60456 - Crystallography and diffraction techniques

Información del Plan Docente

Academic Year	2016/17
Academic center	100 - Facultad de Ciencias
Degree	543 - Master's in Molecular Chemistry and Homogeneous Catalysis
ECTS	2.0
Course	1
Period	Second semester
Subject Type	Optional
Module	---

1.Basic info

1.1.Recommendations to take this course

It is advisable to have basic knowledge of solid state chemistry and crystallography (point / spatial symmetry), although the course can be followed without this previous knowledge. In that case, some complementary readings at the beginning of the course will be suggested.

Class attendance, reading of suggested texts and the continuous work will facilitate passing the subject.

1.2.Activities and key dates for the course

Subject with a workload of 2ECTS that is imparted in the second semester.

The information about schedules, calendars and exams is available at the websites of the Sciences Faculty, https://ciencias.unizar.es/calendario-y-horarios , and the Master, http://masterqmch.unizar.es .

2.Initiation

2.1.Learning outcomes that define the subject

To have assimilated fundamental concepts of spatial symmetry, showing an adequate comprehension of the nomenclature of spatial groups theory and also of its application to Crystallography.

To critically handle theoretical concepts related to the diffraction process and, especially, the relationship between diffraction experiments and crystal internal structures.

To know the different experimental methods commonly used in the diffraction pattern measurements, both for powder and single crystal samples.

To understand the more appropriate single crystal X-ray data collection strategies ensuring an optimal structure resolution

at a molecular level.

To master the fundamental concepts about the phase problem solution and the experimental proceedings adequate for the structure solution from X-ray diffraction experiments.

To interpret main characteristics of direction and to evaluate the data quality.

To integrate X-ray diffraction data to obtain structural information, at crystal or molecular levels.

To interpret structural parameters, obtained from diffraction experiments, in the chemical context.

To know other structural determination processes based on diffraction experiments carried out with radiations different from X-rays.

2.2.Introduction

The course aims to initiate students into Structural Crystallography, a research field closely related to Chemistry, Material Science, Physics, Biology or Geology, in either academic or industrial contexts.

The subject will tackle, in a first theoretical part, the basic concepts of symmetry and the diffraction process, together with an adequate analysis of data collection strategies, crystal structure solution and refinement procedures. In a second part, mainly carried out as practical work, the most commonly used programs for data integration and result's representations will be discussed.

This course is focussed on molecular structure studies from single crystal samples. However, the most common techniques used in the characterization of powder samples and, in a lesser extent, to partially ordered species, will also be presented.

Diffraction-based structural characterization represents the most direct and precise mechanism to understand molecular structure, providing quantitative information for the characterization of molecular and intermolecular parameters of special relevance in Molecular Chemistry and Catalysis.

3.Context and competences

3.1.Goals

The course aims to train the students to recognize the potential application of diffraction techniques in their research field and to be able to follow from data collection and reduction processes to the extraction of the relevant structure information, at molecular or crystalline level.

The comprehension of fundamental concepts associated to diffraction techniques should contribute to the student academic training, providing them self-sufficient criteria to decide the proper application of diffraction techniques to specific chemical problems, in a research environment or in an R&D industrial context, either in production or in quality control departments.

3.2.Context and meaning of the subject in the degree

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Knowledge of crystallographic techniques is fundamental for definitive and precise structural characterization (at molecular or crystalline level) of new substances from solid state samples. The structural knowledge obtained from X-ray diffraction experiments is essential in the rationalization of reactive processes. Moreover, the observed intermolecular interactions can provide key information about the reaction operative mechanisms or about observed macroscopic physical properties.

Furthermore, diffraction techniques turn out to be very useful in the identification of crystalline phases of unknown materials (in pure samples or mixtures) or in the qualitative and quantitative analysis of solid samples. These applications make diffraction a really powerful tool in chemical industry, when dealing with substances in solid state.

It is noteworthy the extraordinary potential of diffraction techniques, either working with single crystals or polycrystalline samples, their complexity and the versatility of different available experimental options for the sample environment. Taking into account these particularities, this course is included in the "Structural Characterization" module, but as an independent optative course during second semester. Undoubtedly, it especially complements the compulsory "Structural Characterization Techniques" course.

This course is recommended to those students motivated for the study of new molecules with high molecular complexity or for those substances requiring the definitive determination of basic molecular parameters. Additionally, the course will provide basic concepts for the analysis of polycrystalline samples, and for the identification and/or quantification of crystalline phases.

3.3.Competences

To be able to design diffraction experiments to obtain relevant structural information (at molecular or crystal level), understanding the most suitable data collection equipment, in laboratory or in large-scale facilities (such as synchrotron sources).

Capacity to extract information about experimental data and perform data reduction processes.

To be able to carry out data treatment, to evaluate its quality and to represent the results, according to the research area under study.

Capacitiy to use the obtained structural information for understanding and interpretation of reactive processes in Molecular Chemistry and Catalysis and set them in the context of analogous or related results.

To be able to design, facing a problem involving a solid (crystalline) sample, the most appropriate experimental methodology to identify or characterize the new substances in the most detailed way.

3.4.Importance of learning outcomes

The precise three-dimensional structural characterization of single crystal samples obtained from X-ray diffraction analysis (or using other related radiations), is a key point in the design of new molecules or materials at nano- micro- or macroscopic scale, and, in particular, in the comprehension of reactive processes in Molecular Chemistry and Catalysis.

In the case of polycrystalline samples, diffraction analysis allows the identification, and sometimes the quantification, of the phases of a specific sample. Therefore, they may be useful in the comprehension of reactive processes involving solids, and may be a technique applied in the quality control mechanism, either before or after the specific chemical process.

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4.Evaluation

A continuous and comprehensive evaluation of the student competences will be carried out based on their performance in the following activities:
1.- Daily class work, including problem solving tests, practical questions, exercises and related activities during the learning process (30%).
2.- Oral presentation of the structural results of a recently published scientific article, jointly chosen by tutors and students and in some way related to the student's research interests. (20%).
3.- Written or oral test (decided by students) at the end of the course dealing with problems and questions about the course contents (50%).
The students that have not passed the subject or wish to improve their score have the option to carry out a global test including a comment on the structural part of a recently published scientific paper related with the Master topic (40%) and the answer of theoretical questions about the course topics (60%).

The number of official examination calls per registration and their use will be subjected to the statements of the *Regulation of Permanence in Master Studies and* *Regulation of the Learning Assessment* . The latest document will also regulate the general design and scoring criteria of the assessment activities, as well as the exam schedules and timetable for the post-examination review.

5.Activities and resources

5.1.General methodological presentation

The learning process will use daily class lectures, previously available to the students, in which tutors will set out questions to the students in order to stimulate their participation in the description and comprehension of basic concepts. From a classical perspective, the class distribution may be classified in the following way:

1.- Theoretical classes (1.4 ECTS).

2.- Problem solving and seminars (0.2 ECTS).

3.- Practical sessions with laptops dealing with real data processing (0.4 ECTS).

In all the activities, the students will be prompted to display an active participation, stimulating discussions about theoretical concepts, and substantial contributions to the practical work.

5.2.Learning activities

Several learning activities are included in the proposed program to help students to reach the expected results: curricular adaptation texts at the beginning of the course to those students requiring them; interacting classes; analysis, study and discussions of complementary texts in a classical learning scheme or using 'flipped classroom' methodologies.

Additionally, some practical exercises about spatial symmetry, systematic absences evaluation, reciprocal lattice or about the Patterson method will be proposed in a collaborative method (suggesting the students to work in groups).

Moreover, the tutors will carry out, at least, one structure solution and refinement following students' indications.

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5.3.Program

According with student's previous knowledge about Crystallography (solid state Chemistry and symmetry) several curricular adaptations may be carried out to the planned program. The scheduled program is originally organized with the following topics:

0 .- Structure/Properties: Paradigm of modern Science. The Crystal: how to simplify a so complex entity? Diffraction experiments: the way to the unambiguous molecular structure.

1 .- Structural Crystallography. General introduction. Crystallography applications.

2 .- Spatial symmetry. Space groups. Nomenclature. International Tables.

3 .- Crystal growth. Crystallization methods and crystal handling.

4 .- X-rays and another radiations used in structural studies. Scientific equipment for diffraction experiments.

5 .- Crystal structure and diffraction. Singlecrystal and powder analysis methods. Reflections and intensities. Bragg law, reciprocal lattice and Structure Factor. Ewald model.

6 .- Diffraction data integration for the molecular structure determination. Structural refinement. Temperature factor. Absolute structure determination.

7 .- Results presentation and validation. Structural data in their context: structural databases.

8 .- Diffraction experiments with powder or partially ordered samples. Some applications and methodologies.

9 .- Singlecrystal diffraction. Programs for structure solution and refinement: WINGX and SHELX. Steps in a conventional structure determination.

10 .- Presentation and discussion (carried out by the students) of recently published scientific articles in 'Molecular Chemistry and Catalysis' field.

5.4.Planning and scheduling

The course timetable and exams dates will be published in the noticeboard of Inorganic Chemistry Department and in the Science Faculty webpage (https://ciencias.unizar.es). Dates for the students' expositions will be agreed with the students along the course.

The students will be provided with diverse teaching material either at reprography or through the University's web tool: https://moodle2.unizar.es/add .

5.5.Bibliography and recomended resources

Basic bibliography

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- Clegg, William [et al.]. Crystal structure analysis: principles and practice. 1st ed. Oxford: Oxford University Press, 2006
- Clegg, William [et al.]. Crystal structure analysis : principles and practice. 2nd. ed. Oxford: Oxford University Press, 2009
- Massa, Werner. Crystal structure determination; translated into English by Robert O. Gould. 2nd completely updated ed. Berlin : Springer, 2004
- Hammond, Christopher. The basics of crystallography and diffraction. 4th ed. Oxford: International Union of Crystallography: Oxford University Press, 2015
- Giacovazzo, Carmelo [et al.]. Fundamentals of crystallography. Edited by C. Giacovazzo . 2nd ed. Oxford: Oxford University Press, 2002
- Giacovazzo, Carmelo [et al.]. Fundamentals of crystallography. Edited by C. Giacovazzo . 3rd ed. Oxford: Oxford University Press, 2011

Complementary bibliography and specialized texts

- Muller, Petter [et al.]. Crystal structure refinement : a crystallographer's guide to SHELXL. Edited by Peter Müller Oxford : Oxford University Press, 2006
- Prince, Edward. Mathematical techniques in crystallography and material science. 2nd ed. Berlin. Springer, cop. 1994
- Prince, Edward. Mathematical techniques in crystallography and material science. 3rd ed. Berlin. Springer, cop. 2004
- Jenkins, Ron; Snyder, Robert L. Introduction to x-ray powder diffractometry. John Wiley & Sons, cop. 1996.
- International tables for Crystallography. Vol. A, Space- group symmetry. Edited by Theo Hahn. 2nd, rev. ed Dordrecht : International Union of Crystallography, 1989
- International Tables for Crystallography, Volumen A1 (Symmetry relations between space Groups). 2nd. ed. Wiley. 2010
- International tables for crystallography. Vol. A, Space- group symmetry. Edited by Theo Hahn . Brief teaching ed. 2nd rev. ed. reprint with corr. Dordrecht : International Union of Crystallography, 1989
- International tables for crystallography. Vol. A, Space- group symmetry. Edited by Theo Hahn . Brief teaching ed. 2nd rev. ed. reprint with corr. Dordrecht : International Union of Crystallography, 2010
- International Tables for Crystallography, Volume A, 5th Edition, Space-Group Symmetry. 5th. rev. ed. Wiley. 2005

Recommended Web sites

- http://www.iucr.org/education
- http://www.iucr.org/books