

Ab initio structure determination with powder diffraction data

Theory and hands-on short course

Course objective:

The course objective is to give a crystallographic and mathematical background, strategies of *ab initio* methods for structure solution using powder diffraction data.

Course description:

The determination of a crystal structure with powder diffraction data is not-routine process and involved several sequential steps: data collection, indexing, space group selection, solution of phase problem and structure refinement. Every step of this process has directed toward finding one approximately true solution. There will be hundreds, even thousands almost correct (in fact, wrong) solutions. The art of structure determination with powder data is the ability to see the correct solution, when it comes across, among many “OK-looking” solutions. Most of problem arises due to the three-dimensional intensity data is severely distorted and presented as a one dimensional data. This is similar to the dismantling of complicated three dimensional objects (“bridge”) into pile of fragments (small and big) and then guessing and rebuilding of the original 3D object. The fragments and other available data have many hints and they require an experienced eyes, steady hands and tools to accomplish of rebuilding the original 3D object. Teaching of tools, their usage, sharpening of skills along with required theoretical knowledge altogether is objective of this course.

Theory:

Part1

- Data collection and reduction and formatting, aberrations on Bragg-Brentano data, preferred orientation, absorption contrast, extinction corrections, particle statistics
- Unit cell, crystal symmetry, space groups, Bravais lattices, atom positions, thermal parameters and cell transformations
- Powder diffraction, Miller indexes, diffracted intensities, atomic scattering factors, anomalous dispersion
- Powder data indexing, systematic absences, space group determination.

Part 2

- Extraction of structure factor amplitudes. Integrated intensities vs. peak shape function. Profile models used for various x-ray laboratory, synchrotron sources, neutron optical configurations
- Phase problem: direct and Patterson methods. Structure recycling, Fourier synthesis, difference Fourier maps. Completing of the structure.
- Rietveld structure refinement. Hints on refinement strategy: what to refine first, early, or later. Diagnostic features of the difference curve.

Practical:

- Data file reading, formatting, manual and automatic phase identification, Search-Match software, presence of amorphous components
- Pattern indexation, space group determination
- Structure solution
- Structure refinement
- Real time demonstration of various software packages.

Course fee: Academic and Government: \$2000, Industry: \$3000, which includes all course materials, breakfast and lunch on days 1-4 and course dinner. All participants will be supplied with notebook computers with installed necessary software, database, and x-ray diffraction data files of samples of various origins for quantification and tools for learning. The course consists of series of lectures and exercises followed by practical sessions on PCs

Course duration: 4 days (two days theory with exercises and two days practical work)