

## 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

**JANA 2006** [Installation notes](#) Vaclav Petricek and Margarida S. Henriques

[Tutorial A](#) CsLiSO<sub>4</sub>: Structure with pseudomorphed 3-fold twinning

[Tutorial B](#) Y(PO<sub>3</sub>)<sub>3</sub>: Modulated crystal structure with crenel

[Tutorial C](#) DyMn<sub>6</sub>Ge<sub>6</sub>: A conical magnetic structure with  $k_1 = (0, 0, 0)$  and  $k_2 = (0, 0, 0.1651)$

[Tutorial D](#) K<sub>2</sub>V<sub>3</sub>O<sub>8</sub>: Solution of (3+1)-dimensional incommensurately modulated structure with twinning (TOPAZ data)

**JANA 2020** [Installation notes](#), [Video](#) Vaclav Petricek

Tutorial example A1 for twinned crystal structure and subgroup transformation, [Step-by-step instruction](#), [Data](#), [Video](#) Vaclav Petricek

Tutorial example A2 for disorder, split atom positions and restraints, [Step-by-step instruction](#), [Data](#), [Video](#) Vaclav Petricek

Tutorial example B for solution and refinement of simple (3+1)*d* structures, [Step-by-step instruction](#), [Data](#), [Video](#) Vaclav Petricek

Tutorial example C1 for commensurate magnetic structure, [Step-by-step instruction](#), [Data](#), [Video](#) Margarida Henriques

Tutorial example C2 for incommensurate magnetic structure, [Step-by-step instruction](#), [Data](#), [Video](#) Margarida Henriques

Tutorial example for bixbyite (TOPAZ data), [Step-by-step instruction](#), [Data](#) Xiaoping Wang

Tutorial example for commensurate magnetic structure  $\text{Tb}_2\text{Ir}_3\text{Ga}_9$  (TOPAZ data), [Step-by-step instruction](#), [Data](#) Feng Ye

**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.

**Olex<sup>2</sup> 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)

**JANA2020** <http://jana-login.fzu.cz>

**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  $2\theta$ .

**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*

**rnc-discord** [Tutorial examples for the refinement of magnetic, occupational and displacive disorder for single crystal in two and three dimensions](#)

Tutorial example for magnetic diffuse scattering (bixbyite, CORELLI data), [Data](#), [Video](#)  
Zachary Morgan

**MANTID 3D- $\Delta$ PDF** [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.

Installation of software package, <https://zjmorgan.github.io/rmc-discord/docs/installation>, [Video](#)  
Zachary Morgan

**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- $\Delta$ PDF 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 Biophysics* **602**, 48-60 (2016).

Golden E. A., Vrieling 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 Biology* **18**, 593-600 (2008).

- Tutorials and Manuals:

*Data reduction*

**MaNDi** [https://github.com/neutrons/mandi\\_autoreduction\\_scripts](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.