

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

Code	44996
Name	Caracterización de sólidos inorgánicos
Cycle	Master's degree
ECTS Credits	5.0
Academic year	2022 - 2023

Study (s)

Degree	Center	Acad. Period
2249 - Master's Degree in Chemistry	Faculty of Chemistry	1 First term

Subject-matter

Degree	Subject-matter	Character
2249 - Master's Degree in Chemistry	3 - Aplicaciones de la Química Inorgánica	Obligatory

Coordination

Name	Department
MINGUEZ ESPALLARGAS, GUILLERMO	320 - Inorganic Chemistry

SUMMARY

The course "Characterization of Inorganic Solids" is taught in the first semester of M.U. in Chemistry and is part of the Applied Chemistry module. Its objective is that the students acquire the necessary training to determine and interpret the properties of any type of inorganic solid, as an essential step for its use in the scientific, technological or industrial field. Given that the duration of the subject, which will be provided with a practical part, does not allow to cover all the characterization techniques, those that are of more general application and that provide information on chemical composition, crystalline structure, morphology and thermal behavior of materials, have been selected. In all cases, the techniques under study will be approached from the basic principles strictly necessary to understand their operation. Thus, the equipment and the sample preparation methodology will be studied as soon as possible. Finally, a variety of practical cases will be raised that provide experience in data processing. Among the set of techniques that are studied are those of X-ray diffraction of polycrystalline samples. In this case and using the most advanced diffractometers available to UVEG, phase identification and structural analysis experiments will be carried out. For the morphological characterization, optical and electronic microscopy techniques will be used. The different scanning electron microscopy techniques will be described, both in the acquisition of images (SEM, TEM and HRTEM), and in the chemical characterization by backscattered electrons (SEM-EDX and TEM-EDX). Finally, thermal analysis



methods will also be studied, such as thermogravimetrics (TGA), differential thermal analysis (DTA) and differential scanning calorimetry, with emphasis on the handling of instrumentation and the interpretation of the results.

PREVIOUS KNOWLEDGE

Relationship to other subjects of the same degree

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

Other requirements

Chemistry knowledge acquired during the Chemistry or recommended entry degrees are required.

COMPETENCES (RD 1393/2007) // LEARNING OUTCOMES (RD 822/2021)

2249 - Master's Degree in Chemistry

- Students should apply acquired knowledge to solve problems in unfamiliar contexts within their field of study, including multidisciplinary scenarios.
- Students should be able to integrate knowledge and address the complexity of making informed judgments based on incomplete or limited information, including reflections on the social and ethical responsibilities associated with the application of their knowledge and judgments.
- Students should demonstrate self-directed learning skills for continued academic growth.
- Students should possess and understand foundational knowledge that enables original thinking and research in the field.
- Be able to solve complex chemistry problems, whether in the academic, research or industrial application areas at a specialization or masters-level.
- Possess the necessary skills to develop multidisciplinary activities within the field of chemistry at the master's level.
- Fomentar, en contextos académicos y profesionales del ámbito de la política económica, el avance tecnológico, social o cultural dentro de una sociedad basada en el conocimiento y en el respeto a: a) los derechos fundamentales y de igualdad de oportunidades entre hombres y mujeres, b) los principios de igualdad de oportunidades y accesibilidad universal de las personas con discapacidad y c) los valores propios de una cultura de paz y valores democrático.
- Possess the ability to plan and manage time and resources and gain experience in decision-making.
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- Gain experience in the use of information tools and in the management of the information obtained.
- Be able to defend positions in debates and colloquia in a rigorous and reasoned manner.



- Be able to design, conduct, analyse and interpret complex experiments and data, as a specialist.
- Gain skills and knowledge in different advanced characterisation techniques for selecting the most suitable techniques, according to chemical criteria, for the characterisation of inorganic solids at an industrial level.
- Apply the advanced theoretical and practical knowledge gained in the different specialties of chemistry to R&D and innovation.
- Be able to conduct any type of research in the field of chemistry and/or the chemical industry, as a specialist.
- Be able to present and defend publicly the results obtained in scientific research or as a result of work in a chemical industry.

LEARNING OUTCOMES (RD 1393/2007) // NO CONTENT (RD 822/2021)

- Know the basic fundamentals of X-ray diffraction techniques, electron microscopy and thermal analysis.
- To be able to select the characterization techniques that are most appropriate for each case.
- Know how to detail the experimental methodology, including the selection of experimental variables, interpret the records obtained in each of the techniques studied.
- Know the most relevant applications of each technique.
- Know the procedures and computer programs for the interpretation of results.
- Knowing how to apply the knowledge acquired to contribute to the Sustainable Development Goals (SDGs), such as the sustainable management of water, raw materials and energy sources (SDGs 6 and 7) and to develop a professional work with the least environmental impact and using alternative raw materials (SDGs 11, 14 and 15)

DESCRIPTION OF CONTENTS

1. X-ray diffraction

Basic concepts of Crystallography. X-ray diffraction. Single crystal diffraction vs powder diffraction. Instrumentation. Determination of lattice parameters. Indexing. Determination of the space group of symmetry. Determination of the crystal structure. Polymorphism. Identification and quantitative analysis of crystalline phases. Measurement of stress in a polycrystalline solid. Measurement of the texture in a polycrystalline solid. Measurement of the mean particle size of a polycrystalline solid. Use of database.

2. Electron microscopy

Microscopy fundamentals. Electromagnetic spectrum. Interaction of radiation with matter. Introduction to microscopy and resolution techniques.

Scanning electron microscopy (SEM). Electronic optics. Image formation and interpretation. Working modes and detectors. EM-EDX microanalysis. Qualitative analysis. Sample preparation for SEM Application to the characterization of various inorganic substances, such as ceramic materials.

Transmission electron microscopy (TEM) and high resolution (HRTEM). Parts of the microscope.



Imaging, diffraction patterns and aberration correction. Techniques: parallel beam, STEM. Analytical techniques for TEM: X-ray energy dispersion microanalysis (EDX) Electronic energy loss spectroscopy (EELS), High Angle Anular Dark Field (HAADF). Sample preparation for TEM.

3. Thermal analysis

Thermogravimetric methods (TG), differential thermal analysis (DTA), differential scanning calorimetry (DSC), dynamic mechanical analysis (DMA) and dilatometry: instrumentation and applications in the thermal characterization of inorganic samples and composites.

WORKLOAD

ACTIVITY	Hours	% To be attended
Theory classes	40,00	100
Tutorials	10,00	100
Study and independent work	75,00	0
TOTAL	125,00	

TEACHING METHODOLOGY

This course will be taught through participatory lecture-type theoretical classes, classes with directed practical activity, seminars and workshops. The practical classes, seminars and tutorials will focus on solving practical cases of interest to different industrial sectors, with special attention to the ceramic sector.

The practical classes will include laboratory sessions in which contact with the techniques described will be made, as well as sessions of analysis and exploitation of the data obtained from the different techniques for the practical assumptions raised. Use will be made of the Virtual Classroom platform, a virtual space where all the information deemed appropriate for the development of teaching and the control of student participation in the proposed activities is deposited.

Due to organizational reasons, during the 2022-2023 academic year, attendance has been reduced to 80%

EVALUATION

The qualification of the subject for both the first and the second call will be obtained from:

- Written exams: based on the learning results and the objectives of each subject, in its theoretical and / or practical part that will account for 60% of the final grade.
- The preparation and presentation of work by the students of questions raised by the teacher at the end of each topic will account for 20% of the final grade.
- The continuous evaluation of the activity developed by the student through participatory assistance, problem solving, etc ... will count for 20% of the final grade.



The grade necessary to pass the course is 5 points.

REFERENCES

Basic

- Bermúdez J., Métodos de difracción de rayos X. Principios y aplicaciones, Pirámide, 1981.
- 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.

Additional

- 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.; Humphreys, F. J., Microscopy and Analysis, Taylor & Francis, 1988.
- Heinrich, K. F. J., Electron Beam X-Ray Microanalysis, Wiley, New York, 1987.
- Klug, H. P.; Alexander, L. E., X-Ray Diffraction Procedures for Polycrystalline and Amorphous Materials, Wiley, 1974.
- Wormald, J., Métodos de difracción, Reverté, Barcelona, 1981.
- Kuo, J., Electron Microscopy Methods and Protocols. Springer Protocols, 2014.
- Brandon, D., Kaplan, W. D., Microstructural Characterization of Materials 2nd Edition Wiley Book, 2008.