ed., F. A. Cotton, G. Wilkinson, C. A. Murillo, M. Bochmann; Wiley, 1999.

- *Inorganic Chemistry*, 34th ed., A. F. Holleman, E. Wiberg; Academic Press, 2001.