National School on Neutron and X-ray Scattering

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Single Crystal Crystallography

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The purpose of the lecture is to introduce the basic concepts of crystallography and help you get started in the use of scattering techniques, from both X-ray and neutron sources, to study the structure of single crystal materials. In the handout you will find an outline of the lecture, a list of online resources that I think would be useful and supplementary to the lecture class. I have also added a list of programs that are commonly used in small molecule crystallography, lists of text books and references in crystallography, and a list of existing and future neutron single crystal diffractmeters.

Lecture Outline

Crystal Lattice

Crystallographic Planes and Miller Indices

The Reciprocal Lattice

Diffractions of X-rays and Neutrons

Diffraction Conditions

Structure Factor

Diffraction Intensities

Crystal Symmetry

Structure Refinement

Validation and Presentation of Results

Online Resources in Crystallography [not intend to be a complete list]

Online Dictionary of Crystallography

Maintained by the *Commission for Crystallographic Nomenclature of the International Union of Crystallography*

Website: http://reference.iucr.org/dictionary/Main_Page

e-Crystallography

It is an interactive course, freely available on Internet for the presentation of basic crystallography concepts.

Laboratoire de Cristallographie, Swiss Federal Institute of technology in Lausanne

Website: http://escher.epfl.ch/eCrystallography/

Crystallography 101

An Introductory course by Bernhard Rupp

Website: http://www.ruppweb.org/Xray/101index.html

Symmetry and Space Group Tutorial

Knowledge of space group is essential in all aspects of structural science. The tutorial contains five modules, arranged by crystal class; a sixth module covers special topics.

Jerry P. Jasinski and Bruce M. Foxman, *Department of Chemistry, Brandeis University,*

Website: http://people.brandeis.edu/~foxman1/teaching/indexpr.html

Tricks of the Trade: Interpretation of Structural Results (ACA2007 Meeting)

Talks in the session discussed a wide variety of topics and methods in interpretation and validation of structural results.

Website: http://www.pns.anl.gov/instruments/scd/subscd/scd.shtml

Bilbao Crystallographic Server

It is a web site with crystallographic programs and databases accessible via Internet. The Retrieval Tools are very useful when working with space groups.

Website: http://www.cryst.ehu.es/

The *Condensed Matter Physics Dept. of the University of the Basque Country*

Neutron Scattering Web

Collective Lists of neutron facilities, references and software programs.

Website: http://www.neutron.anl.gov/index.html

Books in Crystallography

Werner Massa, R.O. Gould, *Crystal Structure Determination*, Springer; 2nd *ed*. (**2004**)

Peter Muller, Regine Herbst-Irmer, Anthony Spek, and Thomas Schneider, *Crystal Structure Refinement: A Crystallographer's Guide to SHELXL* (*International Union of Crystallography Texts on Crystallography*), Oxford University Press; (**2006**)

Chick C. Wilson, *Single Crystal Neutron Diffraction from Molecular Materials*, World Scientific Publishing Company; (**2000**)

Alexander McPherson, *Introduction to Macromolecular Crystallography*, Wiley-Liss; (**2002**)

Duncan E. McRee, *Practical Protein Crystallography*, Academic Press; 2nd *ed*. (**1999**)

C. Giacovazzo, H.L. Monaco, G. Artioli, and D. Viterbo, *Fundamentals of Crystallography*, Oxford University Press; 2nd *ed*. (**2002**)

Uri Shmueli, *Theories and Techniques of Crystal Structure Determination*, Oxford University Press; (**2007**)

Reviews in Neutron Crystallography

Piccoli, P. M. B. ; Koetzle, T. F.; Schultz, A. J. "*Single crystal neutron diffraction for the inorganic chemist – A practical guide*" *Comments on Inorg. Chem*. **2007**, *28*, 3–38.

Schultz, A. J.; De Lurgio, P. M.; Hammonds, J. P.; Mikkelson, D. J.; Mikkelson, R. L.; Miller, M. E.; Naday, I.; Peterson, P. F.; Porter, R. R.; Worlton, T. G. "*The upgraded IPNS single crystal diffractometer" Physica B*, **2006**, *385*, 386–1059.

Teixeira, S.C. M.; Zaccai, G.; Ankner, J. *et al*. "*New sources and instrumentation for neutrons in biology*" *Chem. Phys*. **2008**, *345*, 133-151.

Niimura, N.; Bau, R. "*Neutron protein crystallography: beyond the folding structure of biological macromolecules*" *Acta Cryst. A64*, **2008**, 12-22.

Blakeley, M. P.; Langan, P.; Niimura, N.; Podjarny, A."*Neutron crystallography: opportunities, challenges, and Limitations.*" *Current Opinion in Structural Biology,* **2008**, *18*, 1–8.

Myles, D. A. A. "*Neutron protein crystallography: current status and a brighter future*" *Current Opinion in Structural Biology*, *16,* **2006**, 630-637.

Programs for Chemical Crystallography

SHELX97

Programs for crystal structure determination from single-crystal diffraction data.

Link to website: http://shelx.uni-ac.gwdg.de/SHELX/index.html

"A short history of SHELX". Sheldrick, G.M. (**2008**). *Acta Cryst. A64*, 112-122. Open Access

Link to Tutorial: http://shelx.uni-ac.gwdg.de/tutorial/english/intro.htm

GSAS

GSAS (General Structure Analysis System) is a comprehensive system for the refinement of structural models to both x-ray and neutron diffraction data.

Link to website:
http://www.ncnr.nist.gov/programs/crystallography/software/expgui/expgui_Win_readme.html

A.C. Larson and R.B. Von Dreele, "General Structure Analysis System (GSAS)", Los Alamos National Laboratory Report LAUR 86-748 (**2000**).

Link to Tutorial: http://www.ccp14.ac.uk/solution/gsas/singlextalrefine/index.html

CRYSTALS

The CRYSTALS software package for single crystal X-ray structure refinement and analysis consists of CRYSTALS, Cameron and specially recompiled versions of SIR92 and SHELXS.

Link to website: http://www.xtl.ox.ac.uk/crystals.html

Betteridge, P. W., Carruthers, J. R., Cooper, R. I., Prout, K., Watkin, D. J. (**2003**). *J. Appl. Cryst. 36*, 1487.

Link to Tutorial : http://www.ccp14.ac.uk/tutorial/crystals/index.html

JANA2006

A crystallographic program focused to solution, refinement and interpretation of difficult, especially modulated structures.

Link to website: http://www-xray.fzu.cz/jana/jana.html

Petricek,V., Dusek,M. & Palatinus,L. (**2006**). Jana2006. The crystallographic computing system. Institute of Physics, Praha, Czech Republic.

WinGX Program System

WinGX is Windows system of programs for solving, refining and analysing single crystal X-ray diffraction data for small molecules.

Link to website: http://www.chem.gla.ac.uk/~louis/software/wingx/

L. J. Farrugia, *J. Appl. Cryst.* (**1999**), *32*, 837-838.

CCP4

Collaborative Computational Project No. 4 Software for Macromolecular X-Ray Crystallography.

Link to website: http://www.ccp4.ac.uk/

CCP14

Collaborative Computational Project No. 14 in Powder and Small Molecule Single Crystal Diffraction. A collection of the best and most commonly used programs. The software located on the CCP14 site is freely available to academic users.

Link to website: http://www.ccp14.ac.uk/whatsnew.htm

Platon

Single crystal data reduction, structure analysis and CIF validation.

Link to website: http://web.mit.edu/platon_v40505/platon/docs/platon/pl000000.html

A. L. Speck. *J. Appl. Cryst.* **2003**, *36*, 7-13 Open Access

Link to Tutorial: http://www.ccp14.ac.uk/tutorial/platon/index.html

checkCIF

checkCIF reports on the consistency and integrity of crystal structure determinations reported in CIF format.

Link to website: http://checkcif.iucr.org/

ISAW

The Integrated Spectral Analysis Workbench software project (ISAW) can be used to read, manipulate, view, and save neutron scattering data.

Website: http://ftp.sns.gov/ISAW/

A tutorial for single data analysis is available at the IPNS SCD web site.

Link to Tutorial: http://www.pns.anl.gov/instruments/scd/subscd/scd.shtml

Neutron Scattering Software

Collection of software programs for neutron data analysis and visualization.

Website: http://www.neutron.anl.gov/software.html

List of Neutron Diffractometers

SNS (ORNL, USA) Single crystal diffractometers:
TOPAZ single crystal diffractometer [future instrument 2009]
SNAP spallation neutrons and pressure diffractometer
ManDi macromolecular Neutron Diffractometer [future instrument 2012]
HB-3A at HFIR (ORNL, USA) 4-circle diffractometer.
Koala at OPAL (Australia) quasi-Laue image plate diffractometer.
E5 at BENSC (Berlin, Germany) - reactor source, 4-circle diffractometer
FRM II (Germany)
RESI thermal single crystal diffractometer
HEIDI hot single crystal diffractometer
ILL (Grenoble, France) single crystal diffractometers
D3 polarised hot neutron diffractometer
D9 hot neutron 4-circle diffractometer
D10 thermal neutron 4-circle & three-axis diffractometer
D15 thermal neutron normal-beam diffractometer
D19 4-circle diffractometer with 2D PSD for large unit cells
D23 thermal neutron normal-beam diffractometer + polarised neutron option
VIVALDI thermal beam Laue diffractometer
ORIENT EXPRESS new thermal beam Laue diffractometer
LLB (France) single crystal diffractometers:
5C-1: Polarized Hot Neutron Two-Axis Spectrometer
5C-2: Hot Neutron Four-Circle Diffractometer
6T-2: Thermal Neutron Four-Circle Diffractometer with Lifting Counter
SV28 at FRJ-II Jülich (Germany) reactor source, monochromatic diffractometer.
BIX-3 at JAERI (Japan) neutron image plate monochromatic diffractometer for macromolecules.
TriCS at SINQ (Switzerland) continuous spallation source, monochromatic diffractometer.
SXD at ISIS (UK) pulsed spallation neutron time-of-flight Laue diffractometer.
SCD at IPNS (ANL, USA) pulsed spallation neutron time-of-flight Laue diffractometer.
PCS at LANSCE (LANL, USA) pulsed spallation neutron time-of-flight Laue diffractometer for macromolecules.
SCD at LANSCE (LANL, USA) pulsed spallation neutron time-of-flight Laue diffractometer.