Universidad Zaragoza

**Year : 2019/20**

# 60456 - Crystallography and diffraction techniques

## Syllabus Information

**Academic Year:** 2019/20
**Subject:** 60456 - Crystallography and diffraction techniques
**Faculty / School:** 100 -

**Degree:** 543 - Master's in Molecular Chemistry and Homogeneous Catalysis

**ECTS:** 2.0
**Year:** 1
**Semester:** Second semester
**Subject Type:** Optional
**Module:** ---

# 1.General information

## 1.1.Aims of the course

The course aims at training the students to recognize the potential application of diffraction techniques in any chemical research field, either basic or applied, and to be able to follow the whole procces of structural determination, from data collection and reduction processes to the extraction of the relevant structure information, at a 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 a R&D industrial context, either in production or in quality control departments.

## 1.2.Context and importance of this course in the degree

The knowledge of crystallographic techniques is fundamental for definitive and precise structural characterization (at molecular or crystalline level) of new substances or natural products from solid state samples. The structural knowledge obtained from X-ray diffraction experiments results to be 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 involved or specially 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.

## 1.3.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 also 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.

# 2.Learning goals

## 2.1.Competences

To be able to design diffraction experiments to obtain relevant structural information (at molecular or crystal level), understanding which are the most suitable data collection equipments, 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 obtained results, according to the research area under study.

Capacitiy to use the obtained structural information for understanding and for the interpretation of reactive processes in Molecular Chemistry and Catalysis and set it 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 new substances in the most detailed way.

### 2.2.Learning goals

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 basic mechanism for the phase problem solution and the experimental proceedings adequate for the structure solution from X-ray diffraction experiments.

To interpret main directional characteristics of dispersed X-rays and to evaluate 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.

### 2.3.Importance of learning goals

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 fundamental technique applied in the quality control mechanism, either before or after the specific chemical process.

## 3.Assessment (1st and 2nd call)

### 3.1.Assessment tasks (description of tasks, marking system and assessment criteria)

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 (40%).
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 (40%).
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 the *Regulation of the Learning Assessment* (
https://ciencias.unizar.es/normativas-asuntos-academicos) 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.

## 4.Methodology, learning tasks, syllabus and resources

### 4.1.Methodological overview

The learning process will use daily class lectures, with presentations 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 traditional perspective, the methodology may be classified in the following way:

1. Lectures (1.5 ECTS).

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

3. Practice sessions with laptops dealing with real data processing (0.3 ECTS).

Students are expected to participate actively in the class throughout the semester, stimulating discussions about theoretical concepts and substantial contributions to their practical work.

## 4.2.Learning tasks

The course includes the following learning tasks:

- Curricular adaptation texts at the beginning of the course for those students who require them.
- Interactive classes.
- Analysis, study and discussions of complementary texts in a traditional teaching way or using 'flipped classroom' methodologies.
- Collaborative and group work: practical exercises about spatial symmetry, systematic absences evaluation, or reciprocal lattice.
- Tutors will carry out, at least, one structure solution and refinement following 'students' indications.

## 4.3.Syllabus

Depending on the students' previous knowledge about Crystallography (solid state Chemistry and symmetry), several curricular adaptations may modify the syllabus. Therefore, the provisional syllabus of the course will address the following topics:

**Topic 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.

**Topic 1**. Structural Crystallography. General introduction. Crystallography applications.

**Topic 2**. Spatial symmetry. Space groups. Nomenclature. International Tables.

**Topic 3**. Crystal growth. Crystallization methods and crystal handling.

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

**Topic 5**. Crystal structure and diffraction. Single crystal and powder analysis methods. Reflections and intensities. Bragg law, reciprocal lattice and Structure Factor. Ewald model.

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

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

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

**Topic 9**. Single crystal diffraction. Programs for structure solution and refinement: WINGX and SHELX. Steps in a conventional structure determination.

**Topic 10**. Presentation and discussion (by the students) of recently published scientific articles in the ?Molecular Chemistry and Catalysis' field.

## 4.4.Course planning and calendar

The course timetable and exams dates will be published in the bulletin board of Inorganic Chemistry Department and on the Faculty of Science webpage (https://ciencias.unizar.es).

Dates for the students' presentations 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 virtual platform https://moodle2.unizar.es/add.

This course has a workload of 2ECTS and 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.

## 4.5.Bibliography and recommended resources