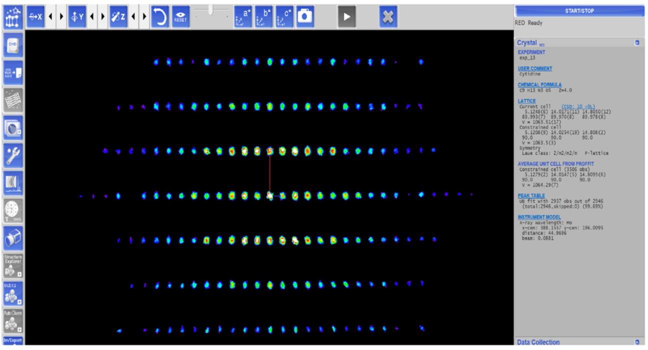
3D viewer for diffraction space

Ewald^{3D}



1. Introduction

Since the earliest experiments, X-ray crystallographers became accustomed to directly observing reciprocal space. Initial experiments were performed using area detectors with photographic film as the detection medium. While accurate measurement of diffracted intensity was not possible, observing the space around reflections was. Initially experiments used flat arrangements of photographic film though since X-ray crystallography experiments involve diffraction outward in all directions from a point, the crystal, it wasn't long before curved, cylindrical X-ray cameras were developed which more closely matched the spherical nature of diffraction experiments. With proper alignment of the crystal, this technique allowed observation of reciprocal lattice planes and lattice lines, measurement of unit cell dimensions, observation of systematic absences and observation of spurious intensities. Regardless, with photographic film glimpses of space between Bragg diffraction could be seen. With the advent of electronic area detectors and computerised measurement, it became possible to achieve even better views of reciprocal space. With modern diffractometer equipment and software, sampling and viewing reciprocal space in its entirety in three dimensions is now possible in short timescales. Ewald^{3D} is a module within CrysAlis^{Pro} which allows not only reconstruction of reciprocal space in three dimensions at the end of a data collection but also offers a live view of data as it is still being collected.

2. Importance of visualising diffraction

Often in analysing X-ray diffraction data it is useful to refer back to the images or frames collected during the experiment to diagnose anomalies or inconsistencies observed further downstream. While the Bragg reflections are usually of the most interest in structure determination, non-Bragg diffraction events such as incommensurate superstructure reflections or diffuse scatter are increasingly studied or often have diagnostic value in identifying and treating structural model problems. When problems arise during structure refinement, the cause may not be obvious from the electron density maps alone and while it is possible to detect phenomenon such as twinning or modulation, they are typically easily visible in the diffraction pattern. Indeed it is possible to misinterpret incommensurate behaviour or twinning as simple disorder when visualising Fourier maps during refinement without referring back to diffraction images. The ability to view reciprocal lattice planes is still considered a useful diagnostic tool and most current diffractometer software packages, including Rigaku's CrysAlis^{Pro}, provide a methods to do so. In the most recent version of CrysAlis^{Pro}, a new tool has been created called Ewald^{3D}, which allows the user to view the entirety of reciprocal space in a three dimensional, interactive viewer.

3. Viewing diffraction in two dimensions

Flat electronic X-ray detectors can provide a direct

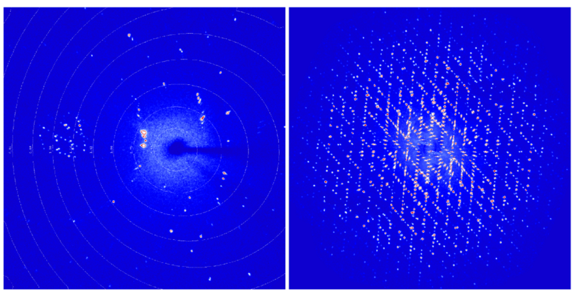


Fig. 1. Visualisation of a twin using detector images (left) vs. an unwarp image of a reciprocal lattic plane (right). While the twin is visible in both its nature is revealed more fully in the unwarped image.

view of reciprocal space, though due to being flat coupled with the nature of diffraction coming from a point, it is difficult to directly observe reciprocal lattice planes or extended rows of reflections. As such, only glimpses of a problem can often be seen this way and viewing reciprocal lattice planes would be much more informative.

4. Unwarp images

Each pixel in a detector image coincides with a point in three-dimensional reciprocal space. Provided that the goniometer angles, detector distance and crystal orientation are known, the coordinates in three-dimensional reciprocal space of a given pixel from the two-dimensional detector image can be calculated. With this information, it is therefore possible to determine contributions of pixels from the two dimensional detector images to a given plane in reciprocal space. An image of any plane can be calculated whether integer or non-integer indices are provided, but most commonly views of planes with integer values, such that Bragg diffraction spots are visible are considered most useful. These plane images are called unwarp images or simulated precession images and can show a two dimensional plane through reciprocal space with pixels generated from real measured intensities. It is important to note however that the calculation of indices from diffractometer coordinates depends on the orientation matrix and thus the unit cell and instrument model influence this. Should the unit cell be incorrect due to difficulty in unit cell finding or otherwise, calculation of useful planes through reciprocal space can become very complex. Nevertheless, unwarp images can be very useful in diagnosing issues, showing real intensities for the space in between the indexed Bragg reflections.

5. Viewing reciprocal space in three dimensions

Another means of viewing information in reciprocal space are tools such as the Ewald Explorer module in CrysAlis^{Pro} which have been available for many years. In such tools, the user must first perform a search of all diffraction images for peaks/reflections. This provides a list of coordinates of the centre of a peak along with the intensity for the whole peak thus pixel-wise information is not recorded. From this list, the peaks can be plotted in three dimensions and are typically then represented by a marker such as a dot or ball in the viewer. Such viewers usually allow peak selection, grouping and indexing.

While there is clearly value in viewing data like this in three dimensions for analysis and even indexing, only positional information with rudimentary intensity information is usually available and the method and parameters used during peak hunting such as background subtraction strongly influence visible information. Typically weaker information such as diffuse scatter is only visible with careful selection of parameters.

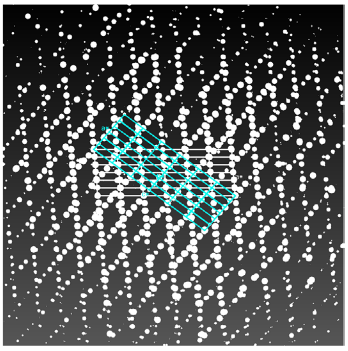


Fig. 2. Visualisation of the twinned dataset (shown in Fig. 1) in Ewald Explorer shows lattices belonging to two twin components with rudimentary intensity information.

6. Viewing diffraction in three dimensions

Both unwarp images and reciprocal lattice viewers like Ewald Explorer while providing slightly different information give useful diagnostic information. Ewald^{3D} combines the two approaches and is a new tool in CrysAlis^{Pro} for visualising the diffraction pattern in three dimensions. Ewald^{3D} provides a three-dimensional reciprocal lattice viewer similar to Ewald Explorer and adds real intensity information producing the equivalent of a three-dimensional unwarp image.

In a traditional two-dimensional unwarped image, as described previously, a unit cell must have been found and a plane of interest relative to it must first be defined by the user. Pixels from the data collection frames are then mapped onto the pixels of the plane of interest according to their coordinates. Ewald^{3D} extends this approach into three dimensions by mapping the diffraction image pixels onto 'voxels' instead of two dimensional pixels*. The intensity of each voxel is calculated by combining those of each pixel in the dataset which intersects with the coordinates of the voxel. This enables a complete three-dimensional reconstruction of reciprocal space with intensity information preserved.

As the entirety of measured reciprocal space is presented in the viewer, there is no need to specify a plane of interest therefore the user does not need to provide any input to the software. Unit cell information is also not required, only an accurate instrument model. As the instrument model is usually determined by instrument calibration, it is usually well determined, but if not, a lack of periodicity or presence of curvature of rows in the Ewald^{3D} viewer can quickly indicate there is a problem. This not only makes Ewald^{3D} easier to use than unwarp images, it significantly reduces the chance that artefacts in the diffraction pattern could be missed due to a poor choice of plane, or an incorrect unit cell.

* Where 2D space can be divided into elements called pixels with equal area, 3D space can be divided into elements called voxels with equal volume.

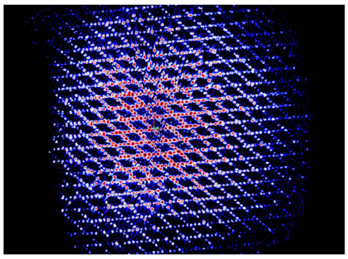


Fig. 3. A view taken from Ewald^{3D} showing the twinned data from Fig. 1 and Fig. 2 with real intensities from the detector images. Note the view is three-dimensional and can be rotated as the user desires.

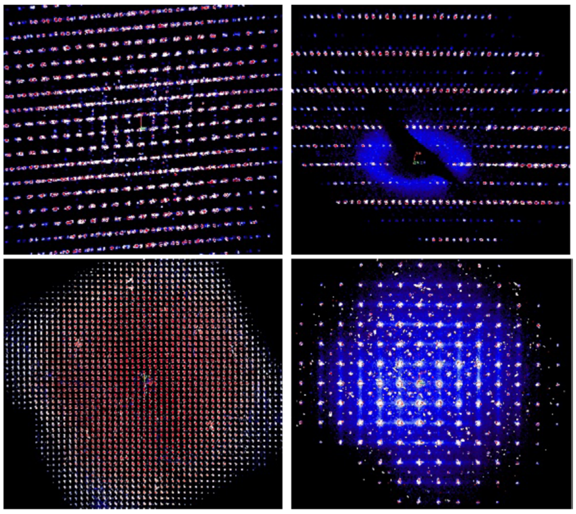


Fig. 4. Selected images showing clockwise from top left: a multi-crystal with a very weak component; an incommensurate superstructure; diffuse scatter; and a protein.

7. Ewald^{3D} features

The Ewald^{3D} viewer has some configuration options, and options for adjusting the view to best highlight diffraction features.

7.1. Fast calculation

Care has been taken to optimise Ewald^{3D}'s data extraction procedure to ensure data preparation does not take long. As an example a protein dataset containing 2000 frames takes under one minute on a mid-range laptop from 2015. Additionally, once calculated, the Ewald^{3D} dataset is stored to disk to avoid the need to perform the calculations each time, unless parameter changes are needed. The progress can also be watched within the three-dimensional viewer while calculations are ongoing for instant visualisation.

7.2. Grid size

The grid size used for partitioning space into voxels can be defined by the user. Large voxels can enhance weak features by combining more pixels into one voxel but at the cost of spatial resolution. Conversely, smaller voxels can be used to improve spatial resolution but weaker features become harder to see.

7.3. Contrast sliders

The diffraction intensities are presented according to pre-defined colour tables. The low and high limits for the colour table can be adjusted with sliders to allow enhancement or suppression of weak or strong information. The colour table can also be chosen according to the user's preference.

7.4. Plane view

Ewald^{3D} replicates the function of unwarp images by displaying only voxels which lie within a plane (up to a specified distance from it). As full three-dimensional information is available, any two-dimensional plane of interest, including those defined by non-integer values, can be selected as a subset of the full Ewald^{3D} dataset. Starting from an initial plane the user may also play through a series of planes by specifying an offset value from the initial plane. Each successive plane shown is offset from the previous by the value specified.

7.5. Live mode

Perhaps the most useful feature of Ewald^{3D} is its live mode. Thanks to the ultra-fast computation of the voxels, Ewald^{3D} is able to compute and display the three-dimensional view as diffraction images are being collected. As new diffraction images are collected by the detector the pixels are immediately read into the Ewald^{3D} viewer and displayed as voxels. The user thus has the option to watch their in-progress data collection through the Ewald^{3D} viewer instead of viewing traditional two-dimensional detector images one-by-one. New data appear as white voxels which fade to their colour table value. This view shows all of the collected data at once rather than only the most recently acquired image as with the traditional detector view.

Details such as twinning, split reflections, incommensurate superstructures all become much easier to spot during the data collection without any treatment of the data. Early diagnosis can allow the user to adapt their approach to better fit the sample while it is still on the diffractometer being measured.

8. Summary

Ewald^{3D} is an extremely useful tool which enables visualisation of reciprocal space in three-dimensions. With this functionality direct observation of non-integer diffraction events is easy and diagnosis of problems is intuitive and straightforward. The live mode allows this to be achieved during data collection for even faster diagnosis and on-the-fly adjustment of strategy for better data quality.