

26:160:535 CHEMICAL STRUCTURE DETERMINATION - FALL 2021

X-Ray Crystallography

(Tentative Syllabus)

Thursdays 6:00-9:00 - Smith 242

Professor: Dr. Roger A. Lalancette 973-353-5646
X-Ray Laboratory - Olson 010 (Apex2) & 008 (Rigaku CrysAlisPro)

Textbook: *X-Ray Structure Determination: A Practical Guide*, Stout & Jensen, 2nd Ed., Wiley, NY, 1989 ISBN: 978-0-471-60711-3. Available on the internet.

I have a number of other books in the X-ray laboratory which may be of some use to you. You will be allowed to check a book out for a period of 2 weeks (so that others may get a chance to look at them).

Crystal Structure Determination, Werner Massa, 2nd Ed., Springer-Verlag, NY
ISBN 3-540-20644-2

An Introduction to X-Ray Crystallography, M. Woolfson, Cambridge Univ. Press, 1970. (2 copies).

International Tables for X-Ray Crystallography, Kynoch Press, Birmingham, England, 1965. Vol. I-IV.

Crystal Structure Analysis for Chemists and Biologists, Jenny P. Glusker, Mitchell Lewis & Miriam Rossi, VCH Publishers, NY, 1994.

Principles of Crystal Structure Determination, G. B. Carpenter, W. A. Benjamin, NY, 1969.

X-Ray Crystallography, M. J. Buerger, John Wiley & Sons, NY, 1942.

For the more biologically-oriented student: *Principles of Protein Structure*, G. E. Schulz & R. H. Shirmer, Springer-Verlag, NY, 1978.

And there are many more.

Also: <http://www.iucr.org/education/pamphlets> website: Teaching Pamphlets: there are 23 of these, mostly short synopses of the different topics, some very worthwhile.

Supplementary Materials: MIT OpenCourseWare:

Go to: <http://ocw.mit.edu/OcwWeb/Chemistry/5-069Spring-2008/CourseHome/index.htm>

Disclaimer: I want to acknowledge that I will be using parts of the MIT Open Course Ware notes as part of this course – this is as specified in the MIT Open Course Ware information.

Tentative Syllabus:

<u>Lec. #</u>	<u>Topics</u>	<u>Stout & Jensen</u>
1	Introduction , Overview of Course, Textbooks and Associated Materials, History of Crystallography, Overview of a Crystal Structure Determination (the steps required to do a structure), X-Ray Generation, the Lattice, Unit Cell, Asymmetric Unit, Crystallographic Conventions 535xraylecture1.doc , FORMULAE.doc , Campana1.ppt	1,2 090221
2	Symmetry in 2D : Definition of Symmetry, Introduction of Symmetry Operators Symmetry in 3-D : 7 Crystal Systems, 14 Bravais Lattices, Reduced Cell (Niggli Matrix) Stereographic Projections, Space Groups, Introduction of Screw Axes and Glide Planes, Point Groups vs. Space Groups 535xraylecture2.doc , screwaxes.doc	3,4 090921
3	Special Lecture: Dr. Alessia Provino - Powder XRD, General and Rietveld (quantitative) Methods.	091621
4	X-Rays and Matter : Convolution Theorem and Fourier Transformation, Introduction of Bragg's Law and Miller Indices 535xraylecture3.doc , 535raylecture3(cont).doc	2,6,7 092321
5	Geometry of Diffraction : Reciprocal Space vs. Real Space, Ewald Construction as a Geometric Interpretation of Bragg's Law, Total Number of Possible Reflections 535xraylecture4.doc	6,7,8 093021
6	Structure Factors : Real Atoms are not Point Atoms (Atomic Form Factors), Thermal Motion (Atomic Displacement Factors), Fourier Transformation Gives Rise to Electron Density, Crystallographic Resolution 535raylecture5.doc	8,9 100721
7	Structure Factors (cont.) : Complex Numbers, Euler's Equation and the Argand Plane, Introduction of the Phase Problem 535xraylecture6.doc	101421
8	Midterm Exam	102121
9	Symmetry in Reciprocal Space : Friedel's Law and Laue Groups, Space Group Determination: $ E^2 - 1 $ Statistics, Systematic Absences, Crystallographic Directions for Triclinic, Monoclinic, Orthorhombic, Tetragonal Systems. 535xraylecture7.doc	10
10	Special Lecture: Dr. Pierre LeMaguères - Director of Scientific Support, Rigaku Americas Corporation, 9009 New Trails Drive, The Woodlands, TX 77381: Data collection on a X-Ray single crystal diffractometer: procedure, best practices and tips	102821
11	Structure Solution : Patterson Function and Harker Sections, Direct Methods 535xraylecture8.doc	11,12 110421

12	Structure Refinement: Different Types of Electron Density Maps (Fo, Fc, F0-Fc, 2Fo-Fc, etc.), Isotropic Temperature Factors, Anisotropic Displacement Parameters, Minimization Functions (Least-Squares Approach), Different R-Factors, Constraints & Restraints 535xraylecture9.doc , 535xraylecture10.doc	15,16 111121
	Structure Refinement (cont.): Problems & Pitfalls: Wrong Space Group, Wrong Atom Type Assignment, Disorder, Twinning, Finding Hydrogen Atoms, Refining Using “Riding Models” 535xraylecture11.doc	111821
13	Anomalous Scattering: Absorption of X-Ray Photons Leads to Loss of Symmetry in Orbital Geometry, which Results in a Violation of Friedel’s Law 535xraylecture12.doc	15,16 120221
14	Practical Aspects & Related Methods: (RECAP) Growing Crystals & Keeping Them Alive (Never Remove the Mother Liquor!), Mounting Crystals on the Diffractometer, How to Begin Collecting Information – Unit Cell, Bravais Lattice, Index Faces for Absorption Correction, Short Introduction to Powder Diffraction, Neutron Diffraction, Crystallographic Databases (ICSD, CSD, CRYST-MET, PDB, Reciprocal Net), Deposition of Structural Data in Data Bases Interpretation & Presentation of Results: Derived Results: Bond Lengths & Angles, Best Planes. & Torsion Angles, Structural Geometry & Symmetry, Structural Diagrams (ORTEP), Parameters/ Constraints/ Restraints, Anisotropic Displacement Parameters, Libration, Hydrogen Atoms, Publication of Results	4,13,18 120921

Grading for the course:

- 1- 7 HOMEWORK SETS TO BE HANDED IN (AND GRADED, AND HANDED BACK) – 20 POINTS
- 2- ONE MIDTERM (ON OCTOBER 21) – 25 POINTS, AND THE FINAL – 35 POINTS
- 3- EVERY STUDENT WILL SOLVE A STRUCTURE ON THEIR OWN (IF THEY HAVE THEIR OWN CRYSTALS TO WORK ON, THEY WILL COLLECT THE DATA AND THEN SOLVE THE STRUCTURE.)
IF THEY DO NOT HAVE MATERIAL TO WORK ON, I WILL PROVIDE A MOUNTED CRYSTAL FOR THEM TO COLLECT DATA, AND THEN THEY WILL SOLVE THE STRUCTURE, AND SUBMIT THEIR RESULTS – 20 POINTS
- 4- NOTES WILL BE HANDED OUT AHEAD OF TIME SO THAT THE STUDENTS WILL BE ABLE TO FOLLOW THE LECTURES WITH LESS DISTRACTION

Academic Integrity

As an academic community dedicated to the creation, dissemination, and application of knowledge, Rutgers University is committed to fostering an intellectual and ethical environment based on the principles of academic integrity. Academic integrity is essential to the success of the University's educational and research missions, and violations of academic integrity constitute serious offenses against the entire academic community. The entire Academic Integrity Policy can be found here: <http://academicintegrity.rutgers.edu/academic-integrity-policy/>

Accommodation and Support

Rutgers University Newark (RU-N) is committed to the creation of an inclusive and safe learning environment for all students. RU-N has identified the following resources to further the mission of access and support:

- **Students with Disabilities:** Rutgers University welcomes students with disabilities into all of the University's educational programs. The Office of Disability Services (ODS) is responsible for the determination of appropriate accommodations for students who encounter barriers due to disability. In order to receive consideration for reasonable accommodations, a student with a disability must contact ODS, register, have an initial appointment, and provide documentation. Once a student has completed the ODS process (registration, initial appointment, and documentation submitted) and reasonable accommodations are determined to be necessary and appropriate, a Letter of Accommodation (LOA) will be provided to the student. The student must give the LOA to each course instructor, followed by a discussion with the instructor. This should be completed as early in the semester as possible as accommodations are not retroactive. More information can be found at ods.rutgers.edu. Contact ODS: (973) 353-5375 or ods@newark.rutgers.edu.
- **Religious Holiday Policy and Accommodations:** Students are advised to provide timely notification to instructors about necessary absences for religious observances and are responsible for making up the work or exams according to an agreed-upon schedule. The Division of Student Affairs is available to verify absences for religious observance, as needed: (973) 353-5063 or DeanofStudents@newark.rutgers.edu.
- **Counseling Services:** Counseling Center Room 101, Blumenthal Hall, (973) 353-5805 or <http://counseling.newark.rutgers.edu/>.
- **Students with Temporary Conditions/Injuries:** Students experiencing a temporary condition or injury that is adversely affecting their ability to fully participate in their courses should submit a request for assistance at: <https://temporaryconditions.rutgers.edu>.
- **Students Who are Pregnant:** The Office of Title IX and ADA Compliance is available to assist students with any concerns or potential accommodations related to pregnancy: (973) 353-1906 or TitleIX@newark.rutgers.edu.
- **Gender or Sex-Based Discrimination or Harassment:** Students experiencing any form of gender or sex-based discrimination or harassment, including sexual assault, sexual harassment, relationship violence, or stalking, should know that help and support are available. To report an incident, contact the Office of Title IX and ADA Compliance: (973) 353-1906 or TitleIX@newark.rutgers.edu. To submit an incident report: tinyurl.com/RUNReportingForm. To speak with a staff member who is confidential and does NOT have a reporting responsibility, contact the Office for Violence Prevention and Victim Assistance: (973) 353-1918 or run.vpva@rutgers.edu.

Learning Resources:

- **Rutgers Learning Center** (tutoring services)

Room 140, Bradley Hall

(973) 353-5608

<https://sasn.rutgers.edu/student-support/tutoring-academic-support/learning-center>

- **Writing Center** (tutoring and writing workshops)

Room 126, Conklin Hall

(973) 353-5847

nwc@rutgers.edu