ADT3D short work-flow

Automated Diffraction Tomography (ADT) technique[1-4] is based on electron diffraction tilt series acquired through a step-wise tilt of a selected crystal around an arbitrary (non-crystallographic) axis. The so obtained non-oriented (off-zone) diffraction patterns cannot be analyzed directly. *ADT3D* software package is able to transform a stack of 2D off-zone diffraction patterns in a 3D diffraction volume, to determine the unit cell parameters through automatic clustering and manual routines and to extract intensities that can be used for structure solution by direct methods. Moreover the 3D diffraction reconstruction can be used for identifying special features as polycrystallinity, twinning, super-structure modulations, incommensurate reflections, etc...

This tutorial is thought for leading the student through the use of *ADT3D* software package. The idea is to simulate a real data analysis, starting from a raw ADT acquisition consisting of a stack of 2D off-zone diffraction patterns (acquired from a single nano crystal by a tilt series performed in our lab) and ending with 3D reconstructed diffraction volume, cell parameters and an intensity hkl file. Together with the installation file you find four exemplary *mrc* files: $BaSO_4$, $Na_2Ti_6O_{13}$, natrolite and Hemimorphite_BeamStop. More information about the data sets are in § 5. You can start the analysis opening the BaSO4 *mrc* file and go thought the functions of the program.

More information is available within the software manual.

The following is a brief outline of the analytical steps that lead you from the uploading if a raw data stack up to the reflection intensity integration.

- Loading data. An *mrc* file containing a stack of 2D off-zone diffraction patterns is the standard input format. An *mrc* file can be created from a stack of *tif* images using *TIF2MRC* converter (not included here).
- Inspecting the 2D patterns.
- Shifting the diffraction patterns to a common center.
- Finding the tilt axis azimuth.
- Rotating the 2D patterns in order to have the tilt axis in vertical position.
- Binning the data to save memory space.
- Creating a 3D volume (that can be saved in *mrc* format).
- Visually inspecting the 3D volume.
- Finding the reflections.
- Determining the unit cell with automatic or manual routines.
- Extracting intensities and compiling a standard *hkl* file usable for structure solution.

Proposed examples

In this tutorial we propose 4 exemplary ADT data sets from relatively easy crystal structures. For all of them you have an *mrc* file containing a 2D diffraction patterns stack collected with ADT

method. All the acquisition are performed coupling ADT with precession of the beam.[3,6] All the three data sets are already binned for saving memory (see § 2.4). If you still have problem with the memory of your computer, bin them again. The tilt axis azimuth is between 40° and 50° for all the data sets except for Hemimorphite_BeamStop, where the tilt axis is between -40° and -30°. The *cif* files for the first three data sets are provided and you can use them for comparing cell parameters.

5.1 $BaSO_4$

The first example is BaSO4. This structure is relatively easy as the cell is orthorhombic primitive and contains only 5 independent data atoms. You can load the 2D diffraction patterns stack and go through the whole procedure exploring all the options. After reconstructing the 3D reciprocal space, you can easily determine the cell parameters with the automatic procedure. A *Threshold* of 250 or more is suggested for the *Find Reflection* parameters. Then you can try to refine (or spoil) your result changing manually the cell parameters (pay attention to the determinant of the cell vector matrix!). Once the cell is satisfactory, you can rotate the volume looking for the two glide plane extinction families and extract the intensities. Pay attention to your cell setting and compare it with the values reported in the *cif* file. If the setting is different are different (i.e. a, b and c are exchanged), change the cell accordingly… or take care in the space group selection when you run *SIR* or *SHELX*.

5.2 $Na_2Ti_6O_{13}$

The second example is $Na_2Ti_6O_{13}$. You don't need to go through all the options of *ADT3D* and you can run directly all the steps until the cell determination (the cell parameters have a global scale factor due to the missing fine calibration in the present test version, but angles and length ratios should be correct). For the *Find Reflection* parameters, use *Min. volume* 5, *Max. volume* 5000 and *Threshold* 250. These values are important later for a correct intensity extraction. On the contrary, for the *Cell Determination* parameters you are free to try until you get a good result. Suggestion, take care of the *cut off center* and use the full difference vector space. Also in this case you can extract an *hkl* file with the intensities that you can use later for *SIR* or *SHELX* tutorials.

5.3 Natrolite

The third example is natrolite – $Na_2Al_2Si_3O_{10}\cdot 2H_2O$. Natrolite is a more complicate case, having an orthorombic F-centered (pseudo-tetragonal) cell and a more complicate structure. For natrolite you can consider a good result just finding the correct cell. Can you distinguish between the orthorhombic and the tetragonal setting? Which margin of error do you have?

5.4 Hemimorphite_BeamStop

The forth examples comes from another inorganic sample. Unlike the other data seta, this data set was acquired using a beam stopper in order to shield the transmitted beam. The presence of the beam stopper prevents to perform an automatic pattern centering so you have to set the center by the use of a Friedel pair in any one of the slides where a pair is clearly recognizable. You have also to select *use beam stopper mode* in the *Centering routine* panel.