Chemistry 6181 **MWF 11:05** [redacted]

Course title: "Chemical Crystallography"

Topic: Atomic scale structure analysis of small molecules and materials (not macromolecules) using x-ray and neutron elastic scattering. The course will cover the use of single crystal and powder diffraction for structure analysis.

Instructor: [redacted]

Classroom: MoSE G021

Texts: Course not based on a single text. The following books will be useful resources:

"Fundamentals of Powder Diffraction and Structural Characterization of Materials", 2^{nd} edition, Springer. Pecharsky and Zavalij.

"International Tables for Crystallography, Vol. A, Teaching Edition" This is available electronically through the GT library.

Grading: Student presentation and report (combined 10% of course grade): Topics will be assigned by the instructor. Report and presentation will be on the same topic. Reports (3 pages) will be evaluated by the instructor (5%). Presentations (15-20 minutes total time including questions) will be evaluated by the instructor and other class members (5%).

"Hour Exams": (total 50%) Two exams will be given during normal class periods.

Final Exam: (40%) The exam will be given during the scheduled exam period.

Problem sets: There will be some non-graded problems assigned to help with test preparation

Review sessions: Review sessions prior to test will be held if requested.

Office hours: By appointment. Please make appointments by e-mail, as I will have access to my calendar while I am looking at the e-mail.

Topics to be covered by the instructor

1) Introduction to atomic scale structure analysis of small molecules and materials using x-ray and neutron elastic scattering
2) Production and nature of x-rays and neutrons suitable for “crystallography”.
    a. X-rays: Tubes, synchrotrons etc
    b. Neutrons: Radioactive sources, reactors, spallation, moderation
    c. Polarization
3) Introduction to waves and interference phenomena
    a. Wave characteristics
    b. Interference
    c. Plane and spherical waves
    d. Double slit interference
    e. Diffraction from a finite slit
    f. Diffraction and sampling
    g. Intro to optical transforms
4) Interaction of x-rays and neutrons with mater
    a. Coherent elastic scattering, incoherent elastic scattering, absorption, inelastic scattering.
    b. Scattering from atoms – the form factor
    c. Resonant elastic scattering
    d. Magnetic scattering of neutrons
    e. Processes useful for detection such as gas ionization, scintillation, charge generation in semiconductors, and nuclear reactions.
5) Scattering from clusters and particles –
    a. Scattering from molecules and clusters (Debye function)
    b. Small angle scattering
6) Translational symmetry
    a. Introduction to lattices, unit cells, lattice planes, Miller indices, reciprocal lattice
7) Diffraction geometry (single xtal and powder)
    a. Reflection of lattice planes and Bragg’s law

b. Reciprocal space and the Ewald construction
c. Reflection overlap in powder diffraction patterns

8) A digression: understanding DNA diffraction using what we have learnt
9) Point symmetry in molecules, crystals and their diffraction patterns
10) Space group symmetry in 3D.
    a. Screw axes and glide planes
    b. Putting all types of symmetry together
11) Space group assignment.
    a. Laue symmetry
    b. SHG and other physical measurements to help assign symmetry
    c. Systematic absences,
    d. Renninger effect (see Werner Massa's book).
12) Theory behind diffracted intensities.
    a. Relationship between intensity and structure factor
    b. Calculation of structure factors including thermal parameters
    c. Representation of structure factors on an Argand diagram
    d. Extinction
13) Measurement of diffraction patterns and data reduction
    a. Diffractometer types (kappa, eularian cradle etc.)
    b. Detectors (point detectors and area detectors for both x-rays and neutrons)
    c. Optics (monos, mirrors including capillaries and neutron guides),
    d. Selection of samples
    e. Indexing/orientation matrix
    f. Absorption corrections, extinction corrections, Lp correction
    g. Integrating 2D powder patterns
    h. Texture/preferred orientation in polycrystalline samples
14) Sample preparation
    a. Crystal growth
    b. Polycrystalline samples
15) The role and nature of Fourier syntheses in "crystallography"
16) Structure solution methods (solving the "phase problem")
    a. Patterson methods
    b. Direct methods

    c. Charge flipping
    d. Isomorphous replacement and MAD
    e. “Real space” methods

17) Scattering-Density Maps
    a. Different types of Fourier map (F, difference etc)
    b. Map resolution
    c. Maximum entropy maps

18) Refinement (optimization) of structures using least squares and other methods.

19) Powder profile modeling
    a. The Rietveld method for structures using powder diffraction data
    b. Structure factor estimation using the LeBail and Pawley methods

20) Solving structures from powder diffractions patterns

21) Restraints, constraints, twinning, chirality and absolute structure

22) The derivation and presentation of results: graphics, coordinates, distances and angles, thermal parameters

23) Incommensurate “crystals” and “quasicrystals”

24) Examples of Synchrotron X-ray and neutron use

25) Non-ambient experiments: Low T, high T, high P, reactive gases

26) Using multiple scattering data sets for cation substitution and other problems

27) Disorder, diffuse scattering and the Pair Distribution Function

Assignment of student presentation and report topics

Each student should prepare a ~20 minute power point presentation on their assigned topic. The presentation should be designed to provide their classmates with an introductory understanding of the assigned topic. Questions regarding the appropriateness of content are welcome. A copy of the presentation should be forwarded to the instructor 2 days before the presentation date.

Each student should prepare a 3 page written report on the same topic. The report should be delivered to the instructor prior to Finals week.

Student	Topic	Presentation Date
	Single crystal growth in gel media	
	Single crystal growth by vapor transport. This should include high temperature chemical vapor transport and low temperature solvent vapor transport.	
	“Twinning” in crystallography	
	What is a “composite” crystal and why are they important? Note that this is not about “composites”, it is about composite crystals.	
	Devices for performing crystallographic (powder or single crystal diffraction) experiments at low temperatures	

Resource books for crystallography

1) "Crystal Structure Analysis for Chemists and Biologists", Glusker, Lewis and Rossi, VCH, 1994.

2) "International Tables for Crystallography", brief teaching edition, Vol. A, Kluwer, 1993. The full version just has additional space groups. The notes are the same.

3) "Crystal Structure Analysis A Primer", Glusker and Trueblood, Oxford, 1985. This book is a simpler version of book 1).

4) "X-ray Structure Determination A Practical Guide", Stout and Jensen, Wiley, 1989. A good introductory book.

5) "Fundamentals of Crystallography", ed. Giacovazzo, Oxford Scientific Publications, 1992. A good crystallography book, but not for absolute beginners

6) "Structure Determination by X-ray Crystallography", Ladd and Palmer, Plenum, 1994. A reasonable book. Aimed at possible practioners not people who just need an overview.

7) "X-ray Diffraction", Warren, Dover, 1990. A classic. Very mathematical. It is about X-ray scattering not crystal structure determination.

8) "Elements of X-ray Diffraction", Cullity and Stock, Prentice-Hall, 2001. Primarily about powder diffraction

9) "The Rietveld Method", Ed. R. A. Young, Oxford Science Publications, 1993. Devoted to Rietveld analysis. Rietveld analysis is an approach to extracting structural information from powder diffraction patterns. It requires that you already have a good knowledge of what the structure is like.

10) "Structure Determination from Powder Diffraction Data", Eds. David, Shankland, McCusker and Baerlocher, Oxford Science Publications, 2002.