UCLouvain

lchm2251

2020

Structural chemistry by diffraction methods

Due to the COVID-19 crisis, the information below is subject to change, in particular that concerning the teaching mode (presential, distance or in a comodal or hybrid format).

3 credits	22.5 h + 7.5 h	Q1

Teacher(s)	Filinchuk Yaroslav ;
Language :	English
Place of the course	Louvain-la-Neuve
Main themes	1. Introduction. Refreshing the basic knowledge of crystallography: symmetry & principles of diffraction. Phase problem 2. Single crystal diffraction experiment: geometries, diffractometers and detectors, resolution 3. Powder diffraction experiment. Experimental geometries, instruments. Angular resolution. Complementarity of techniques. Powder vs single-crystal diffraction. Possibilities and limits of different diffraction techniques. 4. Systematic absences, space group determination. Reconstruction of reciprocal space sections from single crystal data. Indexing - a challenge for powder diffraction. 5. Modern structure solution methods: charge flipping & direct space methods 6. Classical structure solution methods: Patterson and direct methods, molecular replacement, isomorphous replacement, use of the anomalous dispersion, MAD (multi-wavelength anomalous dispersion) and SAD (single-wavelength anomalous dispersion) methods 7. Completing structure solution: difference Fourier maps, structure refinement, constraints and restraints 8. Last touch: absolute structure, validation of results. Problems: defects, twinning, disorder. Diffuse scattering 9. Quality of the data, interpretation of results. Publishing the results in a thesis or a publication. Databases, Pearson symbol, Wyckoff sequence, structure type. 10. Description of a structure, structural chemistry. Identifying bonding schemes. 11. Going beyond a structure.Structural evolution and reactivity under non-ambient conditions: with time, temperature, hydrostatic or gas pressure. Large facilities, writing a proposal
Aims	1 - theoretical and experimental methods of X-ray and neutron diffraction - determination of crystal structure from single-crystal and powder data - ability to interpret structural information in terms of bonding & structural chemistry knowledge - - - - *The contribution of this Teaching Unit to the development and command of the skills and learning outcomes of the programme(s) can be accessed at the end of this sheet, in the section entitled "Programmes/courses offering this Teaching Unit".*
Evaluation methods	**Due to the COVID-19 crisis, the information in this section is particularly likely to change.** Examination involving one theoretical question, one computer exercise and an explanation/evaluation of a crystal structure report.
Teaching methods	**Due to the COVID-19 crisis, the information in this section is particularly likely to change.** Lectures will be given using PowerPoint slides with an extensive use of web-based applications, crystallographic software, databases etc. A number of problems will be solved on a computer during the classes and as a part of exercises.
Content	1. Introduction. Refreshing the basic knowledge of crystallography: symmetry & principles of diffraction. Phase problem 2. Single crystal diffraction experiment: geometries, diffractometers and detectors, resolution 3. Powder diffraction experiment. Experimental geometries, instruments. Angular resolution. Complementarity of techniques. Powder *vs* single-crystal diffraction. Possibilities and limits of different diffraction techniques. 4. Indexing - a challenge for powder diffraction. Reconstruction of reciprocal space sections from single crystal data. Systematic absences, space group determination 5. Structure solution: Patterson and direct methods, molecular replacement, isomorphous replacement, use of the anomalous dispersion, MAD (multi-wavelength anomalous dispersion) and SAD (single-wavelength anomalous dispersion) methods 6. Modern structure solution methods: charge flipping & direct space methods 7. Structure refinement, constraints and restraints, absolute structure, validation of the results 8. Problems: defects, twinning, disorder. Diffuse scattering

	9. Quality of the data, interpretation of results. Publishing the results in a thesis or a publication 10. Description of a structure, structural chemistry. Databases, Pearson symbol, Wyckoff sequence, structure type. Identifying bonding schemes 11. Going beyond a structure. Structural evolution and reactivity under non-ambient conditions: with time, temperature, hydrostatic or gas pressure. Sequential refinement. Large facilities, writing a proposal
Inline resources	http://www.ruppweb.org/Xray/101index.html - short interactive course with an emphasis on macromolecular crystallography.
Bibliography	1. C. Giacovazzo, Ed.,Fundamentals of crystallography (IUCr Texts on Crystallography, Oxford University Press, 2002). 2. Y. Pecharsky, P. Zavalij, Fundamentals of powder diffraction and structural characterization of materials (Springer, second edition, 2009). 3. W.-K. Li, G.-D. Zhou, T. Mak, Advanced structural inorganic chemistry (IUCr Texts on Crystallography, Oxford University Press, 2008). 4. R. Tilley, Crystals and crystal structures (Wiley, 2006).
Other infos	**Exercises:** 1. Crystal structure models: NaCl, CsCl, diamond, graphite, CaCO3. Working with International Tables for Crystallography volume A: space groups, special positions. Calculating a powder pattern (PowderCell, Mercury, Diamond) 2. Indexing (CrysAlis, Dicvol), space group determination (CrysAlis, ChekCell), profile matching (Fullprof) 3. Structure solution by charge flipping (Platon), global optimization (FOX) 4. Structure solution by direct methods, refinement on single-crystal (Shelxs & Shelxl; WinGX) and powder (Fullprof) data
Faculty or entity in charge	CHIM

Programmes containing this learning unit (UE)				
Program title	Acronym	Credits	Prerequisite	Aims
Master [120] in Chemistry	CHIM2M	3		🔍