GEOSCIENCES 5535-5536/CHEMISTRY 5525-5526
X-RAY CRYSTALLOGRAPHY

I. CATALOG DESCRIPTION

5535-36 X-RAY CRYSTALLOGRAPHY

5535: Provides a thorough grounding in the principles of the crystalline state including lattices and symmetry, leading to the formal description of structures and surfaces and the interpretation of published crystallographic data. 5536: Covers methods of single-crystal and powder X-ray diffraction for the determination of the atomic arrangement of atoms within crystalline materials.

Pre: 3504 or CHEM 3615. (3H, 3C)

II. MEASURABLE LEARNING OBJECTIVES

On successful completion of 5535, students will be able to:

- Describe the geometrical concepts of the crystalline state.
- Interpret crystallographic symmetry including point, plane and space symmetry groups and the associated technical notation.
- Perform calculations to determine geometrical data such as bond lengths and angles from crystallographic data.
- Describe in technical terms crystallographic structures, planes and surfaces.
- Interpret and assess published crystallographic data on structures.

On successful completion of 5536, students will be able to:

- Perform single-crystal X-ray diffraction measurements.
- Analyze single-crystal diffraction data to determine unit-cell parameters and symmetry of the crystalline sample.
- Solve and refine the structures of crystalline solids from single-crystal diffraction X-ray data.
- Perform powder X-ray diffraction measurements.
- Analyze powder diffraction data to identify the phases present in the sample, and to perform Rietveld and LeBail refinement to determine the structures of the phases.
- Critically assess the results of structure solution and refinement from diffraction data.

III. BACKGROUND

The physical and chemical properties of molecules, minerals and other materials are determined by the geometrical arrangement of the atoms within the molecule or material. The theory behind these geometrical principles is covered in 5535. Whereas partial information on atomic structure can be often be inferred from a wide variety of spectroscopic or similar techniques, the only way to unambiguously determine the arrangements of atoms within molecules, minerals or other materials is

to crystallize the material and perform a diffraction experiment on the crystals with X-rays or neutrons. Understanding and interpreting crystallographic data is therefore an essential skill for many chemists, geologists, biologists and materials scientists and will be taught in 5535. Single-crystal X-ray diffraction is used by many research students and groups to characterize new compounds and minerals. In the form of powder diffraction, X-ray diffraction provides the primary means of phase identification through reference to a standard "finger-print" file of tens of thousands of inorganic and organic compounds. If several phases are present, the method can be used to quantitatively analyze the phase proportions. The methods of collecting and analyzing X-ray diffraction data from both single crystals and powders will be taught in 5536. The technical skills that will be provided to students by this course sequence extend to a number of active research areas within the University.

This sequence will be taught at the graduate level because students require basic understanding of undergraduate-level concepts of crystalline materials (atoms, bonding and periodicity) such as those provided by courses in mineralogy, materials science or chemistry (inorganic and organic). However, qualified senior undergraduates may take the course with instructor permission. Requirements for graduate and undergraduate students will be the same.

IV. PREREQUISITES AND COREQUISITES

Either 3504 or CHEM 3615 (or their equivalents for graduate students) is a prerequisite because the proposed courses assume an undergraduate level of understanding of the concepts of atoms, molecules, and the their packing and bonding to form crystal structures, as well as the mathematical tools and concepts used in these courses.

V. TEXTS AND SPECIAL TEACHING AIDS

Th. Hahn (Editor) INTERNATIONAL TABLES FOR CRYSTALLOGRAPHY, VOLUME A, TEACHING EDITION. Dordrecht, Netherlands, Kluwer Academic Publishers, 2002, iv, 160 pp.

W. Clegg, A.J. Blake, R.O. Gould and P. Main, CRYSTAL STRUCTURE ANALYSIS – PRINCIPLES AND PRACTICE. Chester, United Kingdom, International Union of Crystallography, 2001, vii, 280 pp.

Other readings in the course will be assigned from key papers in scientific journals and special publications, probably comprising roughly 100 published pages. In 5536 extensive use will be made of in-house teaching manuals, commercial instruments and software.

VI. SYLLABUS:

5535	Percent of Course
1. Lattices, periodicity, unit-cells, vectors, basis sets	15
2. Atomic coordinates, inter-atomic distances and angles, metric tensor	15
3. Planes, Miller indices and reciprocal space.	10
4. Transformations: rotations, basis change, change of cell size.	10
5. Symmetry and crystallographic point groups	15
6. Plane groups, Bravais lattices	10
7. Space groups, generators, interpretation of International Tables	15
8. Crystalline structures	10
Total	100

5536	
Percent of Course	
1. The production and properties of X-rays	10
2. Principles of X-ray diffraction	15
3. Single-crystal diffractometry.	10
4. Space group determination	10
5. Structure solution from diffraction data.	10
6. Structure refinement from single-crystal diffraction data	15
7. Principles of X-ray powder diffractometry	10
8. Interpretation of X-ray powder diffraction data	10
9. LeBail and Rietveld refinement of powder diffraction data	10
Total	100