CRYSTL & MOLEC STRC II 16:160:536:01, Fall 2021

Instructors	Sanjeeva Murthy (Chemistry) murthy@chem.rutgers.edu Tom Emge (Chemistry) emge@chem.rutgers.edu
Lectures	Wednesday 4:00 - 6:00 PM Busch CIP-120 (Center for Integrative proteomics)
Lab sessions	3 h blocks. By arrangement and in groups of four
Credits	3
Prerequisites	01:160:327, 328, or equivalent
Office hours	By prior arrangement via email
Text book	X-ray Crystallography by G.S. Girolami, University Sciences Books, 2015
Description	In this graduate level course, students will learn the fundamental and practical aspects of X-ray diffraction methods in sufficient detail to: (1) solve the structures of crystalline materials from X-ray diffraction data, (2) analyze data from powders, fibers, polymers and disordered materials, and (3) critically evaluate crystal structures and XRD data reported in the literature. The course will emphasize both the theoretical understanding of X-ray diffraction methods as well as laboratory hands-on work in collecting and analyzing x-ray diffraction data.
Organization	The course consists of one 2-hour lecture per week, and lab sessions as will be discussed on the first day of the lecture. See the course schedule for more details.
Syllabus	**Symmetry and the Crystalline State**: Transitional symmetry and the unit cell; notation for lattice planes and directions; symmetry operations; point groups and space groups. **The Theory and Experimental Aspects of X-ray Diffraction**: The geometrical conditions for diffraction; the reciprocal lattice and Ewald sphere; the form and structure factors; the selection and mounting of crystals; diffraction experiments and data analysis. **Fourier Analysis**: Fourier transforms; the relationship between diffraction data and the electron density distribution within a crystal; the phase problem; Fourier maps. **Structure Solution and Refinement**: Methods of structure solution, including Patterson maps, and direct methods; structure models and their refinement; validation and critique of structure refinements. **Special Topics**: Depending on the time available and interest of class, additional topics could include, state-of-the-art single crystal data collection, Rietveld structure refinement using powder diffraction data, analysis of helical and fibrous diffraction patterns from biological structures, analysis of data from disordered structures such as polymers, small-angle x-ray scattering and synchrotron radiation techniques.
Grading	Grades for this course will be based on the completion of the x problem sets to be assigned, lab practical assignments, midterm exam, and oral and written components of the final exam. Problem sets – 20%. Lab assignments – 30%. Two quizzes (midterms) – 40%. Final oral presentation – 10%. Total – 100%.

Laboratory Syllabus (Five 1-hr sessions)
Students must be on the roster & must attend all 5 sessions.

1. Safety; introduction to the x-ray generator; optically assessing the sample via microscope; mounting the crystal on the diffractometer; assessing the crystallinity of the sample; introduction to data collection; determination of unit cell and Bravais lattice; data collection strategy; initiate data collection.
2. Assessment of collected data and structure solution; examination of resolution, completeness and crystallinity requirements; data reduction, including integration, scaling and absorption correction; test and critique of several solution methods.
3. Polymer, fiber, powder diffraction; measurement of crystallinity, anisotropy, % amorphous.
4. For powders, phase identification and relative abundances (semi-quantitative methods); Single crystal structure refinement.
5. Completing and correcting the model; least-squares and conjugant gradient refinements; key model convergence tests; use of checkCIF; publication requirements; precision, accuracy and reproducibility of the result; in-class exam.