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New Course Request **Indiana University** IUB Campus

Check Appropriate Boxes: Undergraduate credit ☐ Graduate credit ☒ X Professional credit ☐ 82

1. School/Division COAS/Chemistry Dept. 2. Academic Subject Code CHEM
3. Course Number C631 (must be cleared with University Enrollment Services) 4. Instructor Maren Pink
5. Course Title Chemical Crystallography

   Recommended Abbreviation (Optional) _____
   (Limited to 32 Characters including spaces)
6. First time this course is to be offered (Semester/Year): Fall 2008
7. Credit Hours: Fixed at 3 or Variable from _____ to _____
8. Is this course to be graded S-F (only)? Yes _____ No X
9. Is variable title approval being requested? Yes _____ No X
10. Course description (not to exceed 50 words) for Bulletin publication: General understanding and hands-on laboratory experience in crystallography as anana lytical method. Topics will consist of theory on physics and mathematical concepts used in crystallography, the relation of physical and chemical properties to structure data, common databases, utilization of appropriate software for data work-up, solution, refinement, and visualization structures.
11. Lecture Contact Hours: Fixed at 3 or Variable from _____ to _____
12. Non-Lecture Contact Hours: Fixed at 0 or Variable from _____ to _____
13. Estimated enrollment: 10 of which 100% percent are expected to be graduate students.
14. Frequency of scheduling: _____ Will this course be required for majors? no
15. Justification for new course: Method isnot taught, however it is a routine analysis for inorganic chemists and material scientist.
16. Are the necessary reading materials currently available in the appropriate library? Yes.
17. Please append a complete outline of the proposed course, and indicate instructor (if known), textbooks, and other materials.
18. If this course overlaps with existing courses, please explain with which courses it overlaps and whether this overlap is necessary, desirable, or unimportant.
19. A copy of every new course proposal must be submitted to departments, schools, or divisions in which there may be overlap of the new course with existing courses or areas of strong concern, with instructions that they send comments directly to the originating Curriculum Committee. Please append a list of departments, schools, or divisions thus consulted.

Submitted by:

Jan P Rolly Date 1/4/07
Department Chairman/Division Director

Approved by:

Date 1/15/08
Dean

_____ Date _____
Dean of Graduate School (when required)

_____ Date _____
Chancellor/Vice-President

_____ Date _____
University Enrollment Services

After School/Division approval, forward the last copy (without attachments) to University Enrollment Services for initial processing, and the remaining four copies and attachments to the Campus Chancellor or Vice-President.

UPS 724

University Enrollment Services Final—White; Chancellor/Vice-President—Blue; School/Division—Yellow; Department/Division—Pink; University Enrollment Services Advance—White

Chemical Crystallography

Objective

Crystal structure analysis as analytical method and tool for strategic synthesis. Understanding of physical and chemical properties from structure data for both molecular (metal-organic, organic) and extended (inorganic, polymeric, materials) structures. Relation of crystallographic analysis and other analytical methods; limits of the method, alternatives.

Content

- Radiation safety (ALARA, Distance-Shielding-Time).
- X-rays, their generation. X-ray spectroscopy vs. diffraction.
- Other diffraction methods (electron, neutron).
- Crystallization: Methods and goals.
- Solid state and crystalline materials.
- Symmetry (Plane and space groups).
- Crystal lattice. Symmetry and systematic absences.
- Theoretical concepts of crystal structure analysis: Laue cones and Bragg equations.
- Reciprocal space, reciprocal lattice and Ewald sphere.
- Atom form factors and structure factors.
- Collection of diffraction data, Corrections (Lorentz, Polarization, Absorption).
- Fourier series, Electron density function, Patterson function.
- Phase problem and structure solution.
- Patterson and heavy atoms methods.
- Isomorphous replacement.
- Direct methods.
- Structure refinement (difference maps, least squares, extinction, thermal displacement parameters),
- Structure data interpretation (R-values, esd's, thermal displacement, packing, weak interactions). Difficult structures (disorder, chiral structures, missed symmetry, pseudo-symmetry, disorder, twinning (merohedral, pseudomerohedral (reticular), non-merohedral), phase transitions, modulation, aperiodic crystals).
- Experimental methods of crystal structure characterization: powder (transmission vs. reflection) and single crystal methods (film methods: rotation, precession, Weisenberg; four-circle; area detectors).
- Data bases (CSD, PDB, ICSD, PDF).
- Limits of the method. Common pitfalls.
- New developments (e.g., new solution methods: charge flipping, new experiments: Laue and time resolved experiments).

Requirements: math, physics, general chemistry, inorganic chemistry

Exams & Grading: Midterm 30%, Final 30%, Individual project 40%
90-100% A 80-89% B 70-79% C 60-69% D

Assistance: one AI for lab, homework, exams

Required time: one semester with one 1.5 h or two 45 min lessons and one 2 hour lab session per week

Credit hrs: 3

Recommended books:

Werner Massa: Crystal structure determination
George H. Stout and Lyle H. Jensen: X-ray determination – A Practical Guide
Ulrich Müller: Inorganic Structural Chemistry
Jack Dunitz: X-ray Analysis and structure of Organic Molecules
Carmelo Giacovazzo: Fundamental of Crystallography
International Tables of Crystallography (available in the lab but not for check-out)
Manuals

Laboratory Schedule: See below. Includes time on the diffractometers and an the computers allocated for the class

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Laboratory Schedule
Programs that will be used are commercial and IU has site licenses (Bruker, Xseed, etc) or freely available (SHELX, wingx, crystals).

0) Radiation safety
- Read guide and take exam

1) Preparing pins for goniometer heads
- Pull glass fibers
- Prepare glass rods, capillaries, or loops

2) Selecting, cutting, and mounting crystals
- Select (if appropriate cut) crystals of a known sample and mount on rod, into capillary or loop, using a microscope with polarizing filter
- Literature and web pages on crystallizing single crystals suitable for X-ray crystallography
- Special mounting techniques (low temperature transfers, mounting under inert gas etc.)

3) Crystal indexing - Mount a crystal and align goniometer head
- Take matrix frames and index crystal, refine matrix
- Index rotational (non-merohedral) twin

4) Data collection
- Set up data collection
- Face index for numerical absorption correction

5) Data reduction
- Set up integration

6) Data reduction
- Evaluate integration
- Correct for absorption (SADABS and Face index)

7) Solution and refinement of an easy structure
- Solve and refine a known structure
- Patterson versus direct methods

8) Solution and refinement of a disordered structure
- Solve and refine a known structure
- Application of restraints and constraints

9) Refinement and reporting of structures
- Final refinement
- CIF format
- Creating a report and tables

- Visualization programs

10) Databases

-CSD, (ICSD, PDB)

11) Evaluating structures

- PLATON
- Various web tools (CIF-check)

12) Evaluating structures

- Evaluate a structure the from literature

13) Final project

- Mount crystal of an unknown compound, index, collect data, solve, and refine structure and create CIF and publication for Acta Cryst. C or E

If time permits

14) Other methods: Powder diffraction

- Preparing powder, mounting options for BRUKER and SCINTAG
- Collect and interpret data