Fall 2021

CHEM 488: Introduction to Theory and Practice of X-ray Crystallography.

1. Basic Information

Time:	Tues/Thurs 11:00 – 12:30, Discussion Friday 11:00-12:30
Location:	SGM 307
Unit Value:	4 units
Grading:	Letter Grade
Instructor:	Professor Mark Thompson; met@usc.edu
	SGM 216; 740-6402
Office Hours:	By Appointment
Prerequisites:	CHEM 300L; CHEM 322abL or CHEM 325abL or instructor's permission.
Class Web Page:	USC Blackboard page

EVERYONE is expected to be in class and follow all COVID guidelines set by USC; failure to do so may result in expulsion from the course. The lectures will also be recorded on Zoom and will be available for you to review after class on the course Blackboard page. Any student that would like to take this class in an on-line mode must present a written OSAS accommodation from the Office of Student Accessibility Services that permits them to take the class online-only.

2. Course Goals and Learning Objectives

The main goal of this course is to provide you with a practical understanding of crystallography and crystal structure determination. You will be introduced to the concepts of three-dimensional symmetry, X-ray diffraction theory, crystallographic data collection, structure solution, and structure refinement. Our goal was for you to carry-out a complete single-crystal X-ray structure analysis. We are installing a new diffractometer early int eh Fall so every student will take part in a crystal solution as part of this course.

After successfully completing this course, you should be able to:

- Understand the origin, interpretation, uses, and limits of crystallographic studies.
- Understand the nature of the solid state, the types of solid state structures, and their relationship to solution and gas phase structures.
- Prepare and select crystalline samples, and evaluate their suitability for diffraction studies.
- Understand crystallography terminology reported in the literature.
- Critically analyze crystallographic results to evaluate their reliability and utility.
- Collect data and solve structures using the departmental diffractometer

3. Recommended Textbooks

- W. Massa, *Crystal Structure Determination*, 3rd ed., Springer 2016
- Inorganic Chemistry text with a good coverage of group theory.

4. Supplementary Textbooks and References

- Christopher Hammond, *The Basics of Crystallography and Diffraction*, 4th ed., Oxford Science Publications 2016 (IUCr Texts on Crystallography 12)
- Jenny P. Glusker, Kenneth N. Trueblood, *Crystal Structure Analysis A Primer*, 3rd ed., Oxford Science Publications 2010 (IUCr Texts on Crystallography 14)
- Dennis W. Bennett, *Understanding Single-Crystal X-Ray Crystallography*, Wiley-VCH 2010
- Richard Tilley, *Crystals and Crystal Structures*, Wiley 2006
- Walter Borchardt-Ott, *Crystallography*, 2nd ed., Springer 1995
- Alexander J. Blake et al., *Crystal Structure Analysis Principle and Practice*, Oxford Science Publications 2009 (IUCr Texts on Crystallography 13)
- Mark Ladd and Rex Palmer, Structure Determination by X-ray Crystallography, 4th ed., Kluwer 2003 (more advanced)
- Carmelo Giacovazzo et al., *Fundamentals of Crystallography*, 2nd ed., Oxford Science Publications 2002 (IUCr Texts on Crystallography 7)
- Peter Müller et al., *Crystal Structure Refinement A Crystallographer's Guide to SHELXL*, Oxford Science Publications 2005 (IUCr Texts on Crystallography 8)
- Theo Hahn, *International Tables for Crystallography Volume A Space-group symmetry*, 5th ed., Springer 2005
- Bob B. He, *Two-Dimensional X-Ray Diffraction*, Wiley 2009

5. Reasons for Taking this Course

Crystals and the understanding of crystals play a vital role in many subjects, among them mineralogy, inorganic, organic and physical chemistry, physics, metallurgy, materials science, geology, geophysics, biology, and medicine. Practically all information about the molecular structure of matter at atomic resolution is the result of crystallographic analysis. Diffraction methods have contributed to our fundamental understanding of minerals and ceramics, and to the design of material properties, pharmaceuticals and engineered enzymes. It is essential to understand and appreciate the origins of molecular structural information and the biological, chemical and physical implications derived from this information. Crystallography underscores the importance of interdisciplinary science directly and tangibly affects all our lives through advances in biosciences up to space-age high tech materials development.

For a long time, X-ray crystallography remained a highly specialized technique that was inaccessible to the average undergraduate or even the postgraduate. However, with the development of more and more sophisticated diffractometers, the steady rise in computing power, and the advances in crystallographic computer software, X-ray crystallography has become a routine method. It is now more important than ever to have a basic knowledge of the process of crystal structure determination and be able to evaluate crystallographic results and data.

6. Course Description

The first part of this course focuses on the foundation and background of X-ray crystallography, starting with general aspects of symmetry, crystal systems, Bravais lattices, and unit cells, moving on to the concepts of diffraction theory, structure factors and reciprocal space. The second part of the course covers the various steps associated with a single-crystal X-ray structure analysis. This includes methods for growing crystals, the process of data collection, space group determination, structure solution and refinement, and the use of crystallographic software for molecular structure determination (e.g. SHELXL).

7. Schedule of Classes

- You need to read the textbook ahead of the lectures! You guys are grown-ups, so I won't be telling you exactly what to read for the next lecture. Look at your notes and figure it out. You should be able to find the relevant sections of whatever book you choose to use as your textbook from the following topics and the table of contents.

1. Course introduction: Overview, textbooks; Introduction to crystals and crystal structures.

2. Symmetry: Definition of symmetry, molecular symmetry, symmetry elements and symmetry operators, point groups, character tables.

3. Symmetry in 3D: Space groups, screw axes and glide planes, the unit cell and crystallographic conventions.

4. X-rays and matter: Generation of X-rays, diffraction, convolution theorem and Fourier transformation, Bragg's law and Miller indices.

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6. Geometry of diffraction: Reciprocal space vs. real space, Ewald construction.

7. Structure factors: atomic form factors and atomic displacement factors, electron density map, crystallographic resolution.

8. Symmetry in reciprocal space: Friedel's law, Laue groups, space group determination, systematic absences, crystallographic directions.

9. Growing crystals, X-ray diffraction instrumentation, practical aspects.

10. Crystal Structure Analysis: X-ray data collection and reduction, phase problem, Patterson and Direct Methods.

11. Structure refinement: Different type of electron density maps, anisotropic displacement parameters, least-square refinement, R- factors, crystallographic parameters, constrains and restrains.

12. Problems and pitfalls: Wrong space group, atom assignment, disorder, twinning, artifacts, hydrogen atoms, absolute configuration.

13. Structure report: Computation of structural parameters, computer graphics and molecular structure display, crystallographic databases.

8. Exams and Grading

Your final grade will be based on one midterm, one final exam, and homework assignments, as follows:

Grade Breakdown	Proportion
Midterm	30 %
Final Exam	40 %
Homework	30 %
Total	100 %

8.1 *Examinations*: The midterm and the final exam are closed book tests for which you are only allowed a calculator, a ruler, and a pen. The final covers the course material studied during the entire semester. The tests use a combination of multiple choice and free response questions. Students who are not able to attend an examination must notify the instructor before the test. The Final Exam for this course is December 14th, 8:00-10:00 AM.

8.2 *Homework*: The homework consists of problems and exercises that test your understanding of the course material and help you prepare for the in-class exams. Homework will be posted on the class web page. Homework must be turned in to the course blackboard page at the beginning of class on the day specified on the assignment. If you tun in something that is a mess don't expect much in the way of a score.

9. Statement for Students with Disabilities

Any student requesting academic accommodations based on a disability is required to register with Disability Services and Programs (DSP) each semester. A letter of verification for approved accommodations can be obtained from DSP. Please be sure the letter is delivered to me (or to TA) as early in the semester as possible. DSP is located in GFS 120 and is open 8:30 a.m.–5:00 p.m., Monday through Friday. Website for DSP (https://dsp.usc.edu/) and contact information: (213) 740-0776 (Phone), (213) 740-6948 (TDD only), (213) 740-8216 (FAX) dspfrontdesk@usc.edu.

10. Statement on Academic Integrity

USC seeks to maintain an optimal learning environment. General principles of academic honesty include the concept of respect for the intellectual property of others, the expectation that individual work will be submitted unless otherwise allowed by an instructor, and the obligations both to protect one's own academic work from misuse by others as well as to avoid using another's work as one's own. All students are expected to understand and abide by these principles. SCampus, the Student Guidebook, contains the University Student Conduct Code (see University Governance, Section 11.00), while the recommended sanctions are located in Appendix A.