Methods and Tutorials – Single Crystal Diffraction

Single crystal diffraction and magnetism

- Background material:

Piccoli P. M. B., Koetzle T. F., Schultz A. J., "Single crystal neutron diffraction for the inorganic chemist - A practical guide", *Comments on Inorganic Chemistry*, **28**, 3-38 (2007).

Michels-Clark T. M., Savici A. T., Lynch V. E., Wang X. P., Hoffmann C. M., "Expanding Lorentz and spectrum corrections to large volumes of reciprocal space for single-crystal time-of-flight neutron diffraction", *Journal of Applied Crystallography*, **49,** 497-506 (2016).

Sheldrick G. M., "Crystal structure refinement with SHELXL", *Acta Crystallographica Section C-Structural Chemistry*, **71**, 3-8 (2015).

Petricek V., Dusek M., Plasil J., "Crystallographic computing system Jana2006: solution and refinement of twinned structures", *Zeitschrift für Kristallographie - Crystalline Materials*, **231**, 583-599 (2016).

Collin Broholm's lecture on magnetic neutron scattering, online: http://cins.ca/docs/ss2013/lectures/Broholm.pdf

Wills A. S., "A walk through of the maths behind Bertaut's method of representational analysis of magnetic structures"

Petricek V., Henriques M. S., Dusek M., "Solution and Refinement of Magnetic Structures with Jana2006", *Acta Physica Polonica A* **130**, 848-851 (2016).

Wilson C. C., "Single crystal neutron diffraction from molecular materials", World Scientific, 2000 (book).

Squires G. L., "Introduction to the Theory of Thermal Neutron Scattering", Cambridge University Press, 2012 (book).

Lovesey S. W., "The Theory of Neutron Scattering from Condensed Matter Volume II", Oxford University Press, 1986 (book).

- Tutorials and Manuals:

Data reduction

TOPAZ Introduction

Neutron TOF Data Reduction for Modulated Crystals (TOPAZ)

Instruction videos for Single Crystal data reduction. (HB-2C data)

Manuals for Single Crystal data reduction. (HB-2C data)

Data analysis

SHELX Tutorials and Talks

SHELX for neutrons George Sheldrick

Refinement of small molecules against neutron data Xiaoping Wang

GSAS & EXPGUI GSAS/EXPGUI refinement example Xiaoping Wang

GSAS-II Single crystal structure refinement with TOF data in GSAS-II Robert Von Dreele (Argonne, 2016)

JANA2006 Installation notes Vaclav Petricek and Margarida S. Henriques

Tutorial A $CsLiSO_4$: Structure with pseudomerohedric 3-fold twinning

Tutorial B $Y(PO_3)_3$: Modulated crystal structure with crenel

Tutorial C $DyMn_6Ge_6$: A conical magnetic structure with $k1$ = (0, 0, 0) and $k2$ = (0, 0, 0.1651)

Tutorial D $K_2V_3O_8$: Solution of (3+1)-dimensional incommensurately modulated structure with twinning (TOPAZ data)

ISODISTORT Instructions Branton Campbell

ISOVIZ and ISOVIZQ Installers

Tutorial Exercises

FULLPROF

Hands-on example Single Crystal, Commensurate Huibo Cao

Hands-on example Single Crystal, Incommensurate Huibo Cao

Bilbao Crystallographic Server Hands-on demonstration Manuel Perez-Mato

- Software

  SHELX https://shelx.uni-goettingen.de/

  Program package for the determination of small and macromolecular crystal structures

  ShelXle https://www.shelxle.org/shelx/eingabe.php

  A graphical interface for SHELXL

  Olex² Crystallography Software http://www.olexsys.org/Software

  Program package to solve, refine and finish small-molecule crystal structures using an intuitive user interface

  JANA http://jana.fzu.cz/

  Crystallographic Computing System for Standard and Modulated Structures

  Structure solution and refinement of crystal / magnetic structures (CW and TOF neutron)

  GSAS & EXPGUI https://www.ncnr.nist.gov/xtal/software/downloads.html

  Software package to fit structural models to x-ray and neutron diffraction data. It can be used with both single-crystal and powder diffraction data (Rietveld analysis). EXPGUI is a Graphical user interface to GSAS

  GSAS II https://subversion.xray.aps.anl.gov/trac/pyGSAS

  Python project for determination of crystal structures using both powder and single-crystal diffraction with extensive visualization capabilities.

  FullProf https://www.ill.eu/sites/fullprof/

  Crystallographic programs *mainly* developed for Rietveld analysis of neutron (constant wavelength, time of flight, nuclear and magnetic scattering) or X-ray powder diffraction data collected at constant or variable step in scattering angle 2theta.

  Isotropy Software Suite https://stokes.byu.edu/iso/isotropy.php

  A collection of software which applies group theoretical methods to the analysis of phase transitions in crystalline solids.

  SARAh http://fermat.chem.ucl.ac.uk/spaces/willsgroup/software/

  Simulated annealing and representation analysis of magnetic structures

  SpinWaveGenie https://github.com/SpinWaveGenie/SpinWaveGenie

  Library for simplifying linear spin wave calculations of magnetic structures

  VESTA https://jp-minerals.org/vesta/en/download.html

  A three-dimensional visualization system for crystal and magnetic structure analysis

CCP14 Program repository http://ccp14.cryst.bbk.ac.uk/mirror.htm

Collection of software for single crystal and powder diffraction.

Diffuse Scattering

- Tutorials and Manuals:

  Data analysis

  rmc-discord Tutorial examples for the refinement of magnetic, occupational and displacive disorder for single crystal in two and three dimensions

  MANTID 3D-ΔPDF Usage of DeltaPDF3D algorithm

- Software

  rmc-discord https://zjmorgan.github.io/rmc-discord/

  An atomistic reverse Monte Carlo (RMC) refinement program for the analysis of diffuse scattering from disordered single crystals

  DISCUS_SUITE Diffuse program collection https://github.com/tproffen/DiffuseCode

  Diffuse scattering & defect structure simulation and refinement, including PDFs from n/X data

  Javelin https://github.com/rosswhitfield/javelin

  Python program for X-ray and neutron single crystal nuclear and magnetic diffuse scattering analysis

  YELL https://github.com/YellProgram/Yell

  Diffuse scattering interpretation using the X-ray 3D-ΔPDF refinement

  SCATTY https://joepaddison.com/software/

  Ultrafast calculation of diffuse-scattering patterns from atomistic models

Protein Crystallography

- Background material:

Blakeley M. P., Podjarny A. D., "Neutron macromolecular crystallography", *Emerging Topics in Life Science* **2**, 39-55 (2018).

Ashkar R., Bilheux H. Z., Bordallo H., Briber R., Callaway D. J. E., Cheng X.,. Chu X.-Q, Curtis J. E., Dadmun M., Fenimore P., Fushman D., Gabel F., Gupta K., Herberle F., Heinrich F., Hong L., Katsaras J., Kelman Z., Kharlampieva E., Kneller G. R., Kovalevsky A., Krueger S., Langan P., Lieberman R., Liu Y., Losche M., Lyman E., Mao Y., Marino J., Mattos C., Meilleur F., Moody P., Nickels J. D., O'Dell W. B., O'Neill H., Perez-Salas U., Peters J., Petridis L., Sokolov A. P., Stanley C., Wagner N., Weinrich M., Weiss K., Wymore T., Zhang Y., Smith J. C., "Neutron scattering in the biological sciences: progress and prospects", *Acta Crystallographica Section* D **74**, 1129-1168 (2018).

Oksanen, E.; Chen, J. C.-H.; Fisher, S. Z., "Neutron Crystallography for the Study of Hydrogen Bonds in Macromolecules", *Molecules* **22**, 596 (2017).

Fisher Z., Jackson A., Kovalevsky A., Oksanen E., Wacklin H., Chapter 1 – Biological Structures, *in Exp. Methods Phys. Sci.*, Ed. F. Fernandez-Alonso and D. Price, Elsevier, **49**, pp 1-75 (2017).

Niimura, N., Takimoto-Kamimura, M., Tanaka, I., "Neutron diffraction in studies of protein dynamics and functions Application of", *Encyclopedia of Analytical Chemistry: Applications, Theory and Instrumentation*, 1-30 (2016).

O'Dell, W. B., Bodenheimer, A. M., Meilleur, F., "Neutron protein crystallography: a complementary tool for locating hydrogens in proteins", *Archives of Biochemistry and Biophys*ics **602**, 48-60 (2016).

Golden, E. A., Vrielink, A., "Looking for hydrogen atoms: neutron crystallography provides novel insights into protein structure and function", *Australian Journal of Chemistry* **67**, 1751-1762 (2014).

Langan, P., Chen, J. C.-H., "Seeing the chemistry in biology with neutron crystallography", *Physical Chemistry Chemical Physics* **15**, 13705 (2013).

Niimura, N. and Podjarny, A., "Neutron protein crystallography", Oxford University Press. 250 pp. (2011)

Glusker J., Kovalevsky A., Hanson L., Fisher Z., Mustyakimov M., Mason S., Forsyth T., Langan P., "Using Neutron Protein Crystallography to Understand Enzyme Mechanism", *Acta Crystallographica Section* D **66**, 1257-1261 (2010).

Adams, P. D.; Mustyakimov, M.; Afonine, P. V.; Langan, P., "Generalized X-ray and Neutron Crystallographic Analysis: More Accurate and Complete Structures for Biological Macromolecules", *Acta Crystallographica Section* D ***65***, 567-573 (2009).

Blakeley, M. P., "Neutron macromolecular crystallography", *Crystallography Reviews* **15**, 157-218 (2009).

Langan P., Fisher Z., Kovalevsky A., Mustyakimov M., Valone A. S., Unkefer C., Waltman M. J., Coates L., Adams P. D., Afonine P. V., Bennett B., Dealwis C., Schoenborn B. P., "Protein Structures by Spallation Neutron Crystallography", *Journal of Synchrotron Radiation* **15,** 215-218 (2008).

Niimura, N., Bau, R., "Neutron protein crystallography: beyond the folding structure of biological macromolecules", Acta *Crystallographica Section* A **64**, 12-22 (2008).

Blakeley, M. P., Langan, P., Niimura, N., Podjarny, A. "Neutron crystallography: opportunities, challenges, and limitations", *Current Opinion in Structural Biol*ogy **18**, 593-600 (2008).

- Tutorials and Manuals:

  Data reduction

  MaNDi https://github.com/neutrons/mandi_autoreduction_scripts

  Data analysis

  CCP4 CCP4 YouTube Channels

  Phenix Videos from Phenix workshops

- Software

  CCP4 https://www.ccp4.ac.uk/

  Software for Macromolecular X-Ray Crystallography

  Phenix https://phenix-online.org/

  Software suite for the automated determination of molecular structures using X-ray crystallography and other methods