

**COURSE DATA****Data Subject**

<b>Code</b>	42932
<b>Name</b>	Techniques for the study of crystalline solids
<b>Cycle</b>	Master's degree
<b>ECTS Credits</b>	2.0
<b>Academic year</b>	2023 - 2024

**Study (s)**

<b>Degree</b>	<b>Center</b>	<b>Acad. year</b>	<b>Period</b>
2109 - M.D. in Experimental Techniques in Chemistry	Faculty of Chemistry	1	First term

**Subject-matter**

<b>Degree</b>	<b>Subject-matter</b>	<b>Character</b>
2109 - M.D. in Experimental Techniques in Chemistry	1 - Advanced laboratory of experimental techniques in chemistry	Obligatory

**Coordination**

<b>Name</b>	<b>Department</b>
ROS LIS, JOSE VICENTE	320 - Inorganic Chemistry

**SUMMARY**

Laboratory Subject dedicated to the learning of advanced work methodologies used in the techniques used in the study of crystalline solids, such as electron microscopy, or the powder diffraction of x-rays.

**PREVIOUS KNOWLEDGE****Relationship to other subjects of the same degree**

There are no specified enrollment restrictions with other subjects of the curriculum.

**Other requirements**

Prior knowledge of chemistry and experimental work in the laboratory of chemistry taught in the degrees indicated in the recommended income profile for the student of the master's degree are required.

**OUTCOMES****2109 - M.D. in Experimental Techniques in Chemistry**

- To acquire basic skills to develop laboratory work in biomedical research.
- Be able to access the information required (databases, scientific articles, etc.) and to interpret and use it sensibly.
- Ser capaces de seleccionar y optimizar las variables instrumentales para obtener los mejores parámetros analíticos en las técnicas experimentales estudiadas.
- Ser capaces de emplear las herramientas básicas para el tratamiento de datos experimentales en el laboratorio.
- Realizar estudios relacionados con el análisis y/o la caracterización de sustancias químicas tales como: control de calidad, diseño de protocolos de trabajo para laboratorios, diseño e implementación de procesos de acreditación y validación, diseño y desarrollo de proyectos I+D+I, emisión de informes, certificaciones y/o dictámenes, etc.
- Ser capaces de planificar y gestionar los recursos disponibles de un laboratorio químico, teniendo en cuenta los principios básicos de la calidad, prevención de riesgos, seguridad y sostenibilidad.
- Seleccionar la instrumentación química comercializada apropiada para el estudio a realizar y de aplicar sus conocimientos para utilizarla de manera correcta.
- To prepare a clear and concise memory of the results of your work and the conclusions obtained.

**LEARNING OUTCOMES**

At the end of the teaching-learning process, the student should be able to:

1. Describe the basic principles of the techniques of electron microscopy in the characterization of solids.
2. Prepare solid samples for its study using the techniques of electron microscopy.
3. Optimize the acquisition of images using the techniques of electron microscopy.
4. Describe the fundamental principles of X-ray powder diffraction
5. Design experiments and prepare the samples studied by powder diffraction of x-rays.
6. Simulate powder diffraction patterns of crystalline phases of the different crystalline systems.
7. Identify the different crystalline phases present in a material.
8. Semiquantitatively determination of a mixture of crystalline phases in a material.
9. Regarding to the Sustainable Development Goals (SDGs) in this subject it is expected that students will be able to apply the knowledge learned to help guarantee food quality and safety (SDG 2), acquire a special sensitivity for sustainable management of raw materials and energy sources (SDG 7) as well as for sustainable development compatible with the environment (SDGs 11, 12, 13, 14,



and 15), all while promoting innovation and improvement in processes and products (SDG 9).

## DESCRIPTION OF CONTENTS

### 1. Electronic microscopy

- Sample preparation from powder materials (inorganic products, minerals, ceramic materials) for scanning electron microscopy (SEM).
- Observation and microstructural characterization through MEB.
- Optimization of the control parameters of the MEB equipment for obtaining high resolution images.

### 2. X-ray diffraction (XRD)

#### Powder Diffraction of X-rays (XRD)

- Sample preparation from powder materials (inorganic products, minerals, ceramics) and materials in piece (ceramics, metals and alloys) for consideration by XRD.
- Establishment of the optimum parameters for obtaining diffraction standards of crystalline samples.
- Obtaining the diffraction patterns of the samples prepared.
- Identification of crystalline phases in single-phase samples.
- Identification of crystalline phases in multiphasic samples.
- Determination of crystalline phases in a material.

## WORKLOAD

ACTIVITY	Hours	% To be attended
Laboratory practices	20,00	100
Development of group work	5,00	0
Study and independent work	12,00	0
Readings supplementary material	4,00	0
Preparation of evaluation activities	4,00	0
Resolution of case studies	5,00	0
<b>TOTAL</b>	<b>50,00</b>	

## TEACHING METHODOLOGY

### Presential Activities



Laboratory classes will begin with seminars in which Professor will perform a brief introduction of the objective, fundamentals and experimental practices methodology to perform.

The teacher will held in the laboratory the necessary explanations on operation of the instruments to be used in each practice prior to their use by students and will supervise its use during practices, to enhance knowledge on the techniques used.

Students will carry out the practice following the corresponding protocols or manual of practices.

Classroom activities performed in the laboratory, presentations and exhibitions of works will be part of the ongoing evaluation of the student (formative activities AF2 of verifica and teaching methodology MD1 of verifica)

Written examinations of the subject will be carried out on the date specified in the programming of the assessment tests (formative activities AF4 of verifica and teaching methdology MD1 of verifica).

The competences to acquire from the presential activities will be:

- Generals: CG1 and CG3
- Specific: CE2, CE3, CE4, CE5 y CE6

### **Non-presential activities**

Students will conduct the non-presential activities requested by the teacher (memoirs, reports of practices, etc.) and they will deliver them on the specified date.

The competences to acquire from the presential activities will be:

- Specific: CE7

## **EVALUATION**

1. -Continuous evaluation of the student in classes and seminars (participatory assistance, material handling and equipment, organization of work, understanding and use of the screenplay of practices, performing calculations, team work and presentation of results of the activities carried out in the laboratory)

Along the sessions, focus in the resolution of practical assays, the assistance and participation of the students will be evaluated individually (by oral answers or by writing questions planned by the professor, by planning questions which its answer will be relevant for all the group). Also, these questions will include the design of working protocols, the selection of variables and the tools for the data treatment (verifica competences CE2, CE3, CE5 and CE6). The competences to evaluate: specifics: CE2, CE3, CE4, CE5 and CE6



## **WEIGHT 40**

2.- An assessment of non-classroom-based activities (memories and/or reports of practices delivered which will consist of simulation exercises of dust diffraction patterns of industrial, mineral solids and synthesized in the laboratory)

The reports performed by the students will include the main conclusions extracted from the laboratory work (working protocols, variable selection and data treatment; verifica competences CE2, CE5, CE6 and CE7) and it will be done by couples to improve the group working (consensus decision making: verifica competences CG1 and CE7)

## **WEIGHT 30**

3. -Written examinations (Based on the results of learning the content and on the specific objectives of each subject)

The exam will consist in the resolution of questions and practical examples related to the studied techniques (verifica competences: CE2, CE4, CE5 and CE6).

## **WEIGHT 30**

The minimum grade obtained in each of evaluated parts must be equal to or greater than 4.5 in order to average between them.

The minimum overall grade to pass the course is 5.0.

## **REFERENCES**

### **Basic**

- Aballe M., J. López Ruiz, J.M. Badía y P. Adeva (eds.), Microscopía Electrónica de Barrido y Microanálisis por Rayos X, CSIC y Rueda, Madrid, 1996.
- Bermúdez J., Métodos de difracción de rayos X. Principios y aplicaciones, Pirámide, 1981.
- Goldstein J.I. (ed.), Scanning Electron Microscopy and X-Ray Microanalysis. A Text for Biologists, Materials Scientists, and Geologists, Plenum Press, 1981.
- Goodhew P.J. y F.J. Humphreys, Microscopy and Analysis, Taylor & Francis, 1988.
- Heinrich K.F.J., Electron Beam X-Ray Microanalysis, Wiley, New York, 1987.
- Klug H.P. y L.E. Alexander, X-Ray Diffraction Procedures for Polycrystalline and Amorphous Materials, Wiley, 1974.





- Wormald J., Métodos de difracción, Reverté, Barcelona, 1981.

