Bachelor´s Degree in Chemistry

Subject Guide

1. Information about the subject

SUBJECT	Experimentation in Inorganic Chemistry I	**CODE**	GQUIMI01-3-004
EDUCATIONAL OFFER	Bachelor´s Degree in Chemistry	**CENTER**	Facultad de Química
TYPE	Compulsory	**N° TOTAL CREDITS**	6.0
PERIOD	First Semester	**LANGUAGE**	Spanish
COORDINATORS/ES		**EMAIL**	
CARRIEDO ULE GABINO ALEJANDRO		gac@uniovi.es	
LECTURERS		**EMAIL**	
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2. Context

The course Experimentation in Inorganic Chemistry I (EIC1) is taught in the first semester, third year of the Degree in Chemistry, and is integrated in the fundamental module within the Inorganic Chemistry subject. It is an experimental course, consequently, the laboratory skills that had been acquired in the previous experimental course *Basic Laboratory Operations* will be very useful.

The course EIC1 is closely related to the 2º year courses *Concepts and Models in Inorganic Chemistry* and, especially, with *Chemistry of the Representative Elements*. Knowledge about concepts on structure and bonding, properties and chemical reactivity of inorganic compounds

already studied in those subjects, will be consolidated with the preparation and characterization of some of these compounds, which will contribute to the necessary interrelation and equilibrium between the theoretical aspects and the experimental reality.

This subject, in turn, serve as support at varying degrees to subjects that will be studied later in the Degree, especially those related to Inorganic Chemistry, both from a theoretical approach (*Chemistry of Transition Elements*) and also from an experimental one (*Experimentation in Inorganic Chemistry II*).

Teaching Activity	Professor
PA1 (spanish)	Gabino A. Carriedo Ule Marilin Vivanco Fernández Mª Ángeles Villa García Mª Ángeles Álvarez Fidalgo
PA (english)	Mª Ángeles Villa García
PL1 (spanish)	Mª Ángeles Álvarez Fidalgo, Gabino A.Carriedo Ule
PL2 (spanish) PL4 (spanish)	Marilin Vivanco Fernández Mª Ángeles Villa García
PL3 (spanish)	Gabino A. Carriedo Ule
PL1 (english)	Mª Ángeles Villa García

3. Requirements

Like all subjects integrated in the Fundamental Module, EIC1 has as mandatory requirement that the students have passed the courses *General Chemistry* and *Basic Laboratory Operations and Information Technology Tools*, subjects taught in the first year of the Degree in Chemistry. But, it is also mandatory to have passed the subject *Chemistry of the Representative Elements*, which is taught in the second year of the Degree in Chemistry. Also, it is highly recommended to have studied the subject *Concepts and Models in Inorganic Chemistry*.

4. Competencies and learning results

General Competences:

* Show ability for analysis and synthesis (CG-1)

* Solve problems in an effective way (CG-2).

* Demonstrate planning and organization skills (CG-4)

* Ability to make decisions (CG-5)

* Ability to speak and write correctly in English (CG-8).

* Environmental awareness (CG-12)

* Develop critical thinking (CG-17).

* Develop group-working abilities (CG-18).

Specific competences

*Ability to apply chemical analysis principles and procedures for determination, identification and characterization of inorganic compounds (CE-6)

*Safe manipulation of reagents, equipments and chemical devices (CE-25)

*Ability to carry out standard procedures of synthesis (CE-26)

*Use standard instrumentation for the identification and structural characterization of inorganic compounds (CE-29)

*Correct interpretation of the observations and the experimental data obtained in the laboratory in terms of their meaning and theoretical aspects (CE-30)

*Evaluate the risks involved in using chemical substances and laboratory procedures (CE-31)

* Correctly use the inductive and deductive methods in the context of Chemistry (CE-32)

These skills should be translated into the following learning outcomes:

Learning outcomes

* Knowledge of inorganic substances (elements and compounds) and safe manipulation of them.

* Make the right choice and correctly use of the appropriate material for each laboratory experiment.

*Assembling the suitable equipment to carry out reactions, including those used in gases generation and handling.

* Carry out reactions of synthesis and transformation of elements and inorganic compounds under different conditions.

* Purification of inorganic compounds.

* Perform reactions under inert atmosphere.

* Use some instrumental techniques for the characterization of inorganic substances (melting point determination, magnetic balance, conductivity meter, Infrared spectrometer and Vis-UV spectrophotometer.

* Work in an orderly and cleanliness way in the laboratory.

* Carry out experiments with scientific rigor.

* Planning of the experimental work and adequate data collection of the results obtained.

* Correct and critical interpretation of the observations and the experimental data.

* Knowledge of the characteristics of the residues generated in the experiments and their adequate disposal.

* Prepare a laboratory notebook to register and describe the experiments using a clear and correct chemical language.

5. Contents

The experiments that will be performed in this course include the synthesis of inorganic compounds such as: halides, oxides, oxoacids, oxosalts and coordination compounds, with prevalence of those of the representative elements. Different procedures of synthesis and several characterization techniques will be employed. In many cases, the isolated product is going to be used as reagent for the synthesis of new ones, which requires that the compound has to be prepared with a high yield and purity. Experiments based on the reactivity of some elements and, in the case of sulfur its allotropic transformations, will also be carried out. Furthermore, different qualitative assays implying proton transfer reactions, redox, complex formation or precipitation will be performed. The contents of the course can be grouped into the following modules:

I. Introduction

Introduction to the course. Description of the laboratory material. Introductory notions about safety rules in the laboratory and chemical waste management.

II. Synthesis of inorganic compounds from an element

Preparation of lead(II) salts from lead. Synthesis of lead(II) nitrate by reaction between lead and nitric acid. Preparation of lead(II) chloride. Precipitation of some insoluble lead(II) compounds. Transformation of lead residues in lead(II) sulfate .

Preparation of some copper(II) compounds. Synthesis of copper(II) sulfate pentahydrate from Cu. Preparation of tetraamminecopper(II) sulfate and ammonia and copper(II) double sulfate. Thermal decomposition of the compounds prepared. Conductivity measurements carried out in aqueous media. Variation of the oxidizing character of Cu(II) with the ligands.

Preparation of Sn(IV) compounds. Synthesis of tin(IV) chloride from reaction of metallic tin with chlorine. Synthesis of ammonium hexachloro-stannate(IV). Characterization by IR spectroscopy.

Preparation of zinc dithionite. Zinc reaction with sulfur dioxide. The dithionite anion as a reducing agent.

Preparation of sodium thiosulfate pentahydrate. Sulfur reaction with sodium sulfite in heterogeneous phase and aqueous media. Characterization by IR spectroscopy. Reactivity of the thiosulfate anion.

Preparation of double sulfate of aluminum and ammonium dodecahydrated. Starting from aluminum is obtained a solution of aluminum and ammonium sulfate that crystallizes.

III. Synthesis of inorganic compounds from other compounds

Preparation of potassium tris(oxalato)ferrate(III) trihydrate. Build the stereochemical model of the complex. Measure the magnetic susceptibility. Measure the Specific conductivity. Characterization by IR spectroscopy. Thermal and photochemical stability.

Preparation of K3[Cr(C2O4)3]·3H2O and the cis and trans- [Cr(C2O4)2(H2O)2]·nH2O isomers. Reduction of potassium chromate with oxalates under different experimental conditions. Characterization of the products obtained by measuring their specific conductivity and also

by UV-Vis spectroscopy.

Preparation of copper(I) chloride. Reduction of Cu(II) in the presence of chlorides. CuCl insulation under a nitrogen atmosphere by using a gas/vacuum line. Reactivity of CuCl.

Silica Gel Formation. Protolysis of aqueous sodium silicate. Formation of silica gel with Co(II) adsorbed on the lattice (pink). Formation of a silica xerogel with Co(II) adsorbed on the lattice (blue).

IV. Reactions in non aqueous media

Polymorphic and allotropic transformations of sulfur. a, b, g-sulfur and polysulfur (plastic sulfur).

Preparation of sodium solutions in liquid ammonia. Preparation of liquid ammonia. Preparation of sodium-ammonia solutions. Reduction of ammoniun chloride.

Preparation of the mixed oxide Al2CoO4. Synthesis of the mixed oxide Al2CoO4 by calcination of a gel of coprecipitated aluminum and cobalt hydroxides. Characterization by measuring the magnetic moment.

6. Methodology and working plan

In first place, the professor will present the main ideas concerning the contents of the subject, describing: the Virtual Campus contents; the mandatory general rules to follow in the laboratory during the performance of the experiments; the glass ware and equipment that will be used in the laboratory; the questions related to laboratory safety rules; toxicity and potential hazards associated with chemicals handling; and the procedures to follow for waste disposal. Then, the programmed experiments will be carried out (not necessarily following the order shown in this syllabus), most of them by each individual student, some of them as a team with two students and, occasionally, by more than two students in each group.

The essential contents of the experiments are described in the *Manual* of the course, prepared by the teaching staff that will be available for the students as a pdf document in the Virtual Campus.

All experiments will be performed according to the following general guidelines:

1. Before each laboratory practice, the student must read carefully the description of the experiment and have reviewed the theoretical background.
2. The laboratory instructor will explain the theoretical basis of the experiment, the assembly of the equipment (when necessary), the experimental procedures, the necessary instrumental techniques, the safety precautions that must be taken into account and the waste treatment.
3. The student has to calculate the stoichiometric amounts of the reagents used to check the amounts of them that will be used according to the procedures (recipes).
4. Preparation, when necessary, the experimental equipment.
5. Implementation of the experiment.
6. Interpretation of observations and experimental data.
7. Show to the professor the products and the data. Group discussion of the results, the professor will clarify any doubts and questions arising from the experiment.
8. Management of the waste generated in the experiment.
9. Cleaning of the material, the working place and all the laboratory areas used.

All experiments will be carried out under the laboratory instructor´s monitoring, he will give additional explanations to each individual student or to small groups when necessary, and will take care of the proper implementation of the safety rules. Furthermore, the instructor will ask the students about different questions related to the experiment, in order to check out their level of understanding and ability to perform the experiment.

The students must have a laboratory notebook, where they will describe the experimental procedures performed, the observations, the data obtained and their interpretation, as well as the answers to the questions formulated.

	ON-SITE WORK	**PERSONAL WORK**

Topic	***Total hours***	*Seminars*	*Llaboratory experiments*	*Evaluation sessions*	***Total***	*Group work*	*Individual* work	**Total**
Module I			3					
ModuleII		3	24				25	
Module III		2	27				30	
Module IV		1	12				20	
Total	**150**	**6**	**66**	**3**	**75**		**75**	**75**

TYPES		Hours	%	Total
On site	Expositive sessions			75

	Seminars	6	8	
	Laboratory experiments	66	88	
	Clinical Practices			
	Mentored gorup sessions			
	External activities			
	Exams	3	4	
Personal work	Group work			75
	Individual work	75	100	
	Total	150		

7. Evaluation of the student's learning results

Attendance is mandatory, it is necessary to attend all scheduled laboratory sessions in order to be evaluated.

Ordinary call exam

Final evaluation will be done according to the following criteria:

Topic	Criteria	Method	Grading

			scheme
Laboratory work	• Active participation of the student. • Fulfillment of the implemented measures on security and waste management. • Safe working environment keeping the lab ordered and clean, both in individual and group work. • Experimental skills. • Answers to the questions raised by the professor.	Monitoring by the professor	15%
Individual fulfillment of an experiment closely related to the syllabus	• Balancing chemical reactions, stoichimetric calculus and yields. • Organization and planning of the experiment • Assembling experimental equipments, carry out experimental procedures for the synthesis of pure products. • Correct use of the techniques employed for characterization of the synthesized products. • Correct Interpretation of the results.	Hands-on Lab test	45%

Level of knowledge of the theoretical concepts and of the experimental methods acquired during the performance of the experiments	• Mastery of the subject	Written test	40%

The hands-on Lab test will consist in performing an experiment closely related with one of those of the Syllabus and will be held in the last laboratory session, the written exam, which is common for all students registered in the subject, will be held on the date designed by the Faculty according to the calendar of examinations.

To pass the course, it is mandatory that the student gets a minimum mark of 4 points over 10 in both hands-on Lab test and in the written test. The final mark, weighted sum of the different aspects evaluated, must be equal or higher than 5 points over 10.

Extraordinary call examinations

The student will carry out a hands-on Lab test (an experiment closely related with one of those of the Syllabus) and a written test on the matters related with all experiments of the programme. Each of both tests will contribute to the final mark with 50%, which must be equal or higher than 5 point over 10. It will be necessary to obtain a minimum mark of 4 over 10 in both tests.

8. Resources, bibliography and complementary documentation

Resources

The laboratory where the student will perform the programmed experiments has the material and equipment necessary to adequately carry out them. So, besides glass ware, it is fitted with heating stirrers, heating mantles, balances, low temperature thermometers, Dewar glasses, laboratory ovens, muffle furnaces and vacuum-nitrogen lines. It also has the necessary equipment to obtain the experimental data necessary

to characterize the inorganic compounds, such as infrared spectrophotometer, apparatus to measure melting points, magnetic balance, conductivity meter and Vis-UV spectrophotometer. The laboratory is also fitted with city gas installation (for Bünsen burners) and gas and liquid nitrogen.

Bibliography and complementary documentation

A laboratory Manual will be available to the students (from the Virtual Campus) in which are included general considerations on the laboratory work, security rules and a detailed handout of each of the experiments to perform. It will also be available (from the Virtual Campus) a document with basic information about the toxicity of the chemical products and the identification tags. Information about the theoretical bases of the procedures and experimental techniques used in this course is available in the book: **Fundamentos de Química Inorgánica Experimental**. G. A. Carriedo. Ediciones de la Universidad de Oviedo. 2016.

In this book are cited references of books about Experiments in Inorganic Chemistry that were used to describe the experiments of the course, such as:

Química Inorgánica Preparativa. G. Brauer. Reverté 1958.

Practical Inorganic Chemistry", G. Marr, B. W. Rockett, Van Nostrand Reinhold Co., 1972.

Any data related to the substances and reagents used in the experiments can be obtained from the book

"CRC Handbook of Chemistry and Physics", D. R. Lide, W. M. M. Haynes (Editores), 91ª edición, 2011.

Additional descriptive information about the elements or the inorganic compounds can be obtained from the books:

"Chemistry of the Elements", N. N. Greenwood, A. Earnshaw, Butterworth Heinemann, 2ª edición, 1997.

"Inorganic Chemistry", Holleman-Wiberg. Academic Press. Berlín. 2001.