PETS2
Process Electron Tilt Series user manual

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INTRODUCTION

PETS2 is a computer program for processing a series of diffraction images[1]. It is intended for 3-dimensional electron diffraction data in continuous rotation mode (cRED, MicroED), stepwise mode with (PEDT) or without precession (ADT), and serial ED[2,3].

The original version of PETS was a console-based program with limited graphical capabilities. This manual describes version 2.0 with a graphical user interface and many new options.

PETS2 performs the following basic procedures: peak hunting, refinement of the position of the tilt axis, peak analysis (clustering and difference space generation), indexing, correction of the frame orientation and optimization of distortion parameters, integration of intensities, frame scaling, outlier rejection, integration of intensities for dynamical refinement, calculation of a 3D distribution of intensities in reciprocal space, and reconstructions of sections through the reciprocal space (also known as reconstructed precession images or unwarping). New features allow us to set some of those procedures as automatic tasks and merge pets2 projects.

This manual has – apart from the introduction – six parts. The first part describes how to get started with PETS2, from the installation to the creation of the initial input file. The second part is dedicated to the panels' descriptions and options. The third and fourth sections give all the keywords and commands recognized by PET2 and a list of all the output files created during the data reduction. Before the last part, we give a basic procedure model that should work for most normal/good-quality data. This manual is not a detailed guide for all cases. The procedure given in this manual is a starting point that may need adjustments depending on the data. Some data reductions of 3D ED data are already available as part of the JANA2020 cookbook (http://jana.fzu.cz). In the last section, the dependency scheme presents the interdependence between options or procedures that influence output files and the upcoming steps.

CONTACT: PETS2 is under constant development and can thus suffer from occasional bugs and weird behavior. Don't hesitate to contact us with bug reports and feature suggestions.

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A GETTING STARTED WITH PETS2

A.1. Requirements

PETS2 is currently available as a Windows executable for 64-bit operating system. The graphic card must support OpenGL v4.0 or newer.

A.2. Supported files

Only diffraction stored as TIFF files and compressed TIFFs are supported by PETS2. Any valid monochrome TIFF format is accepted (up to 32 bits).

A.3. Installation

PETS2 software and the documentation are available at http://pets.fzu.cz/ (Figure A-1)

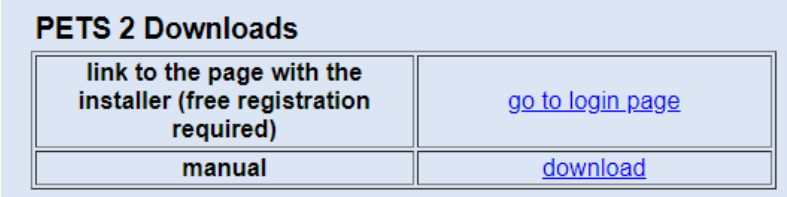


Figure A-1

The download page is http://pets-login.fzu.cz.

You need to log in to access the download page and find the last version with a brief description of the updates. An installer **PETS2_install.exe** is downloaded by hitting the link “last version”. By default, PETS2 is installed in "C:\Program Files\Pets2\pets2.exe". The PETS2 folder contains the executable and the minimal set of dlls needed to run the program.

On the downloading page, it now shows the list of all changes from the existing version to the newest one.

A.4. Creating a new project

A.4.1 Creating a new project from a tilt series of frames using PETS2 GUI

Open PETS2; File/New;
A new dialog opens (Figure A-2)

Fill out basic information: choose a project name, indicate the project directory and the directory where the series of frames are stored.

Press NEXT;

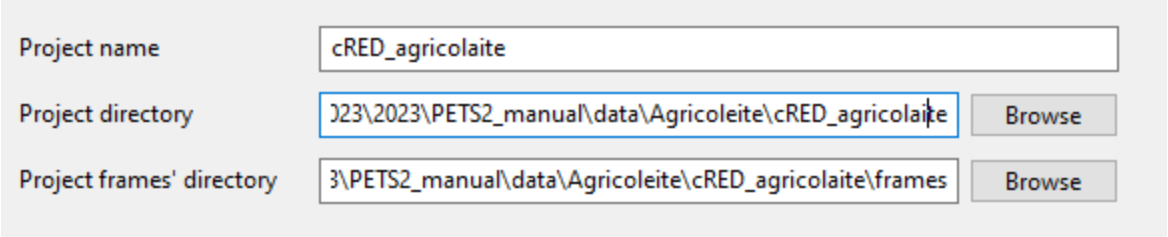


Figure A-2

In the new dialog, specify the project parameters (Figure A-3):

- The **binning**. This is not the binning used by the detector to store the frames, but the extra binning applied by PETS2. It can save time and make nicer pictures. However, it depends on the spatial resolution of the camera. In this example, we use a detector Cheetah 512 x 512 px. We prefer not to bin further the data. With another camera, CCD Veleta 2k x 2k pixels, bin 2 is a reasonable choice from our experience for most data sets. Important: All parameters in the input file given in pixels refer to the original, unbinned images. This concerns Aperpixel, center, reflections, and mask.
- The **wavelength** (120 kV: λ_{120} = 0.0335 Å, 200 kV: λ_{200} = 0.0251 Å, 300 kV: λ_{300} = 0.0196 Å).
- The **detector calibration** in reciprocal angstrom per pixel (non-binned value).
- The **geometry** of the experiment: either precession (PEDT), static frame (ADT), or continuous rotation (cRED, MicroED).
- **Tilt semiangle** for continuous rotation data or precession angle for precession-assisted data (for static frame geometry, the precession angle = 0). Note: for continuous-rotation data, make sure to insert the tilt *semiangle*, i.e. half of the angular range spanned by each frame.
- **Detector type**: Default, ASI Cheetah, Olympus SIS, Ceta-D. If your detector is not in the list, select default. For the defined detectors, some corrections or parameters will be preset (saturation limit, detector noise parameters, cross correction,..).

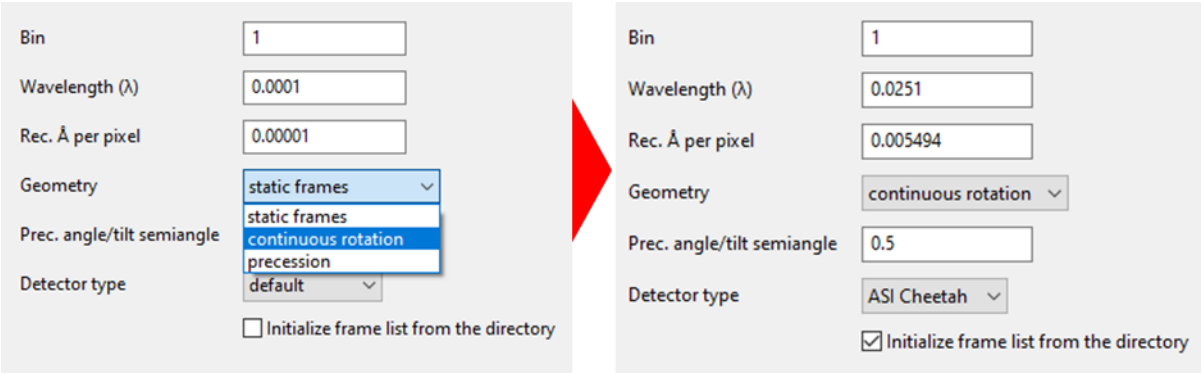


Figure A-3

Hit NEXT;

The list of frames is automatically generated (left panel Figure A-4). On the right panel of Figure A-4, the general information of the selected frame is given. At this stage, only the alpha-tilt needs to be defined to start the data reduction. The other entries will be updated during the data reduction.

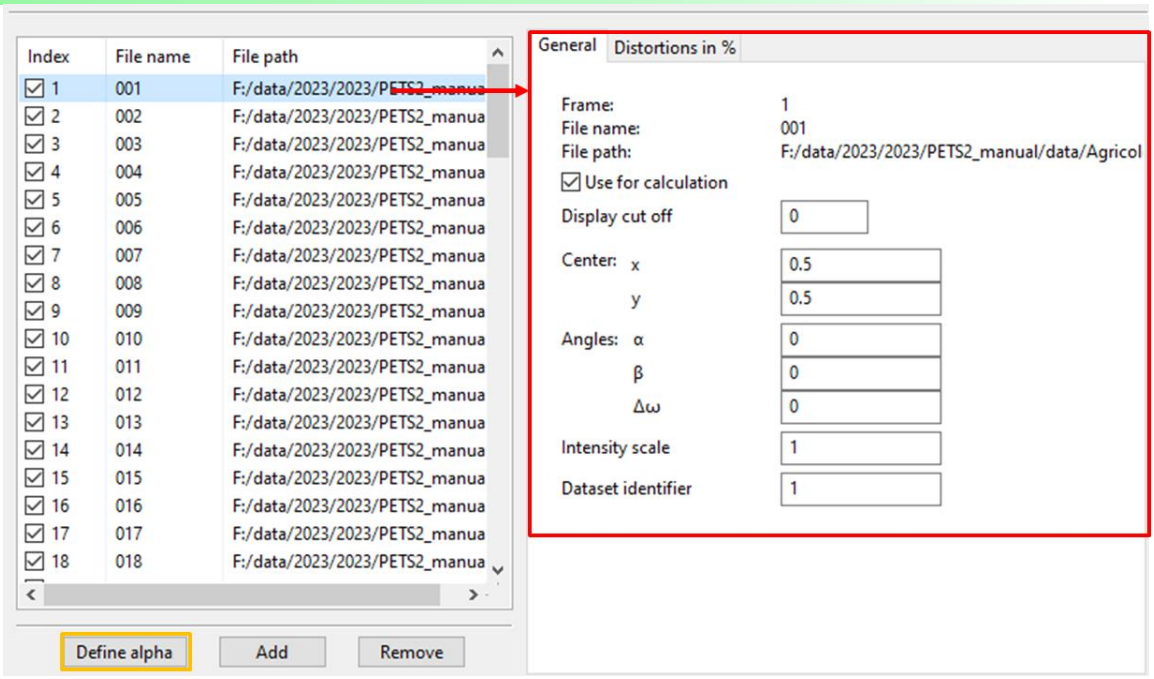


Figure A-4

To add the alpha tilt for all frames:
Press “Define alpha” (Figure A-4).
Indicate the alpha-tilt of the first frame and the tilt increment (angular step between frames);
Press OK;

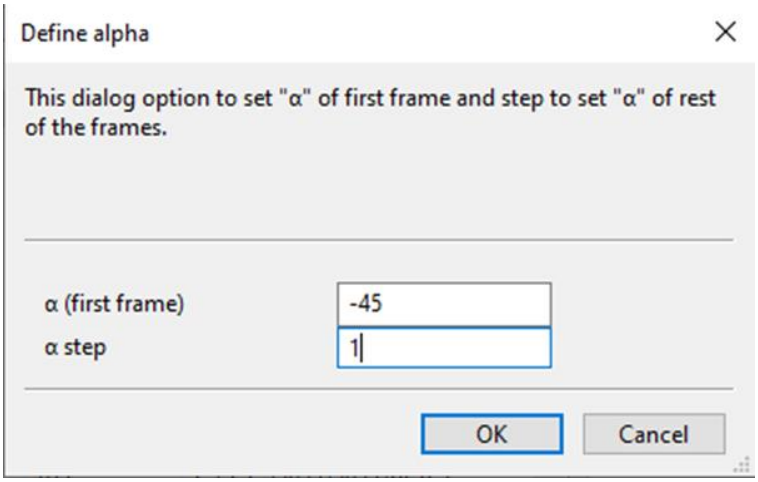


Figure A-5

The alpha angles are automatically generated for all the frames in the list.
FINISH to create the initial PTS2 file.

As soon as the files are defined, a pts2 file is created in the selected directory “jobname.pts2” as well as a sub directory “jobname_petsdata” containing all the output files which will be created during the data reduction.

Note: The data reduction will start with the ideal values of β-tilt = 0 and $\Delta\omega$ = 0. They may be later optimized. The center of the frame will be determined during the first peak search, as well as the intensity scale. The data set identifier is used in case several data sets are combined.

-Select “Add” and go to the directory to select all the TIFF files of the data set. The TIFF will be added after the current list or frames.
- To exclude a frame from the data reduction: uncheck the checkbox in front of the frame path on the left panel or uncheck the checkbox “use for calculation” for the frame to exclude in the General dialog box (right panel in Figure A-4).

-File information can be edited manually by selecting the frame of interest.

When the frame dialog box is closed, you can access it in the “Image options” panel (green panel on the right in Figure B-1) by selecting the “Frame dialog” button or going to the toolbar/Edit/Frame dialog.

A.4.2 Create a starting input PTS file manually

The data reduction can also start with an initial input file containing (at least) a minimal set of instructions. It may be convenient to generate such file directly in the data-collection software and avoid the manual creation of new project in PETS2. The following information is needed to start (Figure A-6). Once the essential information is listed, save the file as a txt file or pts file. When you open it in PETS2 and save the project, a pts2 file will be created with all the other commands/parameters.

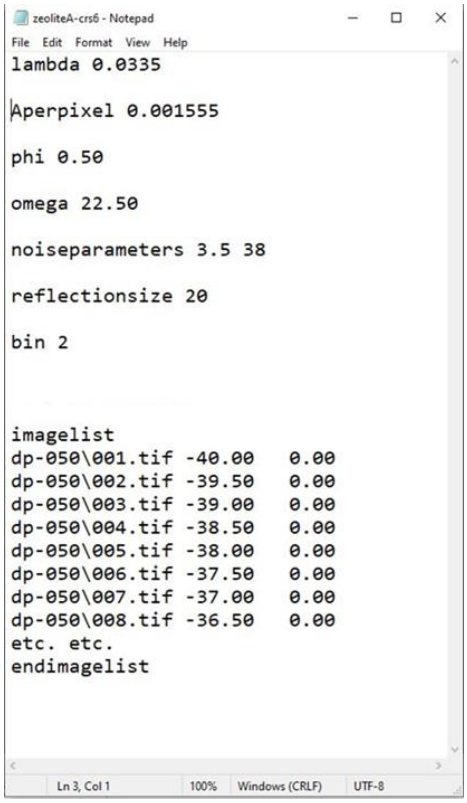


Figure A-6

The keywords are case-sensitive. Their description follows:

- **Lambda**: relativistic wavelength of the incident electrons in angstroms: 100kV = 0.0370 Å, 120 kV = 0.0335 Å, 200 kV = 0.0251 Å, and 300 kV = 0.0197 Å. This is a compulsory keyword.
- **Aperpixel**: the scale of the diffraction patterns given as the size of one pixel in reciprocal angstroms. This is a compulsory keyword.
- **phi**: semi-angle in degrees for the precession or the continuous rotation used during the data collection. Phi can be zero for the static frame experiment, of course. This is a compulsory keyword.
- **reflectionsize**: diameter of the spots on the images in pixels. Used for peak search and integration, should be large enough to encompass the strong spots and small enough to avoid overlaps of neighboring spots. This is an optional keyword. If absent, a default value of 20 is used.
- **bin**: binning of the images prior to any treatment. Saves time, makes nicer pictures, and seems to make the whole analysis somewhat more robust. In most cases it is useful and recommended to use binning, if the experimental frames are larger than 1000x1000 pixels. This is an optional keyword. If absent, a default value of 1 is used.
- **imagelist – endimagelist**: multiline keyword. Each line contains one image. The format is "name of the picture with path" alpha [beta]. The beta angle is optional, set to zero, if absent. This is a compulsory keyword.
- **omega**: orientation of the tilt-axis of the sample holder with respect to the positive horizontal axis of the image. It must be estimated either by calibration or by visual inspection of the series of images. It can also be determined and refined later in PETS. This is an optional keyword. If absent, a default value of 0 is used.
- **noiseparameters**: two parameters for the determination of σ(I). The first value is Gγ, the second is ψ. The uncertainty on each pixel γ is then calculated as σ2(p)=Gγp+ψ. For more information, see Waterman, D. & Evans, G. (2010).[4] This is an optional keyword. If absent, default values of 1 and 40 are used.

A.5. Open an older PTS project or an older PETS version

A.5.1 Open an older PTS project

In previous versions of PETS2, the output files were created in the same directory as the input file, and some files were present/absent. In newer PETS2 versions, the output files are, by default, created in a subdirectory "projectname_petsdata." When an old PTS/PTS2 file is opened for the first time in the latest version, a message appears to inform that all the output files will be moved to a subdirectory (Figure A-7).

To continue with the newest PETS2 version, you must accept.

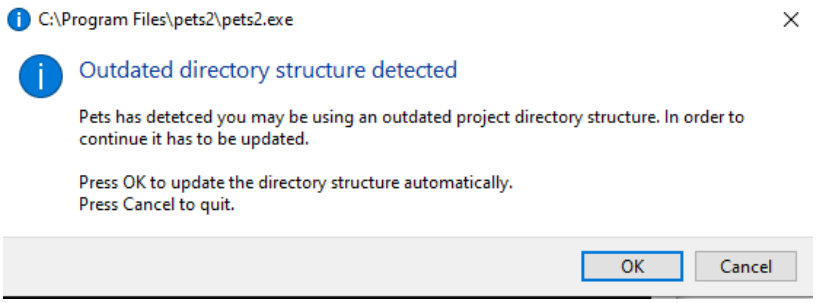


Figure A-7

A.5.2 Open a project in an older PETS version

When a pets2 project is opened, PETS2 checks is a newer version is available online.

In that case PETS2 offers to download the newest version (Yes/No/No and mute update checks for the following 30 days.)

A.6. Backups

Every time the computing thread is started, the current state of the project is saved in a file petsdata/jobname.backup; in case of a crash, the next time the project is opened, it is offered to replace the pts2 with the backup file; in case of a correct termination, the backup is deleted.

B GRAPHICAL USER INTERFACE: layout and available options

B.1. General GUI Layout

The GUI is organized with several panels as shown below (Figure B-1). More layouts are available, and the positions of the panels can be reorganized by dragging them with the mouse.

The general description of the graphical interface will be done panel by panel.

The GUI has 4 main panels + the toolbar:
-the toolbar (purple)
-the command panel (red)
-the main panel with several graphical tabs (blue)
-the options panel (green)
-the console (yellow)
-the debug window (additional console window opened in the background. This window must not be closed) (Figure B-2).

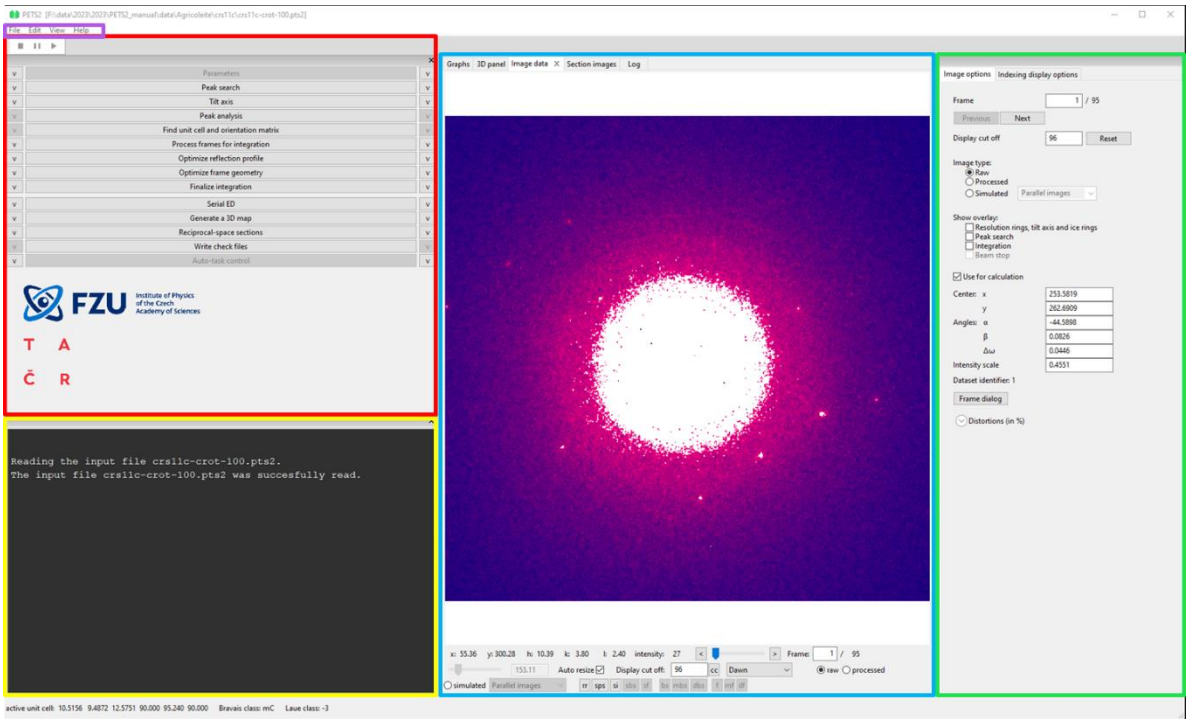


Figure B-1

Figure B-2

On the top left of the window is the name and the path of the PETS2 job and the icons File/Edit/View/Help (Figure B-3)

B.2. The toolbar

The toolbar: File/Edit/View and Help

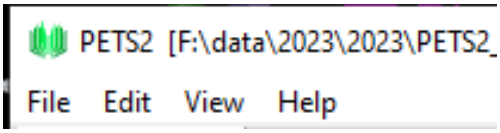


Figure B-3

B.2.1 File

Under File: New/merge/Open/Save/Save as…/Copy to…/Rename to…/Quit.

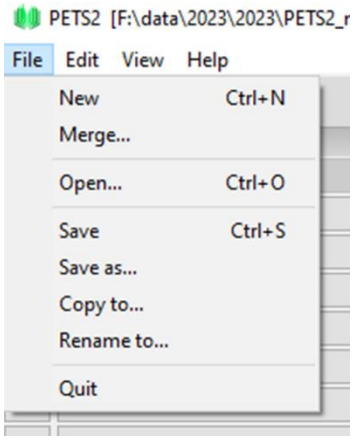


Figure B-4

- **New project**: to create a new PETS2 project (Ctrl + N). the GUI opens a new window to create a new PETS2 project from scratch (from a series of frames). The complete procedure to create a new project is explained in Section A.4.
- **Open an existing project** (pts or pts2 files) (Ctrl +O).
- **Save** the current project (Ctrl +S).
- **Save** the current project **as** …. It saves the current pts2 project under a new name as well as all the related subdirectory with the output files under the new name. The initial pts2 project is also saved and preserved.
- **Copy to**: to copy the pts2 file and the subdirectory of output files somewhere else.
- **Rename to**: to rename the current project (pts2 file and subdirectory) under a new name.
- **Merge projects**: PETS2 allows merging several pts2 projects to create one merged project containing all data. It is useful for beam-sensitive materials where only a few frames can be collected on each

crystal. Merging several data is then necessary to achieve reasonable data completeness, reduce the influence of distortions and beam damage in the unit cell accuracy.

Requirements: The project merging uses the orientation matrix to make data sets match up in the merged project. So, before merging several projects, for each data set to merge, a valid pts2 file must exist with a unit cell/orientation matrix. The unit cells of the different datasets must be close enough to allow a meaningful merging. However, there is no internal limit, and you could, theoretically merge totally different crystals with different unit cells, even though it does not make sense.

- Data sets collected with different camera lengths can be merged.
- Data with different geometries—continuous, precession, no precession, or different precession semi tilt angles— cannot be merged.

Instructions on how to merge separated projects are given in the header of the window “Merge projects” (Figure B-5).

Press “Merge” when the merge project wizard is filled out.

Pets2 will ask if “you would like to open the output project now?”. If YES, the new merged project opens. If NO, the merged project is created but you will continue working on your single initial project.

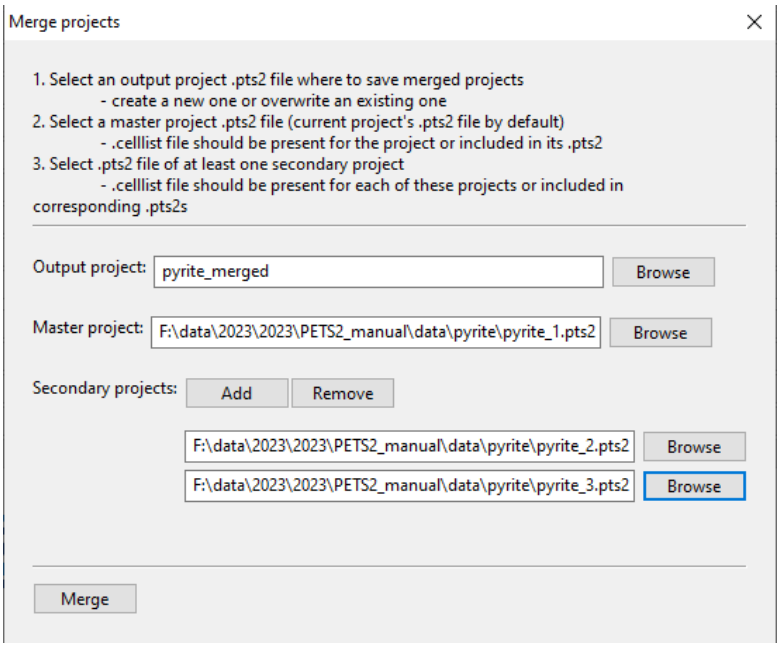


Figure B-5

B.2.2 Edit

Under Edit, two panels are accessible: Frame dialog (Figure B-6) and Settings (Figure B-7and Figure B-8)

B.2.2.a. Frame dialog panel

This panel gives all the information about the series of frames used in the project. It is also accessible from the Options panel/Image options panel. This panel will be described in detail in the Options panel sections.

In frame dialog:

- On the left panel is the list of frames with their path. When the box is checked in front of a frame, this frame is used in the data reduction.
- On the right panel, two windows show the general information (display cut off, tilt angles alpha, beta and omega, center of the frame, scale, dataset identifier for merged project) about the selected frame and the distortion parameters for this frame (magnification, elliptical distortions: amplitude + phase, and Parabolic distortions: amplitude + phase).

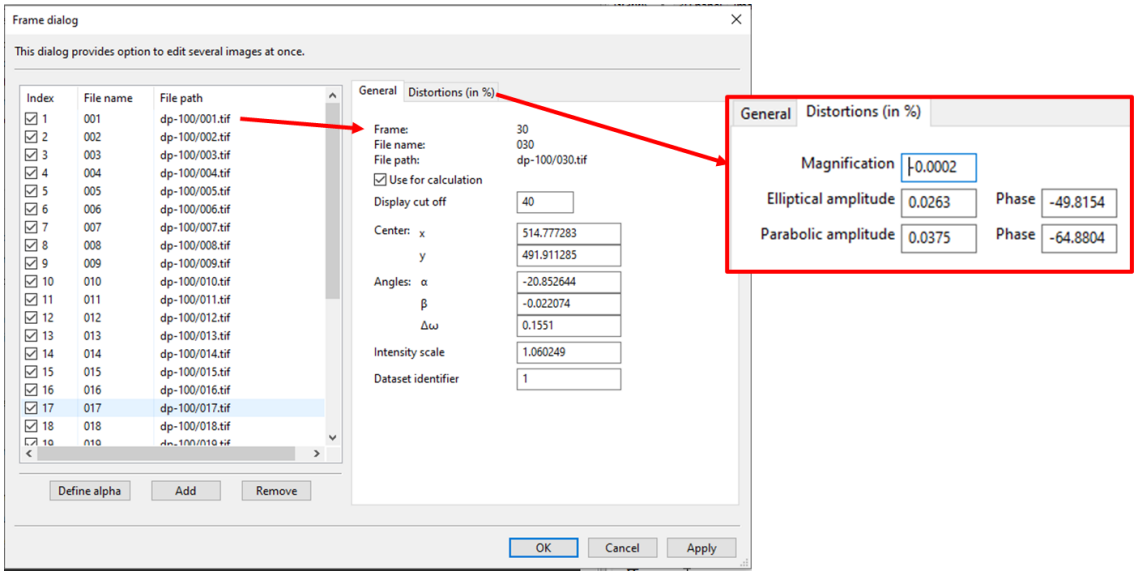


Figure B-6

B.2.2.b. Settings panel

In that section, some saving settings (Figure B-7), colors, size of some items for the different panels, and the font size of the console (Figure B-8) can be adjusted as shown on the two pictures below.

General settings are related to the way you want to handle simulated and processed images. Those images can take up a lot of space. You might choose to keep them or not using the options shown in Figure B-7.
The panels which can be adjusted are:

- The 3D panel with the colors of the 3-dimensional reconstruction of the reciprocal space.
- The cylindrical projection,
- The colors of some overlay's items displayed in the Image data where ED frames are shown,
- The settings related to the sections of the reciprocal space,
- The colors and font of the Console,
- The background and font used in the Graphs and plots.

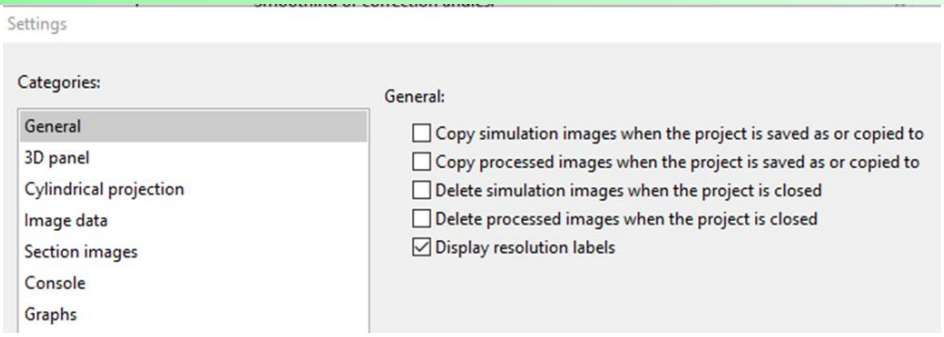


Figure B-7

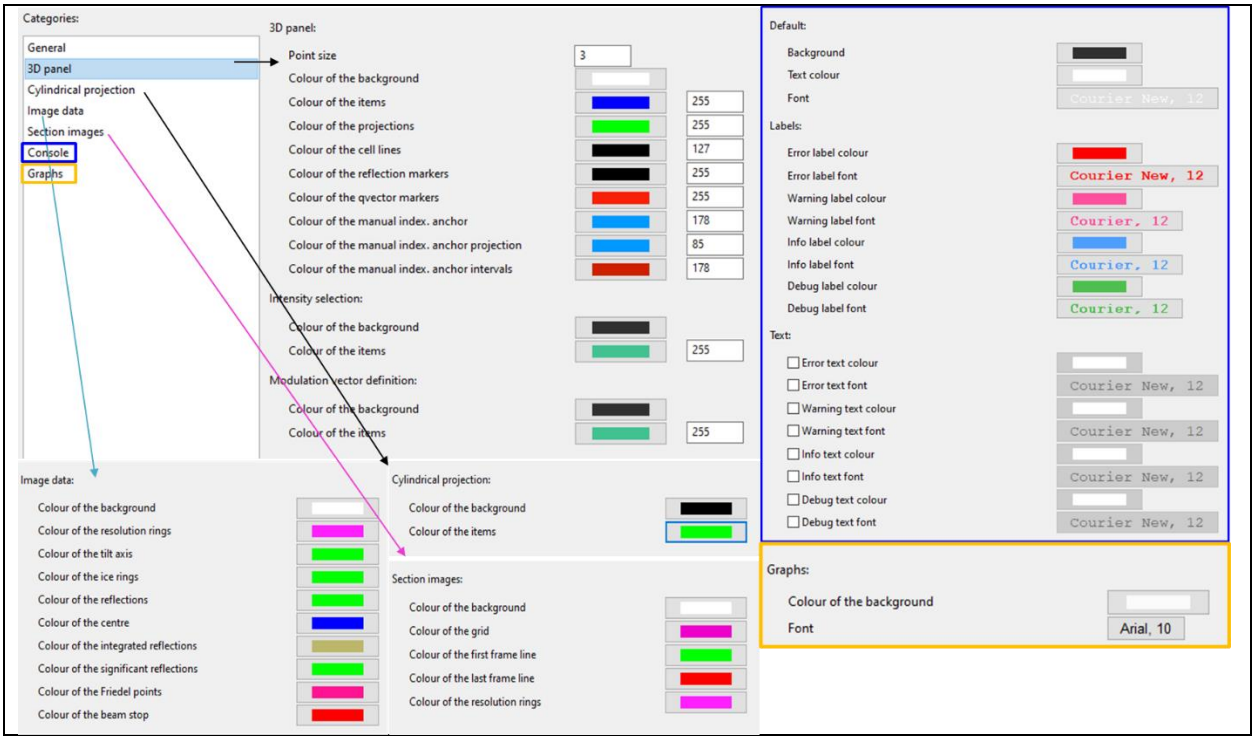


Figure B-8

B.2.3 View

The View menu is summarized below (Figure B-9). It contains the items Main panel tabs, Panels, Tools/Layouts, Reset layout, Show tooltips.

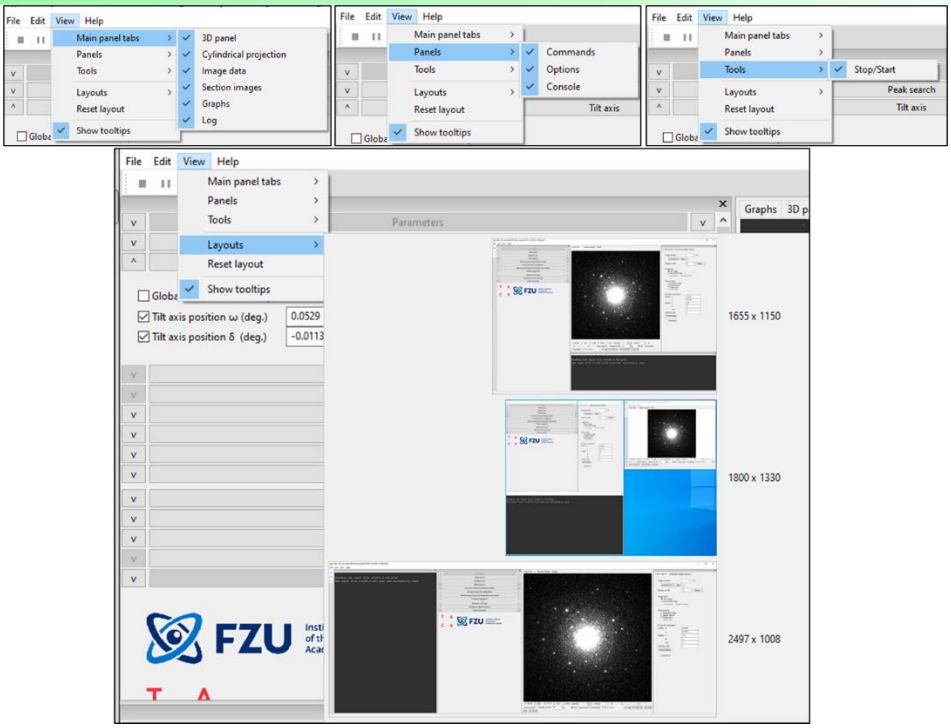


Figure B-9

- Main panel tabs menu. Using checkboxes to display or not specific tabs in the Main panel: 3D panel/ Cylindric projection/Image data/Section images/Graphs/Log.
- Panels menu to display or not the following panels: Commands/Options/Console.
- Tools: stop/start. Icons Stop/Start and Pause can also be found at the top of the Commands panel.
- Layouts: three default layouts are available: 1655x1150; 1800x1330; 2497x1008. They can also be adjusted manually by dragging the window to the desired position. A blue area will appear and show where the panel will be moved.
- Reset layout: go back to the default layout (useful when you mess up the layout manually).
- Show tooltips: check/uncheck that option to activate/deactivate tooltips in the GUI.

B.2.4 Help

Software information, version number, logo etc. (Figure B-10).

A new button "Copy the version information to the clipboard" is now available

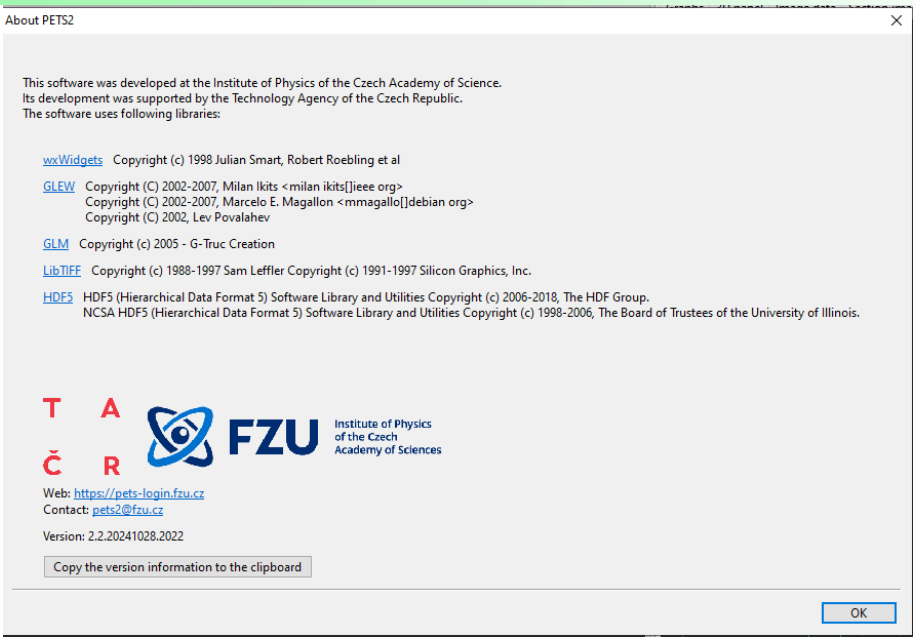


Figure B-10

B.3. Commands or function panel

All the functions with their options for data reduction are available in that panel (Figure B-11). This section aims to introduce the graphical features and briefly describe the functions.

As explained previously, parameters and options related to a command are displayed by hitting one of the arrows ˅ on each side of the main command button (blue in Figure B-11). To run the command, hit the main button (red in Figure B-11).

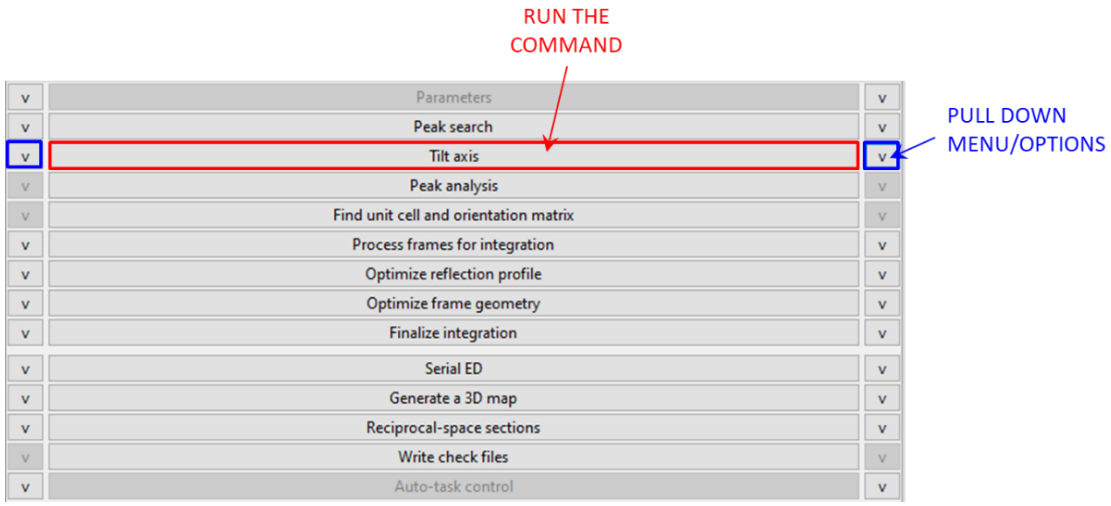


Figure B-11

B.3.1 Parameters

Open the options menu with the left or right arrow on each function button's side (**Error! Reference source not found.**). This menu shows the basic parameters of the experiment.

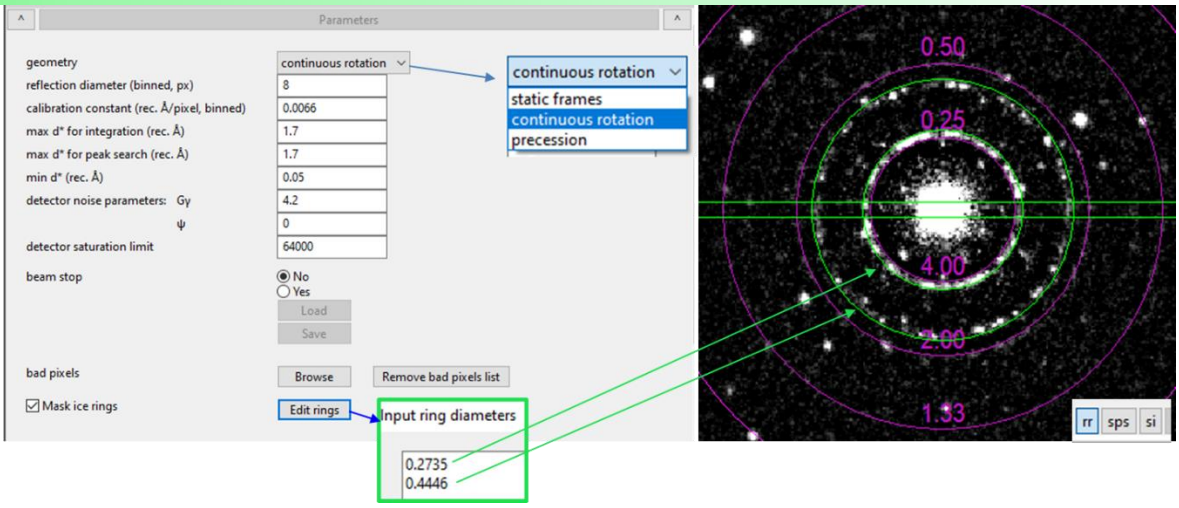


Figure B-12

The geometry of the 3D ED experiment can be set here (**Error! Reference source not found.**): static frames, continuous rotation, or precession. The other parameters needed to start the experiment are:

- The reflection diameter in pixels. The value is the binned one: the binning is set when a new PTS project is created or in the input pts/pts2 file. Used for peak search and integration, it should be large enough to encompass the strong spots, but small enough to avoid overlaps of neighboring spots.
- *d** values: the distances in $Å^{-1}$ on the pattern from the direct beam. Different values of maximum resolution can be set for the peak search and the integration. The *d** minimum is the distance below which no peak is considered (for peak search and integration). By default, *max d** = 1.4 $Å^{-1}$ and *min d** = 0.05 $Å^{-1}$.
- The two detector noise parameters are detector specific. They are used to determine $\sigma(I)$. The one displayed in **Error! Reference source not found.** correspond to the ASI Cheetah detector.
- The detector saturation limit (detector dependent).
- Beam stop: if Yes, use the Main panel to define the BS manually from the first frame (see the Main panel section B.4 to see how to proceed), or "Browse" the xy coordinates (text file) of the beam stop's shape. When the BS coordinates are loaded, the BS outline is displayed on the frames and its position can be adjusted from the Main panel (see section B.4 Main panel for the explanations).
- Bad pixels. This option removes bad pixels during the data processing (detector dependent). To use this option, load a list of pixel coordinates x and y (text file). The coordinates are defined so that the upper-left pixel has coordinates 1,1.
- Mask ice rings. Check this box to avoid the peak search or the integration close to the ice rings positions as shown in **Error! Reference source not found.**. The default mask are two rings defined by their d* values relative to the calibration constant used. The ice rings (green) can be visualized together with the resolution rings (pink) on the "Image data" tab of the Main panel once the center was set (during the peak search) (Figure B-50).

B.3.2 Peak search

Peak search runs when the "Peak search" command is hit (Figure B-13).

The options related to the peak search are accessible by pulling down the option menu using the arrows.

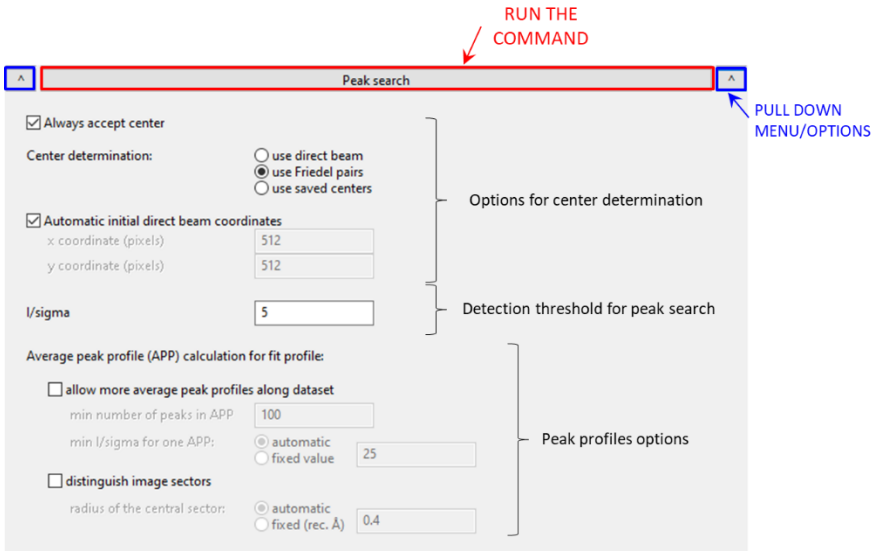


Figure B-13

In the "Peak search options" are given in Figure B-13 with default settings. Three types of options are available and described in the sections below.

-The options for the **center determination**,

-The **detection threshold I/σ(I)** for the peak search,

-The options to define more than one **peak profile** for the data set.

B.3.2.a. Center determination options

- **Always accept center**: when not checked, and the center moves by more than 1/8 of the reflection size, PETS2 asks to either accept the new center or enter manually new coordinates.
- **Center determination modes**:
  -Use direct beam: the center of direct beam is used as center of the diffraction pattern. It can be inaccurate for non-circular direct beam.
  -Use Friedel pairs: PETS2 will look for Friedel pairs on each frame to determine the center of the ED pattern. This option is usually more accurate when the frame contains enough reflections.
  Note: the Friedel pair estimation of the center can be done manually too using the "Image data" panel. The procedure is described in Section B.4.1.c
  -Saved centers: This option is used when the centers were optimized from the geometry optimizations and kept to redo a new reconstruction of the reciprocal space to get more accurate lattice parameters.
- **Automatic initial beam coordinates**: if PETS2 fails to find the center automatically, uncheck this checkbox and enter manually the center's coordinates or use the button "f", "mf" or "df" at the bottom of the Image data tab to manually determine Friedel pairs and the center. The option "f" (= define Friedel pairs), "mf" (= move one Friedel point) and "df" (= delete Friedel point) are active only when the option "automatic initial beam coordinates" is unchecked.

The manual procedure to determine the center is shown in *Section B.4.1.c*

When the Peak search function is run, PETS2 looks for peaks in individual frames within the defined resolution range (*d** max for peak search) and found above the detection threshold (green circles in Figure B-14). The peaks involved in Friedel pairs are visible as green circles with a cross inside and used for the

center determination (when the center mode is Friedel pairs). The center is shown as a blue cross (default color setting). In the “Image data tab” showing the frames, the option “sps” (= show peak search overlay) is checked by default when the peak search starts to show the center and the detected reflections (Figure B-20).

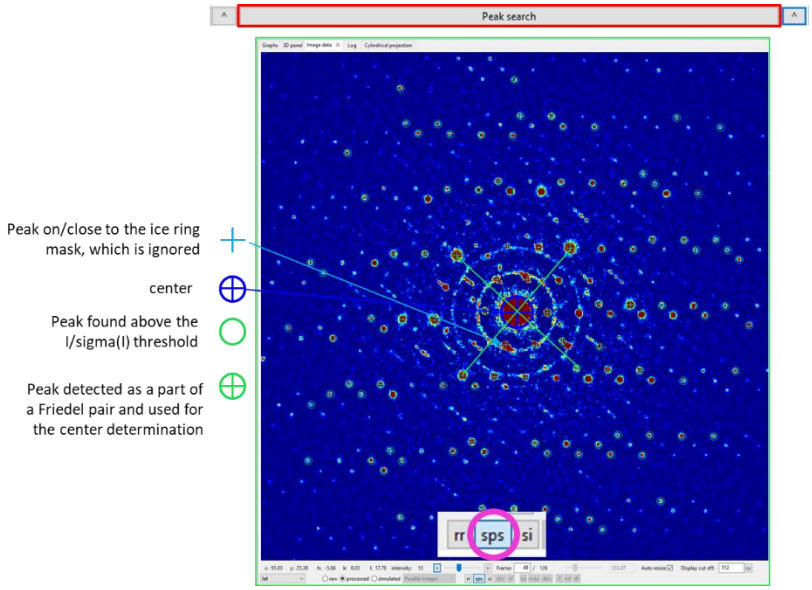


Figure B-14

B.3.2.b. Detection threshold

The detection threshold for peak search is defined as I/sigma(I). Only reflections found above the threshold are recognized as peaks. The sigmas(I) used in the peak search are based on the counting statistics assuming a normal distribution. Sigmas are in fact not following a normal distribution, which is corrected later in the procedure (finalize integration step). The peak search always uses non corrected sigma(I).

B.3.2.c. Peak profiles options

By default, PETS2 defines one average peak profile (APP) per dataset during the Peak search procedure. If two or more datasets are merged, each dataset will have its own APP. This peak profile is used later during the integration of intensities (using “fit profile” option). The average peak profile is defined with its FWHM in pixels and will be listed in the log file “Peak Search” (Figure B-15 *from the log file)*.

```
Stored average peak profiles:
APP nr.      frames   secId   mean I/sigma   Npeaks   FWHM
     1      1 -  96       0           18.8     1954    2.5
```

Figure B-15. example of Average peak profile listing after the Peak search procedure.

There are now two options to allow more average peak profiles in the datatset(s). Defining more APP is useful for data with anisotropic mosaicity, diffuse scattering. A common case are crystals with preferential

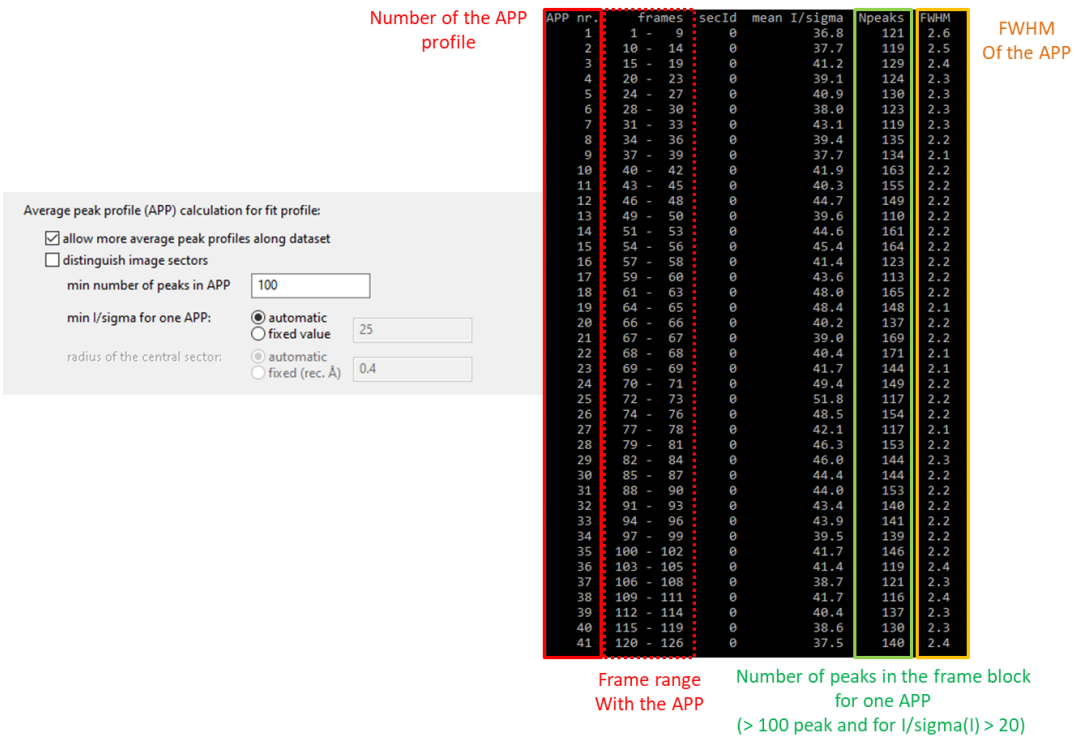
orientation showing sharp reflections in one direction and broader reflections in the last direction visible at high tilt angles. This list is non-exhaustive.

➢ **Allow several APP along a data set** (Figure B-16).

By checking that option, PETS goes through the frames during peak search and create blocks of frames (slices) with individual APP per slice. The number of slices (or APP) depends on the dataset (number of reflections per frame and number of frames), the parameters set for the *minimum number of peaks to define one APP,* and *the threshold in I/sigma(I) for one APP*. The results are given after the peak search in the debug console (additional window when PETS2 opens) as shown in Figure B-16 and in the log file "Peak search".

- The default value for **the minimum number of peaks in APP = 100**. This number is large enough to ensure good statistics and should not be lower too much. For datasets with a lot of peaks per frame, this number can be set higher to avoid too many APP.
- The **minimum I/sigma(I) for one APP** is automatically evaluated in PETS2. The threshold can be adjusted to include weaker or stronger reflections in the APP evaluation and will change the number of APP.

The values of the two parameters can be tested. Setting several APP along the data set is meaningful when an appreciable difference in FWHM for each APP is observed along the dataset like in Figure B-16. Every time the parameters are changed, "Peak search" function must be run again to reevaluate the number of APP.



APP nr.	frames	secId	mean I/sigma	Npeaks	FWHM
1	1 - 9	0	36.8	121	2.6
2	10 - 14	0	37.7	119	2.5
3	15 - 19	0	41.2	129	2.4
4	20 - 23	0	39.1	124	2.3
5	24 - 27	0	40.9	130	2.3
6	28 - 30	0	38.0	123	2.3
7	31 - 33	0	43.1	119	2.3
8	34 - 36	0	39.4	135	2.2
9	37 - 39	0	37.7	134	2.1
10	40 - 42	0	41.9	163	2.2
11	43 - 45	0	40.3	155	2.2
12	46 - 48	0	44.7	149	2.2
13	49 - 50	0	39.6	110	2.2
14	51 - 53	0	44.6	161	2.2
15	54 - 56	0	45.4	164	2.2
16	57 - 58	0	41.4	123	2.2
17	59 - 60	0	43.6	113	2.2
18	61 - 63	0	48.0	165	2.2
19	64 - 65	0	48.4	148	2.1
20	66 - 66	0	40.2	137	2.2
21	67 - 67	0	39.0	169	2.2
22	68 - 68	0	40.4	171	2.1
23	69 - 69	0	41.7	144	2.1
24	70 - 71	0	49.4	149	2.2
25	72 - 73	0	51.8	117	2.2
26	74 - 76	0	48.5	154	2.2
27	77 - 78	0	42.1	117	2.1
28	79 - 81	0	46.3	153	2.2
29	82 - 84	0	46.0	144	2.3
30	85 - 87	0	44.4	144	2.2
31	88 - 90	0	44.0	153	2.2
32	91 - 93	0	43.4	140	2.2
33	94 - 96	0	43.9	141	2.2
34	97 - 99	0	39.5	139	2.2
35	100 - 102	0	41.7	146	2.2
36	103 - 105	0	41.4	119	2.4
37	106 - 108	0	38.7	121	2.3
38	109 - 111	0	41.7	116	2.4
39	112 - 114	0	40.4	137	2.3
40	115 - 119	0	38.6	130	2.3
41	120 - 126	0	37.5	140	2.4

Figure B-16. In that example, the PEDT dataset has 126 frames. With 100 as the minimum number of peaks with a threshold of I > 20 sigma(I), the dataset is cut into 41 blocks, with 41 individual APP. The difference in the APP is defined by their FWHM.

➢ **Distinguish sectors in frames with individual APP**

When this option is selected, the frame is split into 7 sectors with one APP per sector as shown in the Picture below. The first sector is a circle around the center with an **automatic radius** ≈ $\frac{3}{4*\sqrt{7}} d^*_{\max integration}$. This value can be changed by selecting "**fixed**" with the radio button. The other six sectors of 60 degrees each are centered on the projection of the alpha tilt axis on the frame.

When this option is selected independently, there is an internal limit of about 7000 items to define a set of 7 sectors. The I/sigma used for one APP is automatically evaluated by the program and will be different for each dataset.

After the peak search, the FWHM for the APP per sector are listed in the back console. When all FWHM are very close for all sectors, this option does not bring any improvement.

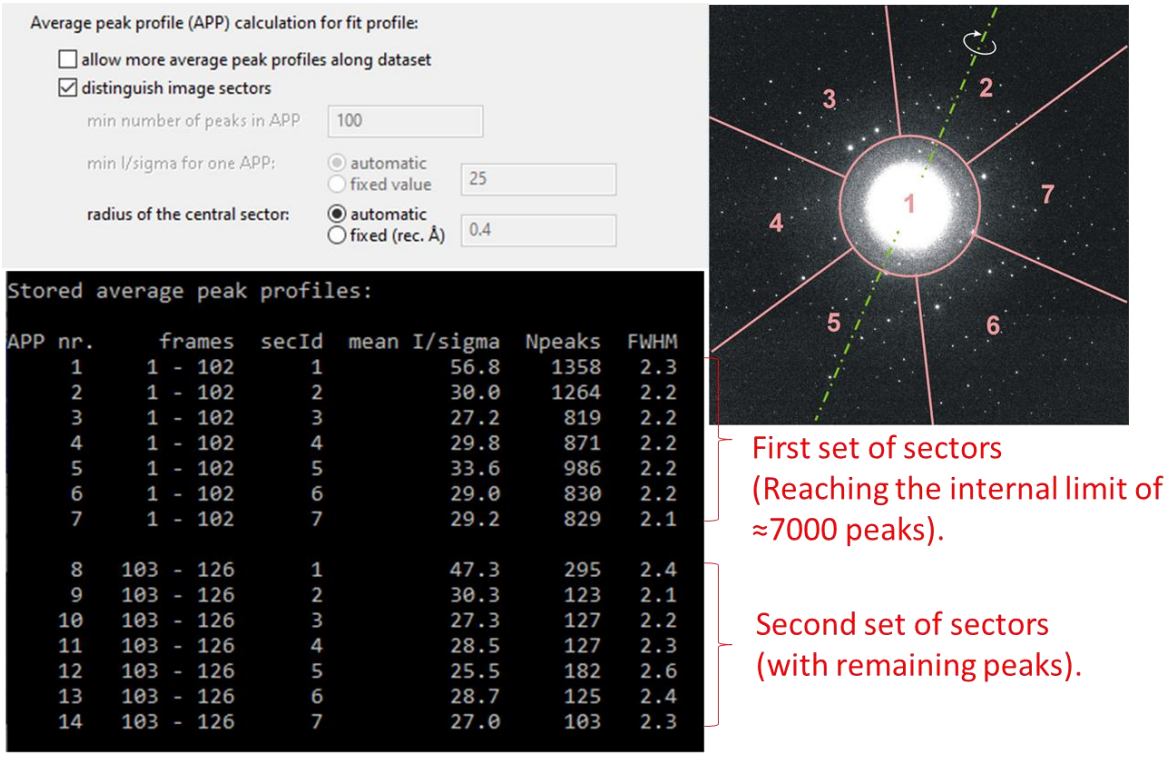


Figure B-17. example of several peak profiles by distinguishing image sectors. In this example the dataset has a lot of peaks. The internal limit to define sectors is 7000 items. That is the reason there are two sets of sectors profiles instead of 1.

➢ **Combining several APP along a data set AND image sectors**.

Generally, even though the options "several APP along a dataset" and "distinguish image sectors" can be used independently in PETS2, there is no obvious reason to distinguish sectors without allowing several APP along a data set.

When both options are selected (checked boxes in Figure B-18).

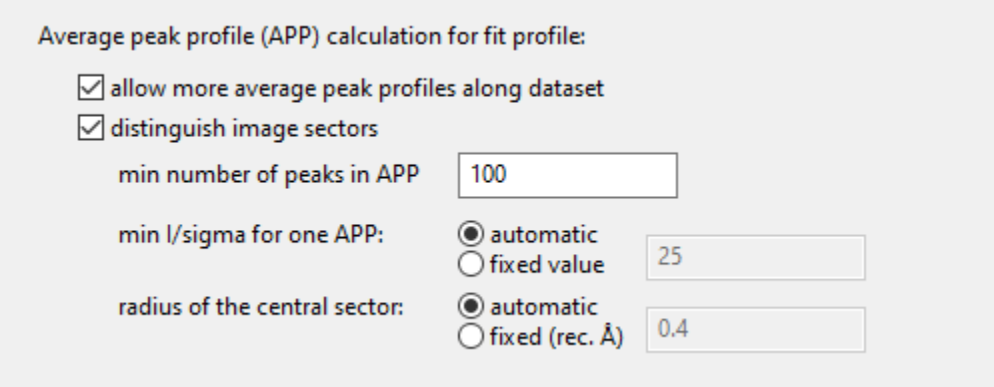


Figure B-18 GUI related to peak profiles in the "Peak Search" command with the two options checked

The options are used as follows:

- The default value for **the minimum number of peaks in APP for one sector = 100**. This number is large enough to ensure good statistics and should not be lower too much. For datasets with a lot of peaks per frame, this number can be set higher to avoid too many APP.
- The **minimum I/sigma(I) for one APP** is automatically evaluated in PETS2. The threshold can be adjusted to include weaker or stronger reflections in the APP evaluation and will change the number of APP.
- **The radius of the central sector** (first sector) is automatically set depending on the *d* max integration* or can be set manually in rec.Å.

Remark: when the option "Allow several APP along a data set" is used alone with the minimum number of peaks in APP = 100, more slices are obtained than when both options are combined. The reason is that now the minimum number of peaks in APP = 100 is for one sector and not the full frame. (see example below in Figure B-19*).*

- One average peak profile:

Stored average peak profiles:

APP nr.	frames	secId	mean I/sigma	Npeaks	FWHM
1	1 - 96	0	18.8	1954	2.5

- Allow more average peak profiles along dataset:

Stored average peak profiles:

APP nr.	frames	secId	mean I/sigma	Npeaks	FWHM
1	1 - 13	0	17.2	123	2.5
2	14 - 19	0	18.2	128	2.5
3	20 - 25	0	18.8	115	2.4
4	26 - 29	0	18.5	111	2.5
5	30 - 34	0	19.9	128	2.5
6	35 - 39	0	18.5	132	2.5
7	40 - 43	0	19.7	139	2.5
8	44 - 47	0	20.0	143	2.5
9	48 - 51	0	18.3	117	2.6
10	52 - 56	0	19.9	136	2.4
11	57 - 60	0	22.0	113	2.4
12	61 - 64	0	17.3	121	2.4
13	65 - 69	0	22.0	118	2.5
14	70 - 74	0	18.4	115	2.5
15	75 - 81	0	16.4	113	2.7
16	82 - 96	0	15.1	102	2.7

- Distinguish image sectors:

Stored average peak profiles:

APP nr.	frames	secId	mean I/sigma	Npeaks	FWHM
1	1 - 96	1	21.9	401	2.4
2	1 - 96	2	11.6	541	2.5
3	1 - 96	3	13.2	522	2.6
4	1 - 96	4	12.8	559	2.6
5	1 - 96	5	12.0	564	2.5
6	1 - 96	6	13.3	519	2.6
7	1 - 96	7	13.1	548	2.6

- Allow more average peak profiles along dataset and distinguish image sectors

Stored average peak profiles:

APP nr.	frames	secId	mean I/sigma	Npeaks	FWHM
1	1 - 31	1	21.6	104	2.4
2	1 - 31	2	10.8	112	2.6
3	1 - 31	3	13.9	137	2.6
4	1 - 31	4	14.6	136	2.5
5	1 - 31	5	11.8	111	2.5
6	1 - 31	6	14.1	129	2.5
7	1 - 31	7	14.4	139	2.6
8	32 - 49	1	21.0	104	2.3
9	32 - 49	2	11.9	161	2.6
10	32 - 49	3	13.3	168	2.6
11	32 - 49	4	13.3	156	2.6
12	32 - 49	5	11.9	178	2.5
13	32 - 49	6	13.4	160	2.6
14	32 - 49	7	13.2	156	2.6
15	50 - 67	1	26.6	104	2.2
16	50 - 67	2	11.8	140	2.5
17	50 - 67	3	11.8	121	2.6
18	50 - 67	4	12.5	154	2.6
19	50 - 67	5	12.3	139	2.5
20	50 - 67	6	12.4	145	2.6
21	50 - 67	7	13.2	143	2.6
22	68 - 96	1	17.9	89	2.5
23	68 - 96	2	11.6	128	2.6
24	68 - 96	3	13.7	96	2.7
25	68 - 96	4	10.5	114	2.7
26	68 - 96	5	11.5	146	2.6
27	68 - 96	6	13.7	85	2.8
28	68 - 96	7	11.0	110	2.7

Figure B-19. example of one dataset processed with the different average peak profile options.

B.3.3 Tilt axis

The angle between the projection of the alpha-tilt axis on the image and the horizontal axis on the frame is called *ω* angle. It depends on the microscope, the camera length, and the exact focusing conditions. Therefore, it cannot be precisely calibrated and may need to be optimized for each data set.

Two checkboxes indicate if the tilt axis position is **refined globally** (a 360-degrees scan) or/and more precisely with "**refine tilt axis position *ω***" (Figure B-20). The sharpest the projection becomes, the better. The details of how it works are beyond the scope of this tutorial. Still, you can quickly assess the quality of the data and accuracy of the parameters by looking at the cylindrical projection of the peak positions shown by PETS2 during the refinement in the cylindrical projection tab of the Main panel. The angle *ω* can be manually changed, and the cylindrical projection replotted using the button "Replot".

Another **angle δ delta** corresponds to the inclination of the alpha tilt-axis in the perpendicular direction of omega (out-of-plane). The value can be refined too and should remain close to zero.

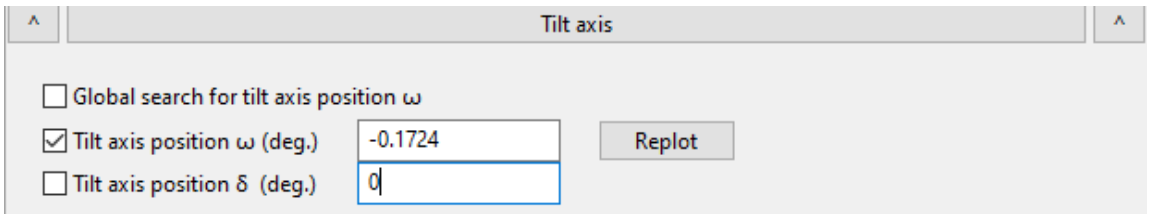


Figure B-20

B.3.4 Peak analysis

There is no option below that command in the GUI. Running "Peak analysis" procedure will recreate the 3D cloud of reflections after clustering and background removal. This is a two-step process.

Before running the peak analysis, a *.cor file (3D cloud without clustering and without background canceling) is the only 3D peak distribution available. After the clustering, three other peak lists are produced: the xyz, the clust, and the diff files. The 3D reconstructions of the reciprocal space are used for the indexing (see sections B.3.5).

Hit "Peak analysis"

- **STEP 1**: PETS2 starts the analysis of the peak positions obtained in the peak hunting procedure. The first step analyzes distance distribution between peaks in the image plane. Then, a sorted plot of inter-peak distances is displayed in the Graph tab (red curve) with its derivative (green). For a good-quality data set, the red curve must contain distinct steps; consequently, the green curve has distinct peaks (Figure B-21).

Hit "Peak analysis (continue)".

- **STEP 2**: In the next step, an auto convolution of the diffraction pattern (difference space) is analyzed, and the groups of peaks in the auto convolution (clusters) are replaced by the cluster centers. Again, a distance distribution is displayed (Figure B-21, right screenshot), and clear-cut steps on the curve indicate a well-defined lattice.

Hit "Peak analysis (continue)" → PETS2 prepared the files needed for unit cell determination and indexing.

You should get the following message in the console:

```
Threshold for considering two peaks the same: 0.0046 reciprocal angstroms.
15928 peaks merged into 6362 clusters, 1670 with more than one peak.
Peak analysis successfully finished.
```

At the end of this procedure three files are produced: jobname.xyz contains the "cleaned" peak list (background canceling), where individual peaks are replaced by the cluster centers without compensation for the missing wedge. jobname.diff contains the difference vectors, and jobname.clust contains the list of cluster centers from the difference vectors with compensation for the missing wedge. The reconstructions are described in Section B.3.5 related to the indexing interface.

What can go wrong?

For data with disorder in the direction of the beam, the first in-plane step will look good (periodicity). However, the second step will mainly show one strong maximum on the very left of the plot (low distances between cluster). It means that the parameter in the out-of-plane direction will look very large (low region of the plot) or no periodicity will be visible in the clust.

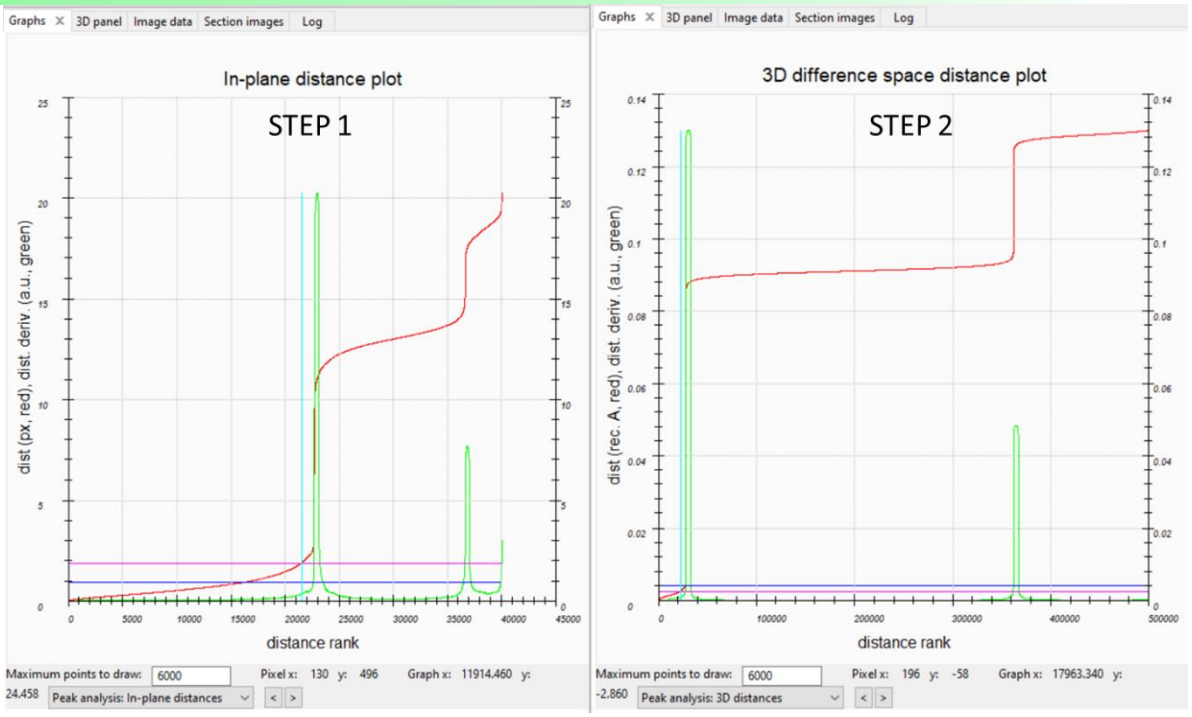


Figure B-21. The two plots obtained from the Peak analysis procedure and visible in the Graphs tab of the main panel.

B.3.5 Find unit cell and orientation matrix

This huge section describes both the graphical interface, and the options related to the indexing step.

Select “Find unit cell and orientation matrix” command to access the indexing panel.

The Main panel tab switches to the 3D panel tab (with the 3D reciprocal space visualization, the indexing options/functions panel (red) with “Indexation display options” (green) are now activated. The basic indexing layout is shown in Figure B-22 with the list of functions on the left, the 3D panel, and the display options (green) on the right-side panel.

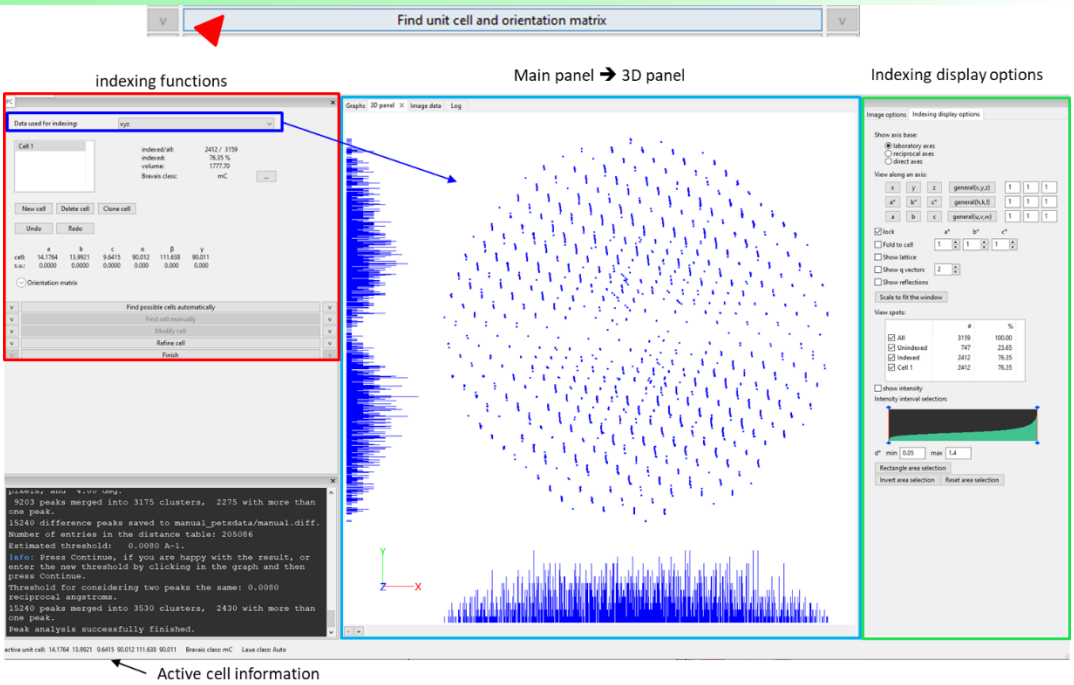


Figure B-22

On the left window, the Indexing panel contains a header (Figure B-23) and several functions to define (a) new unit cell(s), to modify it/them, clone and refine it/them.

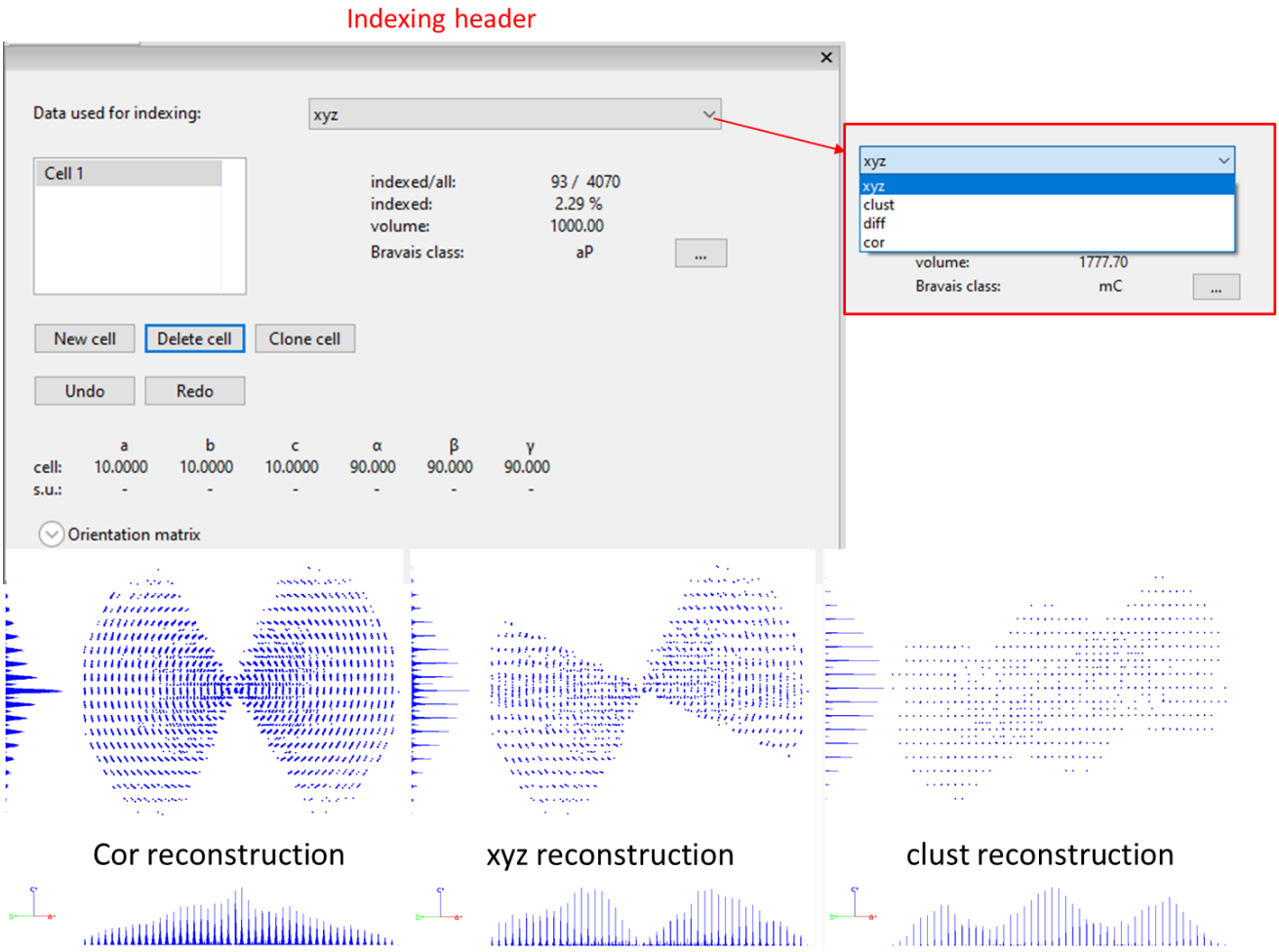


Figure B-23

The header contains first a pull-out menu of all the 3D reconstructions available (xyz, clust, diff, and cor files) (Figure B-23). The selected reconstruction is displayed in the 3D panel tab of the Main panel. The header also contains a list of all unit cells defined with the basic information on the parameters, the Bravais class, the centering, the unit-cell volume, the orientation matrix, and the statistics on how well the unit cell indexes the peaks.

Five buttons/functions are below the cell list: New cell, delete cell, clone cell, Undo, and Redo. Undo and Redo are there to cancel the previous action(s) and, eventually, change your mind again with Redo.

When the indexing panel is opened for the first time, a default Cell 1 is present with parameters 10 10 10 90 90 90 (Figure B-23).

When several unit cells are defined. The active one can be selected by clicking on the name of the cell to activate. All options will be applied to the active cell.

To change the name of the cell, select the cell in the list to change, and click it once more (fast) with the left mouse button.

Next to Bravais class, the icon "…" opens a new window where one can change the crystal system (a = triclinic, m = monoclinic, o = orthorhombic, t = tetragonal, r = rhombohedral, h = hexagonal, and c = cubic), and adjust the centering (standard or non-standard) (Figure B-24). As indicated in the picture, when you define a non-standard centering, the definition of each centering vector needs to be on a separate line. Each vector must have 3+ "number of modulation vectors" items.

The centering (if any) will be accounted for when reflections circles are displayed on the 3D reconstruction.

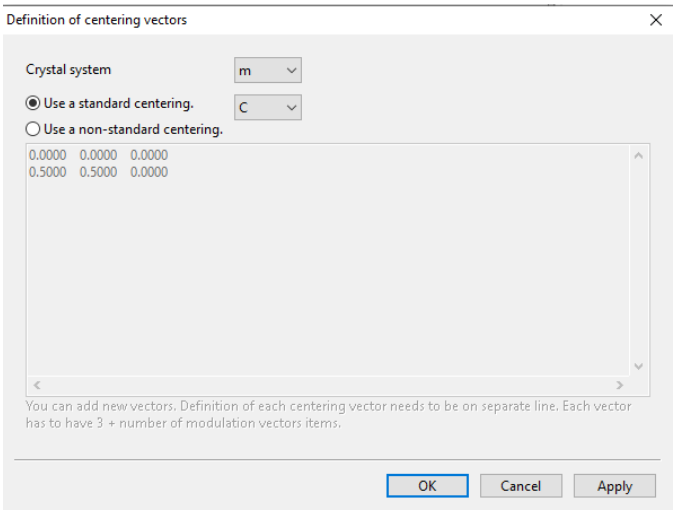


Figure B-24

Below the header is a list of functions and commands (Figure B-25).

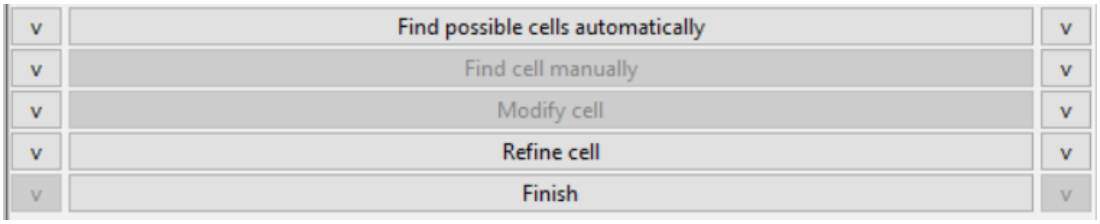


Figure B-25

B.3.5.a. Find possible cells automatically

There are two ways to define a unit cell in PETS2. Either using one of the two automatic indexing methods or using the manual indexing. In that section, we describe options for automatic indexing.

Pull-down the menu of "Find possible cells automatically" with the arrows on each side of the command to access the parameters of that function (Figure B-26). Two main algorithms are available to search for the unit cell automatically: "**from difference space**" (default option) and "**from triplets**".

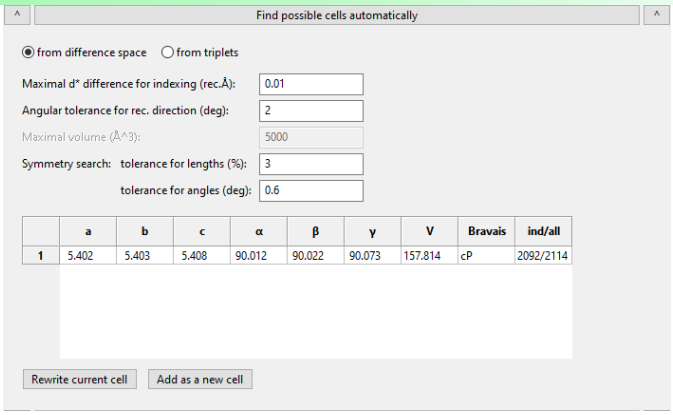


Figure B-26

- "**from difference space**" is the default option and always uses the clust file to determine distinct directions in reciprocal space. The best unitcell is then refined with the selected reconstruction (data used for indexing: cor, xyz or clust). Make sure the clust reconstruction is meaningful and representative of the complete data before using that option (compare it with the .cor or .xyz file); otherwise, the automatic search will likely fail or find a sub-cell.
- The "**from triplet**" option is based on the selected 3D reconstruction (xyz, clust, dif, or cor file).

For both automatic search options, further parameters can be adjusted.

- The **maximal d* difference for indexing** (in rec. Å) is the error allowed between a reflection and the nearest lattice point position (defined by the orientation matrix and the lattice parameters).
- The **angular tolerance** is given in degree and only relevant when the "from difference space" is selected. Within this angle the different reciprocal directions are considered as one direction.
- The **maximum volume** allowed for the unit cell can only be changed when the "from triplet" option is selected.
- The **two tolerance options for symmetry search** on the lengths and angles are used to determine the possible crystal system and centering written as Bravais class in the header.

When the "Find possible cells automatically" command is run, all the possible unit cells found by the algorithm will appear in the list with the corresponding parameters, the most likely Bravais system and the number of reflections indexed by this unit cell. The best cell will be automatically selected. In the simple case shown in Figure B-26, the algorithm found only one cubic unit cell. Sometimes, when the data is more ambiguous, the list can contain a lot of possible unit cells. In the worst case, when no unit cell is found, an error message appears in the console.

Two buttons are below the list of possible unit cells: rewrite current unit cell and Add as a new unit cell.

Remark: In the case of a centered monoclinic cell, PETS2 will choose the settings to minimize the beta angle. It can lead to body centered monoclinic cells instead of the standard C-monoclinic that software usually like.

B.3.5.b. Find unit cell manually

That section presents the tools to manually define a new cell or modify a pre-existing one (Figure B-27). That section works hand and hand with the 3D panel and the “indexation display options” panel.

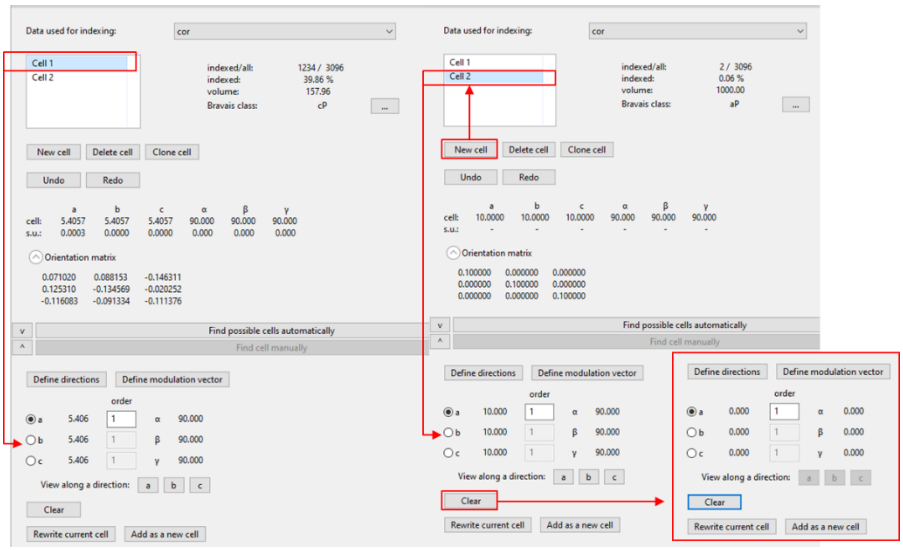


Figure B-27

Select a cell to modify manually in the cell list available or create a new one. By default, a new unit cell is created with parameters 10 10 10 90 90 90 and its parameters are loaded in the section “Find cell manually”. You can either “Clear” the existing parameters to start with 0 0 0 0 0 0 or start defining the parameters from the existing ones (Figure B-27).

To define unit cells parameter manually:

- Rotate the cloud of reflections in the 3D panel until a special orientation is found (periodic stripes on the side of the 3D panel) with one of the main lattice vectors in the plane of the projection,
- Activate the “**Define directions**” option.
- In the 3D panel, **left click and drag** the cursor to where you want to define a parameter, as shown below (Figure B-28).
- **Adjust the order** of the plane (i.e. the number reciprocal-lattice planes between the selected plane and the origin), if the automatic estimation does not provide good value. In the example Figure B-28, the order should be 5 because the cursor was dragged through 5 interplanar spaces.
- To move the 3D cloud again and **find a new direction**, first, **deactivate the “Define directions” button**.
- Use the **view along a direction** (**a**, **b** or **c**) to help find new directions.
- When all the unit cell parameters are defined, hit “**Rewrite current cell**” to replace the previous parameters with the new ones in the header.

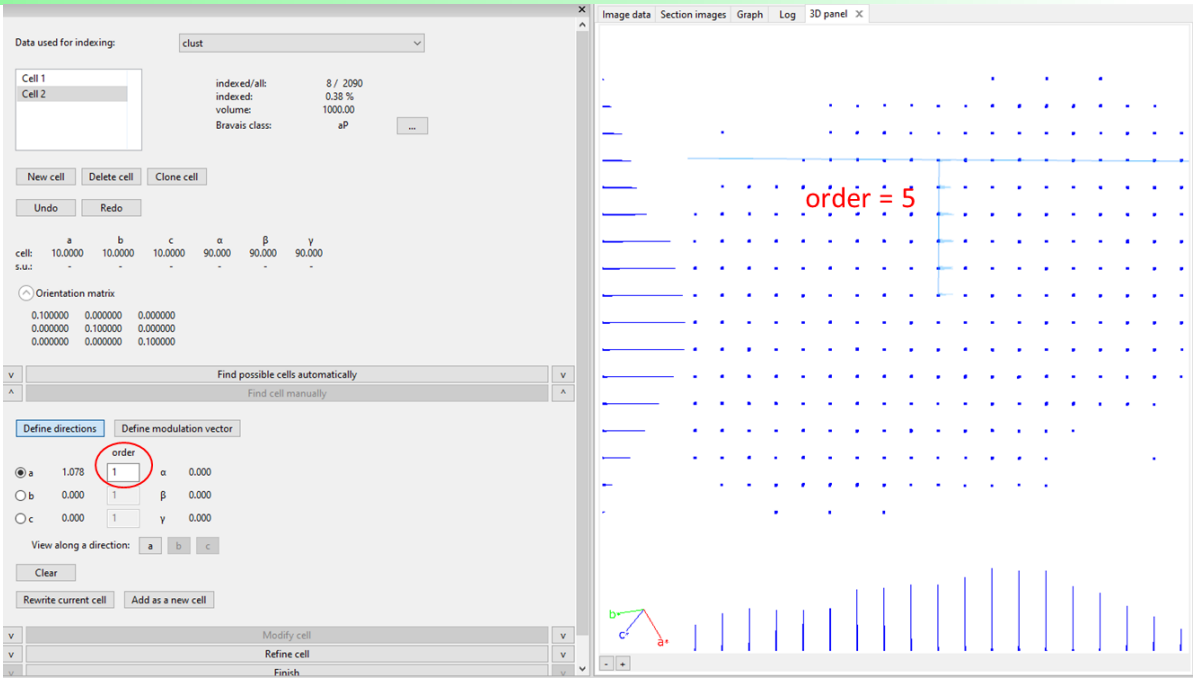


Figure B-28

B.3.5.c. Modify cell

In this section (Figure B-29), several tools help you to modify the active unit cell.

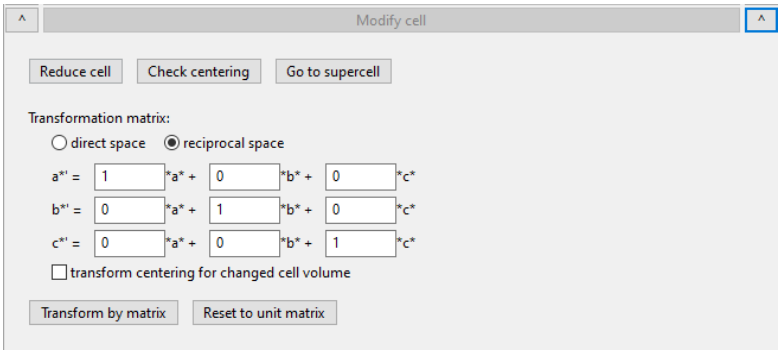


Figure B-29

- **Modify the active unit cell** by a matrix in the direct or the reciprocal space. To apply the transformation matrix, hit "Transform by matrix". The matric can be defined either in the direct space or in the reciprocal one.
  -For centered unit cell, check "transform centering for changed cell volume" to apply the centering in the new settings.
  -To reset the matrix to unit matrix, press "Reset to unit matrix."

- **Reduce** the cell by hitting "Reduce cell",
- **Check centering** to test if there is a centering with "Check centering" (for unit cell defined manually). The result is given in the console like in the example for the cubic pyrite:

```
Check centering: The centering cP was confirmed.
```

```
Unit cell is primitive and search for higher symmetry (centered) cell is
going on .....
Cell with higher symmetry was not found.
   5.406   5.406   5.406   90.00   90.00   90.00    157.96   c P
   7.645   7.645   9.363   90.00   90.00  120.00    473.88   h Robv
```

When a centering is found, it is applied in the header with the Bravais class.

The automatic "Check centering" often fails when some reflections are weak. It can result in acrobatic nonstandard centering.

The best way to check if the new centering is right is to fold the reciprocal space into a 2a 2b 2c cell and display the reflection circles with "show reflections". The option will respect the centering written in the header with the Bravais class Figure B-30.

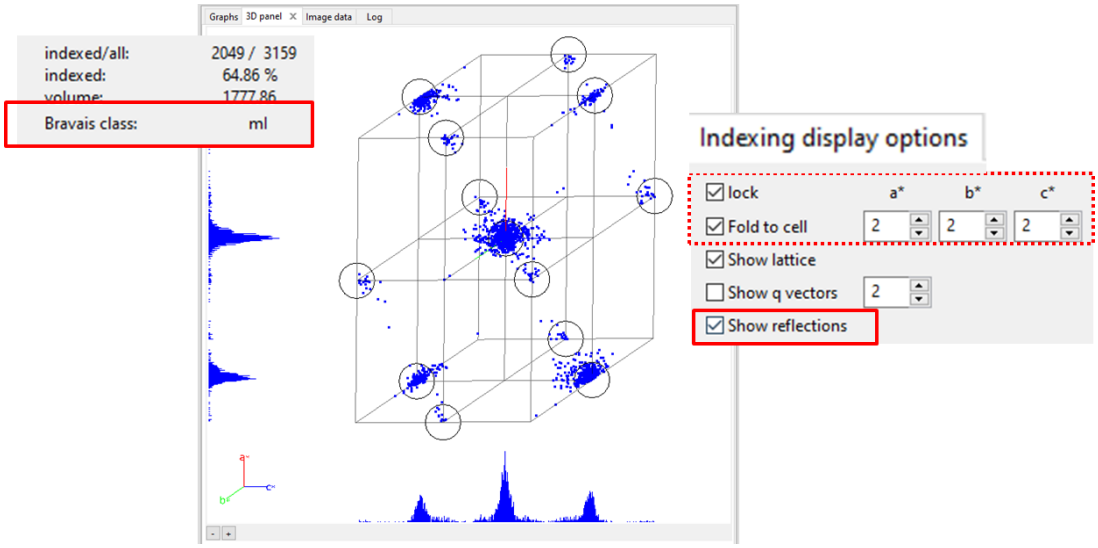


Figure B-30. Example of a I-centered monoclinic unit cell

- **Look for a supercell** with "Go to supercell". The result is given in the console like in the example for the cubic pyrite:

```
Sorry, no frequent fraction vector was found.
No suitable supercell was found.
```

If you are unhappy with the transformation, go into the header and hit "Undo" button below the cell list.

B.3.5.d. Refine cell: Options and methods

The section is dedicated to the refinement of the unit cell parameters with or without restrictions and, optionally, including/refining the global distortion parameters. 3D ED data can be used to get rather accurate lattice parameters when geometrical misalignments and optical distortions are properly accounted for. Geometrical optimizations will be presented in Section B.3.8 Optimize frame geometry. Optical distortions can be refined globally during the refinement of the unit cell (this sections) and frame-by-frame together with the geometrical optimizations.

The refinement starts when the command "Refine cell" is hit.

To define the type of refinement to apply, pull-down the menu of options using the side arrows Figure B-31 .

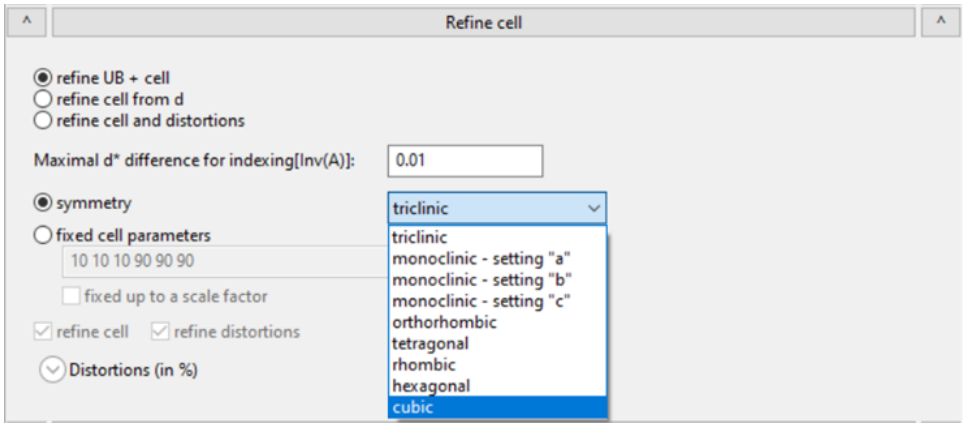


Figure B-31 Panel of unit cell refinement

Radio buttons are used to select one of the three refinement methods (Figure **B-31**);

For all three methods, **the indexing tolerance is defined as the "maximal d* difference for indexing** (in reciprocal Å)." The indexing tolerance is the same parameter as used in the automatic unit cell determination.

Remark: the size of the reflection circles in the 3D panel is proportional to the indexing tolerance.

REFINEMENT METHODS:

- **Refine UB + cell** (refinement against 3D reconstruction of the reciprocal space, recommended data cor and xyz). This refinement is very sensitive to distortions due to misalignments and optical distortions. U is the rotational matrix, and B is the basic matrix with our unit cell. This is the default method.

$$U\ matrix\ rotation = R_z(\theta)\begin{pmatrix} cos\theta & -sin\theta & 0 \\ sin\theta & cos\theta & 0 \\ 0 & 0 & 1 \end{pmatrix},$$

$$B\ unit\ cell\ matrix\ = \begin{pmatrix} a^* & b^*\cos(\gamma^*) & c^*\cos(\beta^*) \\ 0 & b^*\sin(\gamma^*) & -c^*\sin(\beta^*)\cos(\alpha^*) \\ 0 & 0 & c* \end{pmatrix}$$

In a diffraction experiment, the orientation matrix describes the orientation of the single crystal in space. The matrix relates the reciprocal axes of the crystal with the Cartesian coordinate system of the diffractometer.

Possible options for "refine UB + cell":

- "**symmetry**" restriction: will impose crystal system constraints on the unit cell parameters (see picture). This refinement can be done by forcing one symmetry as shown in Figure **B-31**.
- "**fixed cell parameters**": the unit cell will be transformed to be the same as you set. When the checkbox "fixed up to a scale factor" is selected, the refined unit cell will keep the same

proportions as the indexed cell and get as close as possible using a scale factor. The "fixed up to a scale factor" is only accessible with the method "refine UB + cell" because the scaling would correlate with the distortion parameters (the magnification) with the method "Refine cell and distortions."

- **Refine cell from *d*** (refinement using only *d* spacing, recommended data cor and xyz). This option is less sensitive to misalignments and optical distortions as it relies on *d* spacings found on the 2D frames. No symmetry or fixed cell parameters can be imposed for the "refine cell from d" option at this moment.

- **Refine cell and (global) distortions** (refined within the 2D of the frames – minimizes the distance between predicted and observed peak position, only cor data are used). In that mode, both cell and global distortions can be refined together or separately (checkboxes "refine cell" and "refine distortions"). The global distortions are stable and can be calibrated for each camera length and sample holder (assuming perfect measurement settings, like z height, focus and proper excitation of the diffraction length). Correcting distortions helps to get more accurate lattice parameters as well as a more accurate integration of peaks by correcting the position of the integration masks.

  Possible options for "Refine cell and (global) distortions":

  - "**symmetry**" restriction: will impose crystal system constraints on the unit cell parameters (see picture). This refinement can be done by forcing one symmetry as shown in Figure B 31. This is more than advised to set a symmetry for the refinement with distortions as the elliptical distortions correlate with the lattice parameters (97%). The correlation decreases when a symmetry is set.

  - "**fixed cell parameters**": the unit cell will be transformed to be the same as you set.

  - **Distortions**: global optical distortions can be refined with the unit cell parameters. Pull-down the "Distortions" menu (Figure B-32). The section below is focused on the distortion GUI.

B.3.5.e. Refine cell: Global optical distortions

A specific section is dedicated to the GUI for refining global distortions with the lattice parameters.

- **Distortions units:** Two radio buttons are available to switch the display of the distortion values in percentage or pixels. The pixel units are included mostly for historical reasons. They depend on the calibration constant, while the percentage values do not.
- **Distortion refinement modes**:
  - The **Standard mode** (default tab) is sufficient for most data sets (all of them).
  - The **Expert mode** contains additional distortion components. Most of them are used for development purposes, and it is not recommended to mingle with them, unless you know well what you are doing.

The two pictures below (Figure B-32) show the same distortions parameters (in percentage) displayed in the "Standard" and the "Expert" modes. The radial g*Sg parameter is only meaningful for precession geometry. Detailed information about the distortions used in PETS2 is available in Brazda et al. (2022)[5] and a few examples are given in the JANA2020 cookbook.

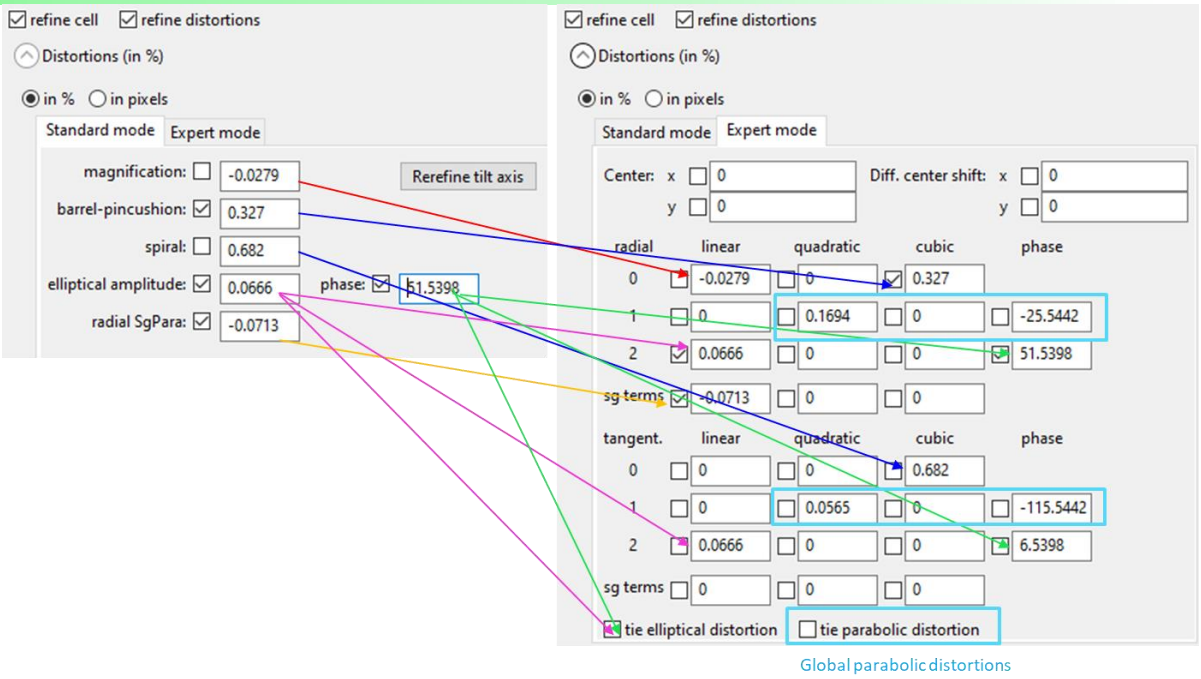


Figure B-32 Global distortions shown in the standard and the Expert mode. Those distortions were obtained frame-by-frame and replaced into global distortions. Magnification is otherwise never refined with the lattice parameters.

➢ What distortion to refine?

By default, (Figure B-33), the barrel-pincushion, the spiral and elliptical distortions (amplitude and phase) are refined with the unit cell parameters. The radial g*Sg parameter is only meaningful for precession geometry.

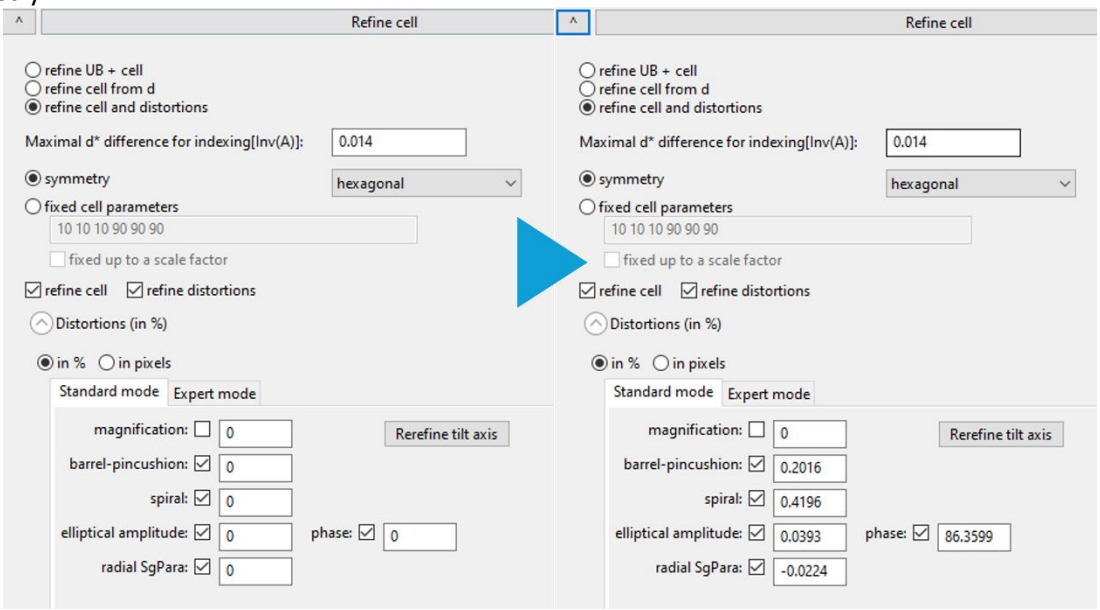


Figure B-33 example showing the default settings for "unit cell + global distortions" refinement for a hexagonal unit cell (PEDT data). Data quality was good enough (+ high symmetry) to include the global elliptical distortions.

- **Barrel-pincushion and spiral** distortions are robust and can always be refined.

- **Elliptical distortions** can be globally refined for good data quality. For poor quality data sets with large misorientation of the frames, leave out the global refinement of the elliptical distortions before any prior optimization of the frame geometry. They strongly correlate with the unit cell parameters, especially without symmetry restriction. Applying a symmetry restriction helps to reduce correlations.
- **Magnification** (scale) correlates with the lattice parameters and is never globally refined with the lattice parameters. The values shown in Figure B-32 comes from the frame-by-frame refinement (see section), and the average value from all frames replaced the initial one (because we checked the option “replace global distortions in the geometry optimizations (See Section Optimize frame geometry).
- **Radial SgParameter** is only valid for precession geometry.
- **Parabolic distortions** are not refined globally in the standard more. They are due to the wrong eucentric focus of diffraction length during the experiment. They are only refined frame-by-frame (geometrical optimization) and the average value can appear in the expert mode only.

An option “**Rerefine tilt axis**” is on the right side of the distortions panel. The tilt axis angle ω *and* δ are usually optimized once more after determining the distortions parameters, especially if the spiral distortion changes significantly.

B.3.5.f. Finish

Press Finish to go back to the main commands panel and continue with the data reduction.

The last selected unit cell will be the active one in the rest of the data reduction.

All unit cells are written at the end of the pts2 file. The older celllist file was discontinued. Pets2 is still able to read the old smr and celllist files, but they will be rewritten after the list of frames in the pts2 file, under the same format as before.

In the pts2 file, the active cell will appear as “active” in front of the cellItem.

B.3.6 Process frames for integration

This command will process the integration of intensities using the lattice parameters and specific parameters listed in the menu below the command.

Pull-out the menu by hitting one of the arrows on each side of the function button to display the parameters (Figure B-34). Hit the command to start processing frames for integration.

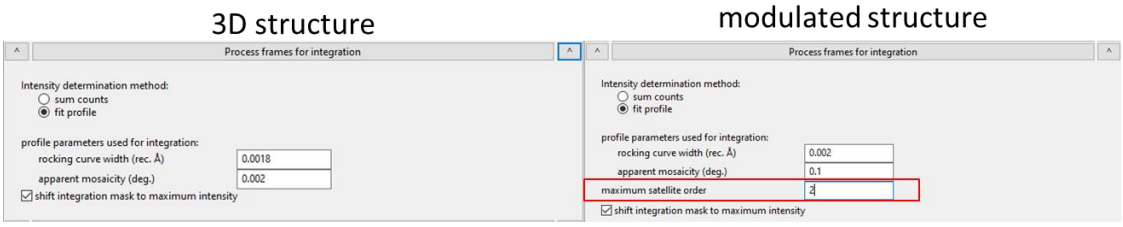


Figure B-34

This function has two sections:

- **Intensity determination methods**: either sum count or fit profile.

- **Profile parameters** to set: *RCwidth* and the *apparent mosaicity* used for the integration.
- For modulated structures (when one/more modulation wave vector is defined): an additional parameter appears to set the maximum satellite order for integration (Figure B-34 right).

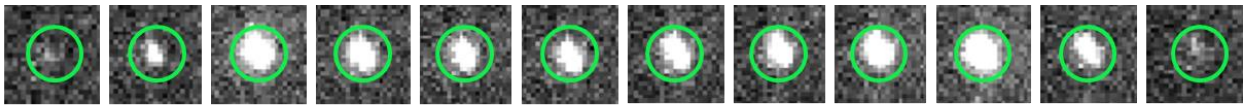
B.3.6.a. Intensity determination methods

Radio buttons are used to select either "sum counts" or "fit profile" methods.

- **Sum counts**: the intensity is determined from the total counts measured inside the mask (S) in all frames the reflection is expected minus the backround contribution (Figure B-35).

$$I(hkl) = \sum_S p_{hkl} - \sum_S b_{hkl}$$

With p_{hkl} the pixel value, and b_{hkl} the pixel background value.



Same reflection hkl present on several consecutive frames

Figure B-35 Example of one hkl reflection present on several consecutive frames with the integration mask (green).

- **Fit profile**: this option uses the average reflection profile(s) defined during the "peak search" procedure from the strong reflections. The APPs are used in integration, and their position (*x*, *y*) and intensity are refined for each reflection. The "fit profile option" is slower than the sum option but usually provides (slightly) better results. The difference is especially important, if reflections on the frames overlap, for weak or saturated reflections. Fit profile is the default option and should work better unless data show very high mosaicity and diffuse scattering.

For better results, the "fit profile" option should be used together with the "**shift integration mask to maximum intensity**" (at the bottom of the menu). For the "sum counts" option, the user can try with and without checking the shift option box.

The **"shift integration mask to maximum intensity**" allows the integration masks to be shifted a little from their ideal positions to reach the real position of the reflection (center of mass of the intensity). This option works well with the "fit profile" integration method. With "sum counts" and rather poor data, it can catch the wrong maximum.

B.3.6.b. Profile parameters for integration

The two main profile parameters of the Rocking curve are used for integration: **the rocking curve width** ($Å^{-1}$) and the **apparent mosaicity** (deg.) (Figure B-34). The Rocking curve is visible on the main panel in the Graph tab. Those two profile parameters are repeated in the section Optimize reflection profile. They are used to estimate the angular extent of the reflections (i.e. on how many frames the reflection is present)

As the process for integration runs, three plots related to the data resolution are produced and displayed in the Graphs tab.

- "Integration: Resolution" graph: for each frame the length of the maximum diffraction vector (d* in reciprocal Å) is given for 2σ, 3σ and 5σ.
- "I/sigma in two shells": for each frame the mean I/sigma is given for reflections with d*< 0.5 $Å^{-1}$ (inner shell with lower resolution), and d*> 0.5 $Å^{-1}$ (outer shell with higher resolution). The default threshold between the inner and outer shell is set to 0.5 $Å^{-1}$ and can be changed in the input pts2 file after the keyword "resshellfraction".
- The Rocking curve or Camel plot for all the data set appears in the Graphs tab at the end of the integration and shows a rocking curve for all reflections by resolution range for some defined I/sigma(I).

See Sections B.4.3.d, for further details about the graphs.

B.3.7 Optimize reflection profile

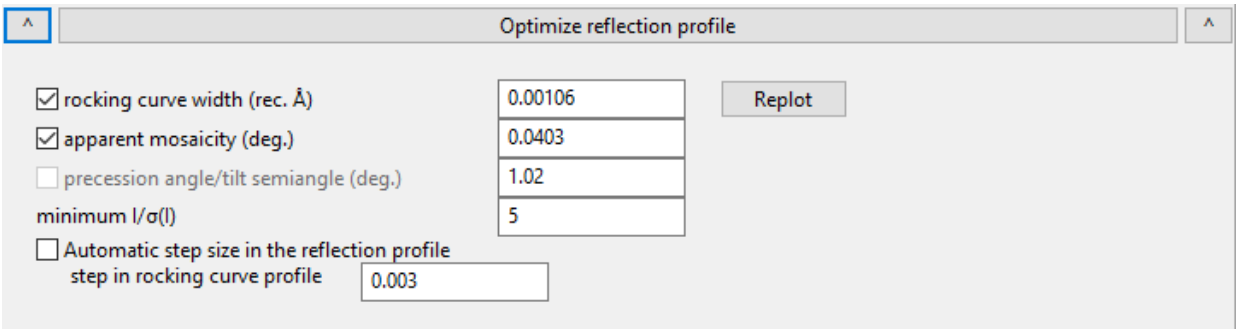


Figure B-36

This is where the global reflection profile parameters are displayed and can be optimized (Rocking curve or Camel plot) (see Figure B-36). They will influence the calculated rocking curve (see the red curve of the Rocking curve in the Graph tab Figure B-37) and the extend of the integration mask during the integration (= process frames for integration). Note that RC width and apparent mosaicity are repeated in the section "process frames for integration".

3 parameters will influence the shape of the calculated Camel plot:

- **Rocking Curve width ($Å^{-1}$)** (RCwidth) (global value). This parameter can be refined.
- **Apparent mosaicity (deg)** (global value). This parameter can be refined.
- **Tilt-semi angle or the precession angle (deg)** depending on the geometry of the experiment – not refined.

2 parameters affect how the curve is displayed:

- **Minimum I/sigma(I)** is used to select the reflections involved in the Camel plot calculation.
- **Automatic step size** in the reflection profile. A too small number will give a very noisy observed Camel (blue curve).

The RCwidth and the apparent mosaicity parameters can be refined by pressing the command button. After the refinement, the RC plot is automatically refreshed in the Graphs tab. They may also be changed manually. It is easier to manually set the RCwidth looking at the low-resolution part of the Camel plot, and the mosaicity with the higher resolution part. After changing the parameters manually, use the "Replot" button to refresh the Rocking curve profile on the Graphs tab.

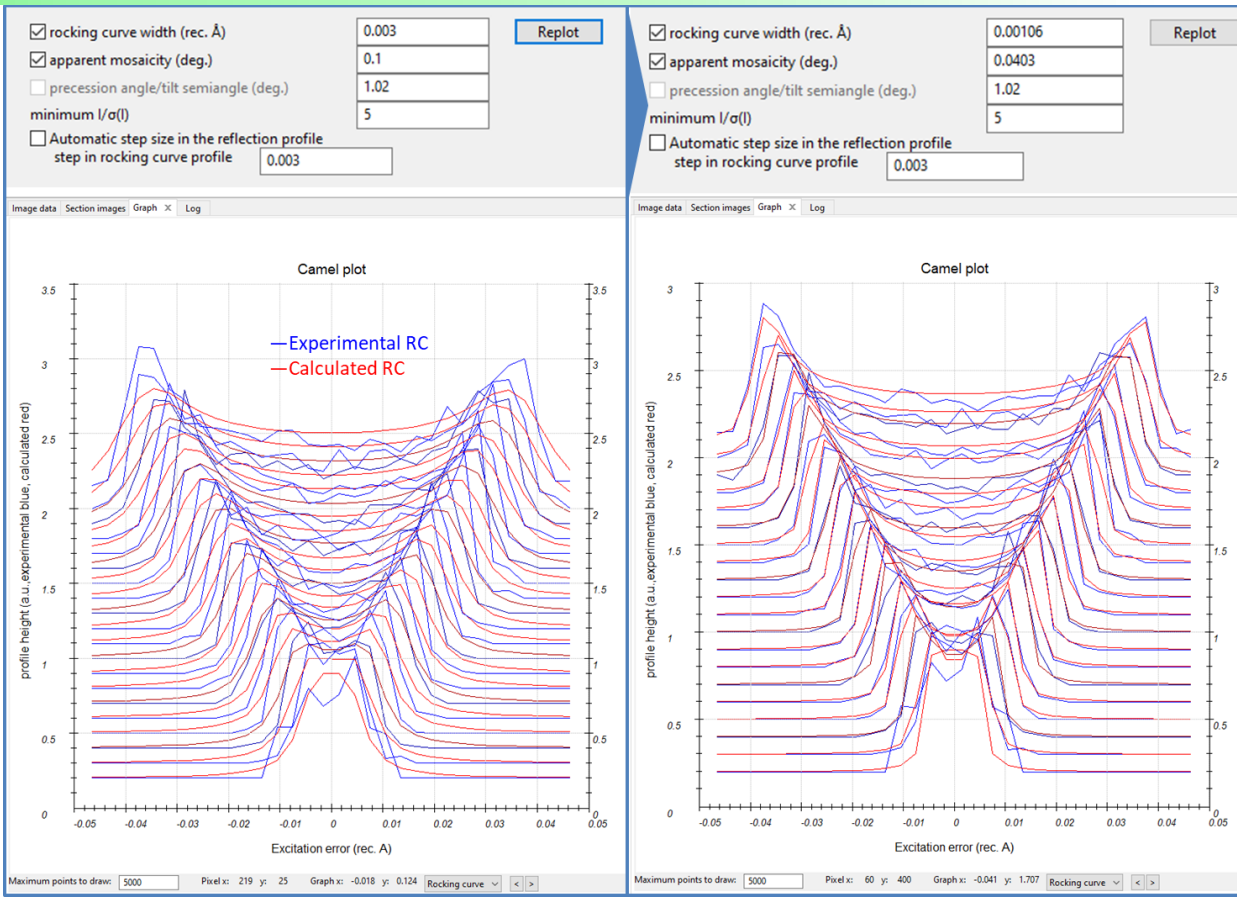


Figure B-37

B.3.8 Optimize frame geometry

This section will optimize the frame-by-frame geometry of the experiment, including orientation angles (alpha, beta and omega), center of the diffraction patterns, RC width and apparent RC mosaicity, and frame-by-frame distortions.

Below are the default settings. As mentioned above, 3D ED data suffers from the misalignment of the frames and 2D optical distortions. Some distortions are globally refined (Section B.3.5.d) with the unit cell, they can also be refined frame by frame in that section.

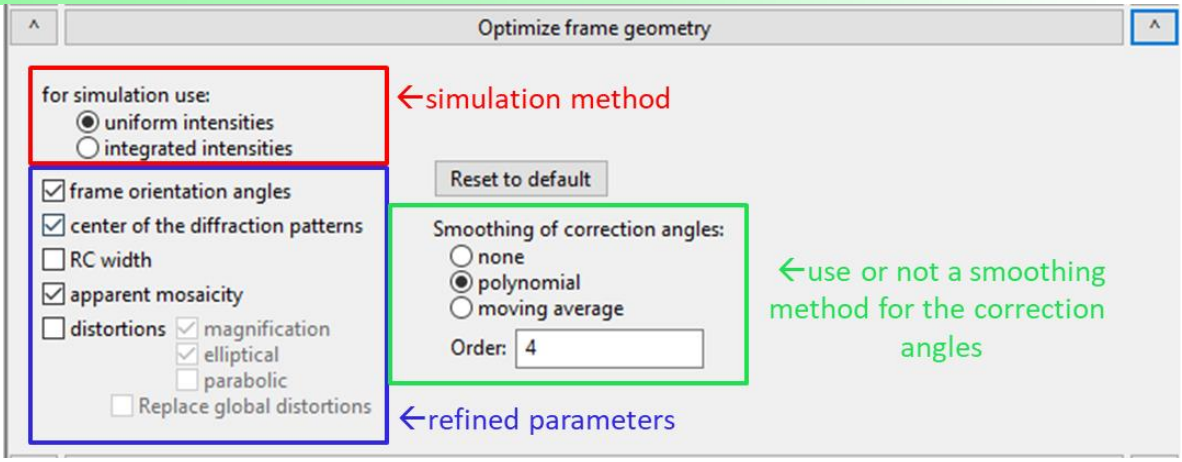


Figure B-38

Hit the command to start the optimization or

Pull down the menu (arrows) to set what to refine and adjust the parameters. The default parameters are shown in Figure B-38.

B.3.8.a. Optimize frame geometry: Simulation methods

The routine generates diffraction patterns based on the previous finalized integration or theoretical uniform intensities. The least-squares minimization routine then tries to reduce the pixel-wise differences of the generated pattern to the background-subtracted experimental diffraction pattern. You can find more details about the method in Palatinus, L. et al. (2019). Acta Cryst B 75, 512-522.

For simulating the frames, PETS2 can use two methods:

- **uniform intensities:** PETS generates uniform intensities with a decay relative to the resolution. This is the safest method for poor quality data or when the observed rocking curve is not fitting nicely the calculated one (high misorientation of the frames). This option can give a noisier optimization.
- **integrated intensities.** Integrated intensities can only be used after a (reasonably successful) Finalize integration step to create the file with integrated intensities necessary in the optimization (see section B.3.9.). This option is used when the Rocking curve is reasonably sharp and can lead to good integrated intensities. When possible, this method gives more precise values.

If you are unsure about the data quality, use uniform intensities first and refine only the orientation angles and the center of the diffraction pattern before including distortions and/or RC parameters in another round of optimization.

B.3.8.b. Optimize frame geometry: Geometrical parameters

Checkboxes are used to select what is refined (blue area in Figure B-38). Possibly,

- The **frame orientation angles** (**Δalpha**-tilt and **Δbeta-tilt**, **Δω** axis),
- The **centers of the diffraction patterns** (**Δx**, **Δy** in pixels).
- The **frame-by-frame RC width** or **apparent mosaicity** (they are correlated),
- **Frame-by-frame distortions**: the distortions that can be refined frame-by-frame are the magnification, the amplitude and the phase of the **elliptical distortion**, and the amplitude and the phase of the **parabolic distortion**.

By selecting "**Replace global distortions**" (blue area in Figure B-38), the three global distortions parameters of the indexing panel will be replaced by the average values of the frame-by-frame optimization.

B.3.8.c. Optimize frame geometry: Smoothing options

The refinement of the tilt-angles (Δalpha, Δbeta tilts, and Δomega) and the distortion parameters can be stabilized using a smoothing method. The smoothing avoids outliers for frames with fewer reflections. Two smoothing algorithms can be chosen.

- **The polynomial method**: the order corresponds to the order of the polynomial equation. In Figure B-39, order = 4 means that the curves with the correction of the angles are described with a polynomial function $Ax + Bx^2 + Cx^3 + Dx^4 + E$ with (A, B, C, and E constants). This is now the default option with order 4.
- **The moving average** option: the order corresponds to how many consecutive frames are used to smooth the curve (Usually from 5 to 10 for precession geometry with 1 degree tilt step, can be 10-20 for continuous rotation geometry and smaller Δα).

The default method is now the polynomial one with order 4. For very misaligned data, you might need more freedom using the moving average method. See Figure B-39.

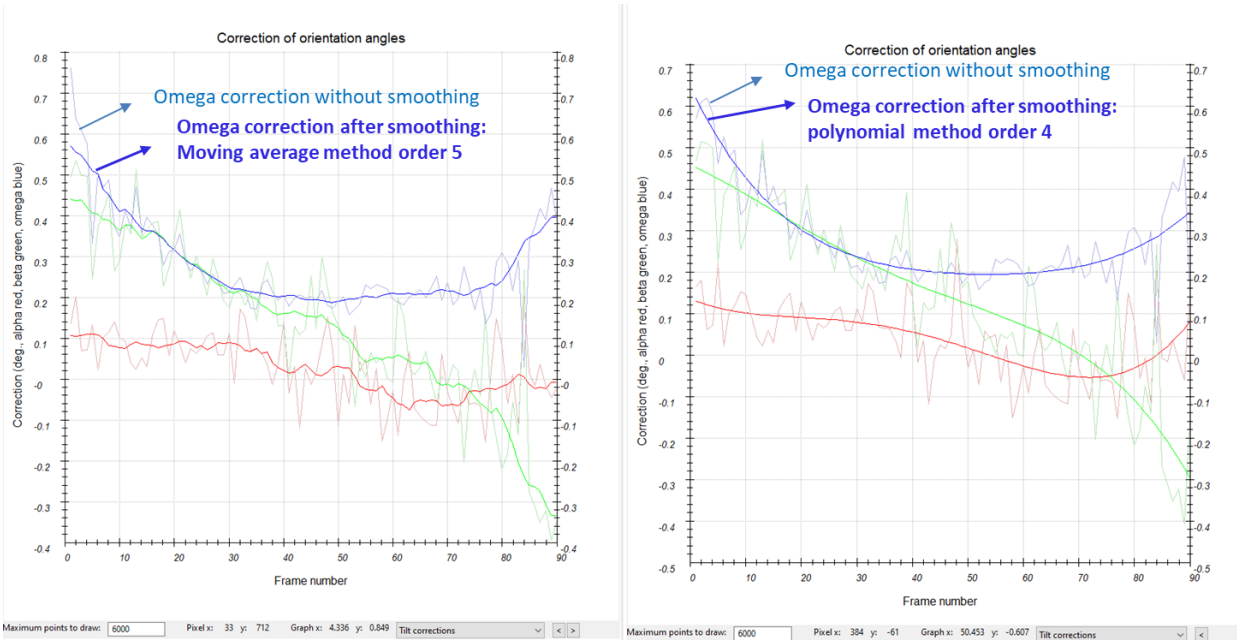


Figure B-39

"**Reset to default**" will reset the frame-by-frame optimization and go back to the "initial" values. Initial values are the initial angles of the input pts2 file. If you refine the geometry several times, "Reset to default" brings you back to the very initial values, not the previous ones.

During the optimization, different plots will be produced and visible in the Graph tab of the main panel:

- Tilt corrections plot
- Origin correction plot
- Frame-by-frame distortion plot
- Profile parameters plot.

(More information on the plots in the dedicated section B.4.3.)

B.3.9 Finalize integration

This function is used at the end of the data reduction to generate the the *hkl*-type files dedicated to structural analysis with the kinematical approach (solution + kinematical refinement) or considering the dynamical theory of diffraction (dynamical refinement)[6–8].

This command will be affected by several parameters and options described below.

Hit the command to finalize the integration and access the options by pulling down the menu using the arrows on the side of the command button (Figure B-40). For each type of integration (kinematical or dynamical), parameters must be chosen and adjusted. However, default parameters should lead to reasonable results. The parameters related to kinematical integration are broken down into sections, as shown in Figure B-40.

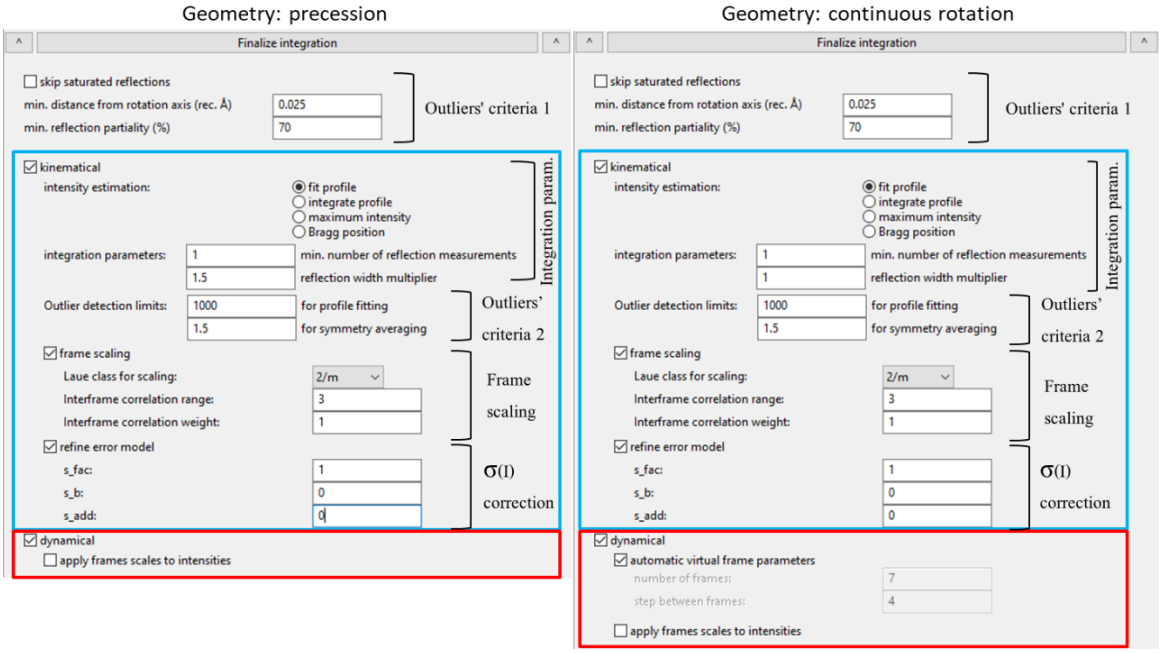


Figure B-40

"kinematical" integration and "dynamical" integration are selected by checkboxes (Figure B-40).

The output files produced are listed below:

- Jobname.hkl – Kinematical approach –This file is a simple list of reflections *h k l* (*m n p*) associated with their intensity and sigma(I) without a header. This file can be used in any software for solution and refinement, considering the kinematical approximation.
- Jobname.cif_pets – Kinematical approach – The same list as mentioned above with an additional header giving additional unit cell and experimental information. This type of file is only recognized by JANA2006/2020 (as far as we know). the .cif_pets file now contain several CIF entries containing the data processing statistics, that are further automatically imported into Jana.
- Jobname.shelx.hkl – Kinematical approach – is the hkl file in the SHELX format

- Jobname_dyn.cif_pets – Dynamical approach – File dedicated to the dynamical refinement in JANA2020 containing a header with unit cell and some experimental parameters. This file contains a list of reflections hkl (mnp) with their intensity and sigma(I) for each (overlapping virtual) frame. See Palatinus et al. (2015a,b)[6,7] and Klar et al. (2023)[8] for more details. the .cif_pets file now contain several CIF entries containing the data processing statistics, that are further automatically imported into Jana.

B.3.9.a. Outlier criteria for kinematical and dynamical integrations

- **Skip saturated reflections**: a check box is used to allow or discard the saturated reflections in the output files (kinematical and dynamical). Saturated reflections should be included for the structure solution and dismissed for the refinements (kinematical or dynamical).
- **Minimum distance from rotation axis** (rec. Å): Reflections found below that limit in distance from the rotation axis are not included in the final files. The area of exclusion (green) can be displayed on the frame together with the resolution rings by selecting the icon “rr” at the bottom of the Graphs tab (Figure B-41).
- **Minimum reflection partiality (%)**: reflections which intensity profiles are covered by less than the threshold value in percent, are omitted from the final integration files. The default value is 70%.

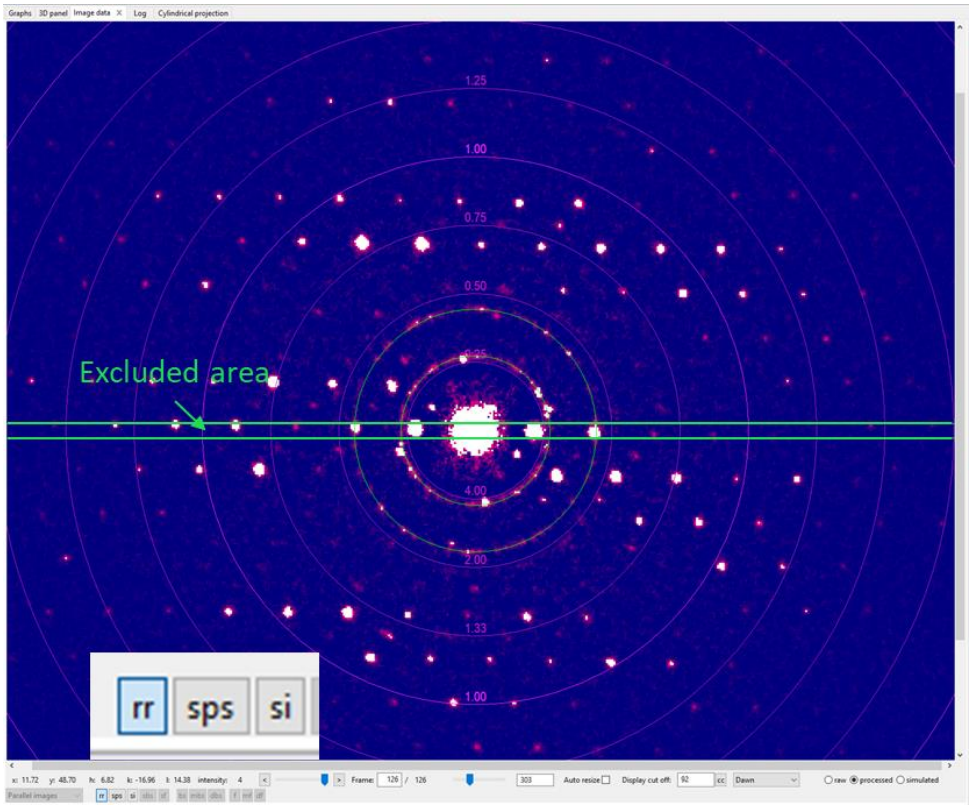


Figure B-41

B.3.9.b. Intensity estimation for kinematical integration

There are 4 possible options to estimate the kinematical intensities and selected with the radio buttons.

- **Fit profile**: This option corresponds to the red dashed area in Figure B-42. The intensity of an *hkl* reflection is defined with the calculated Rocking curve profile on all the resolution range according to the data and the two RC parameters (sigma and moisaicity), and the value of the semi-angle or precession angle. Fit profile integration should produce the best results for medium to high-quality data.
- **Integrated profile**: the intensity of one reflection *hkl* is determined by the area (volume in 3D) defined by all the measurements for this reflection found in consecutive frames (blue area in Figure B-42).
- **Maximum intensity**: the value of the measurement with the highest intensity for one given hkl reflection is used as a global intensity for this reflection (yellow measurement in Figure B-42). Reserved for special cases, generally not recommended.
- **Bragg position**: The intensity of the *hkl* reflection is estimated from the closest measurement from the Bragg position (green measurement in Figure B-42). Reserved for special cases, generally not recommended.

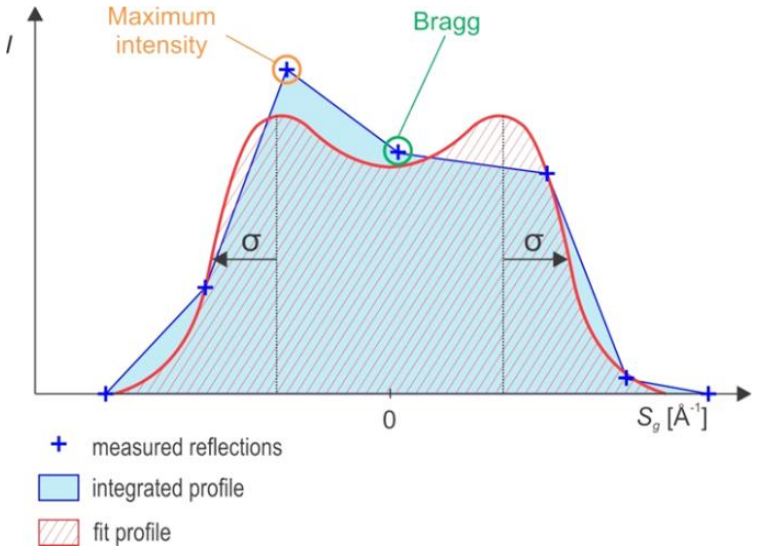


Figure B-42 *Integration options illustrated with a basic Camel plot profile drawn for a sample reflection with precession.*

B.3.9.c. Finalize integration options: additional integration parameters (kinematical)

Two additional integration parameters which are kept most of the time to the default value:

- ***The minimum number of reflection measurements*** (default value 1): how many times a reflection *hkl* must be present on consecutive frames to be considered in the integration. Increasing the number might improve the data quality, but it may reduce the data coverage, especially with data with large tilt steps. The more one reflection hkl is measured on different frames the better its intensity profile can be described, and its intensity properly evaluated. For 1, all reflections are considered. For structure solution, it is often better to keep all reflections. A more selective approach can be used for refinement to avoid outliers.
- ***Reflection width multiplier*** (default value = 1): to extend the integration beyond the Sg limit set by the reflection profile. The reflection width multiplier is how many times the standard deviation of the reflection width is multiplied to extend the integration of the reflection along Sg. The latest option

can be used to compensate for big gaps between frames as is often the case for static frame experiments.

B.3.9.d. Outlier detection limits for kinematical integration

Removing outliers relies on two parameters.

- ***The outlier detection limit* for profile filling** is based on the profile fitting (default value: 1000). Limit for rejecting reflections with too large discrepancy between the intensity on a frame and the fitted profile. Relevant only for the fit profile option.
- ***Outlier detection limit for symmetry averaging*** (default value: 1.5): based on the sigma(I). Limit for rejecting reflections with too large discrepancy between its integrated intensity and the average of all symmetry-equivalent reflections. Another way to describe this outlier's criteria is when the sigma(I) of a reflection is too far from a perfect normal distribution (perfect normal distribution = red line on the plot in Figure B-43).

Rejected outliers appear in red in the Normal probability plot in the Graph tab (Figure B-43). Setting too low value for the Outlier detection limit for symmetry averaging might have a detrimental effect on data coverage as too many reflections are kicked out.

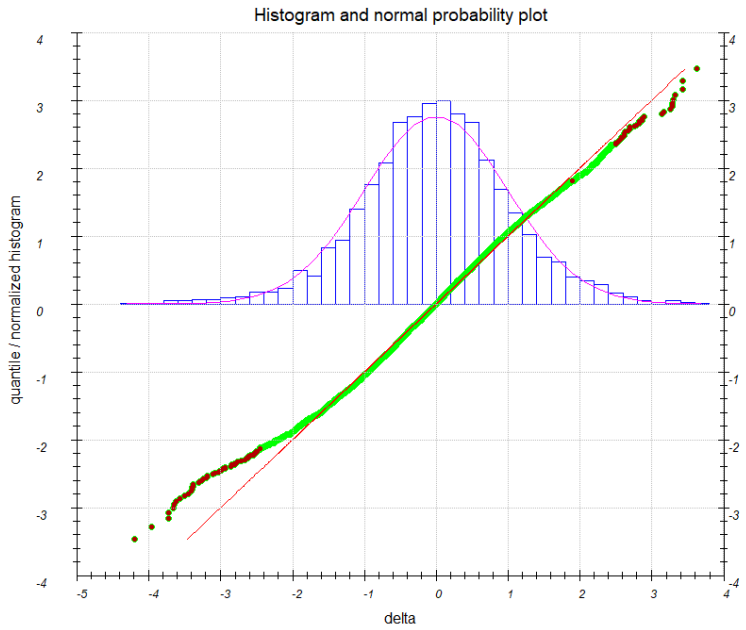


Figure B-43

B.3.9.e. Frame scaling (kinematical)

Frame scaling is used to correct for the uneven illumination of the crystal and other effects affecting the intensities (thickness, orientation). Frame scaling is checked by default and relies on three parameters: The Laue class, the interframe correlation range and the interframe correlation weight (Figure B-44). The scaling is only relevant for kinematical integration.

- **Laue class.** When the frame scaling is checked, select the appropriate Laue class from the list if known. For unknown Laue class: select the option "**Auto**" in the top of the Laue class list. In that case, PETS2

will evaluate the Laue class to apply for frame scaling. The "Auto" option, will be replaced by the best Laue class guess after "Finalizing integration".

- **The *interframe correlation range*** corresponds to how many consecutive frames are considered around the frame to scale. To scale frames independently, set 0. The default value is 3, which can be increased for most datasets. Reasonable interframe correlation range can be typically from 3 to 10 for precession data and from 5 to even 20 for continuous rotation (depending on the Δα). Two small numbers will give noisier plots.
- ***The interframe correlation weight*** indicates how strongly the scales are forced to be close (1 = strong correlation). The result of the frame scaling is visible in the related graph below (Figure B-44). For no correlation, set 0.

The frame scaling results are given as a plot representing the Frame scale per frame in the Graphs tab. The frame scales depend on the crystal shape and thickness and the orientation.

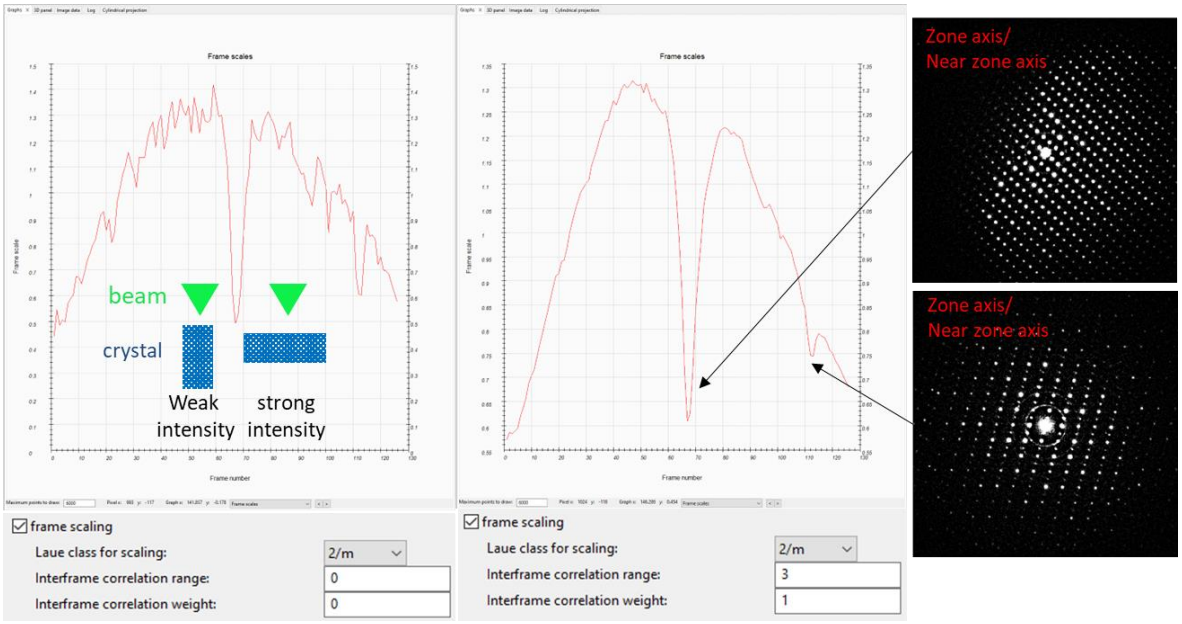


Figure B-44 Two frame scaling optimization for the same dataset with different values for the interframe correlation range and weight. Thicker areas of the crystal (often at high tilt angle) give lower frame scales than thinner parts. An isolated drop of the frame scaling usually indicates a zone axis or near zone axis pattern.

B.3.9.f. Error model to correct sigma(I) (kinematical)

The error model is used to correct the sigma(I). With the initial error model, intensities I_{hkl} are given with their error $\sigma(I_{hkl})$ based on the counting statistics. It assumes a normal distribution of errors which is wrong. Sigmas are corrected to bring back the distribution to a normal distribution. For that PETS2 uses symmetrically equivalent reflections. The refinement finds optimal modifiers (S_{fac}, S_B, and S_{add}) of the counting-statistic sigma(I) to produce a normal distribution of residuals following the equation:

$$\sigma'\langle I_{hl}\rangle(corrected) = S_{fac}\sqrt{\sigma^2\langle I_{hl}\rangle + S_B^2\langle I_h\rangle + S_{add}^2(\langle I_h\rangle)^2}$$

The default values are S_{ac} = 1, S_B = 0 and S_{add} = 0. On the histogram and probability plot (Figure B-45), a satisfying refinement of the modifiers brings the green curve close to the perfect Gaussian distribution (=

red curve). As a side effect, this optimization tends to decrease the number of observed reflections. For more information see Khouchen et al., 2023[9].

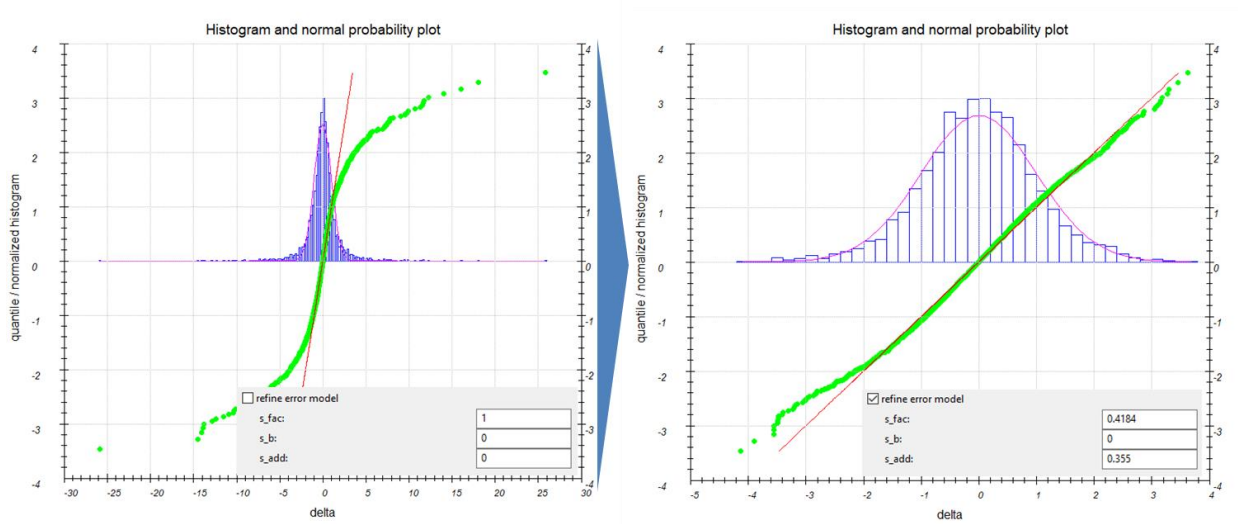


Figure B-45

B.3.9.g. Dynamical integration options

- ***Apply frames scales to intensities:*** For all geometries, the frame scaling can be applied to the dynamical frames by checking the box "apply frames scales to intensities". When the scaling is applied in PETS2, the scaling later defined in JANA2020 frame by frame should be closer to a flat curve.
- ***Automatic virtual frame parameters***: For continuous rotation or static frame geometries, two additional options define how to merge consecutive frames to create overlapping virtual frames (*OVF*) (Figure B-47). This allows reflections to be properly integrated.

By checking the box "automatic virtual frame parameters", PETS2 will set the two following parameters automatically depending on the value of the tilt semi-angle.

- The "**number of frames**" is the number of consecutive frames merged to create one virtual frame.
- The second parameter called "**step between frames**" defines the increment between 2 virtual frames and, thus, the overlap between the *OVF*s.

Typically, real frames are merged to create *OVF* covering about 2.5-3 degrees. A safe overlap can be set to one half of that value. In the example in Figure B-47, the data are from continuous rotation and a Δα tilt = 0.4 degree. By merging 7 frames, each virtual frame covers 2.8 degrees with a 1.6-degree step between two frames, leading to an overlap of 1.2 degree.

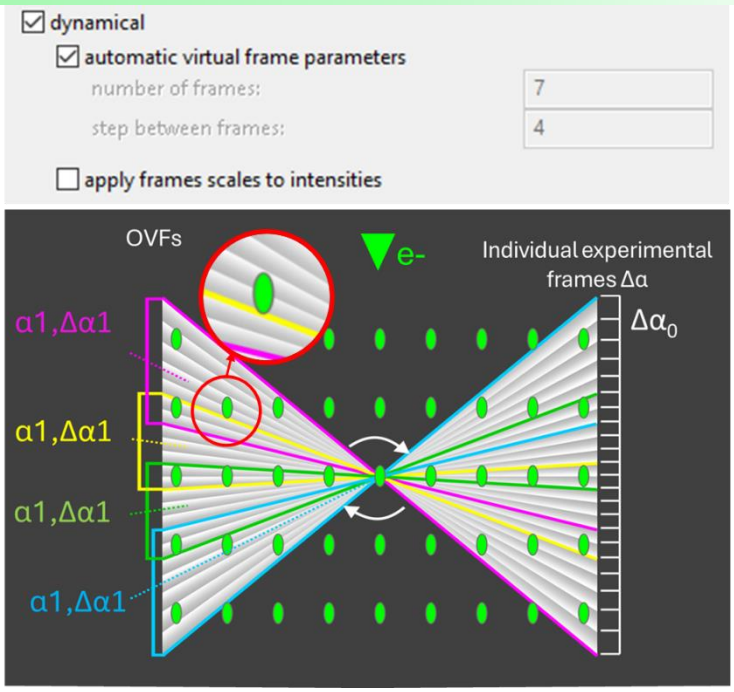


Figure B-46 Scheme showing the concept of overlapping virtual frames. One reflection (red insert) is not integrated by one frame on Δα. Its intensity profile can be better integrated when several consecutive frames are merged into virtual frames.

B.3.10 Serial electron diffraction

Under development, contact us if you experience bugs. We strongly suggest saving at every step so as not to lose the progress of the data reduction in case the program crashes.

B.3.10.a. Introduction

REQUIREMENT: This option can be used when the **lattice parameters** and the **Laue class** are known.

FOR WHAT TYPE OF DATA? The serial electron diffraction data reduction can be used for electron diffraction frames with or without precession or with continuous rotation.

- collection of individual frames randomly oriented on different crystals for very beam sensitive materials (with or without precession).
- combination of several short tilt series (continuous rotation/precession/static geometry) which are individually insufficient to define the unit cell (no merging possible).
- combination of an incomplete data set for which the unit cell can be defined with additional individual randomly oriented frames from different crystals to complete the coverage.

B.3.10.b. Available options

Below is the basic description of the “Serial ED” parameters found when the menu below the command is pulled down.

Before doing anything with the “serial ED” command, make sure the expected Laue class is indicated as shown in Figure B-47.

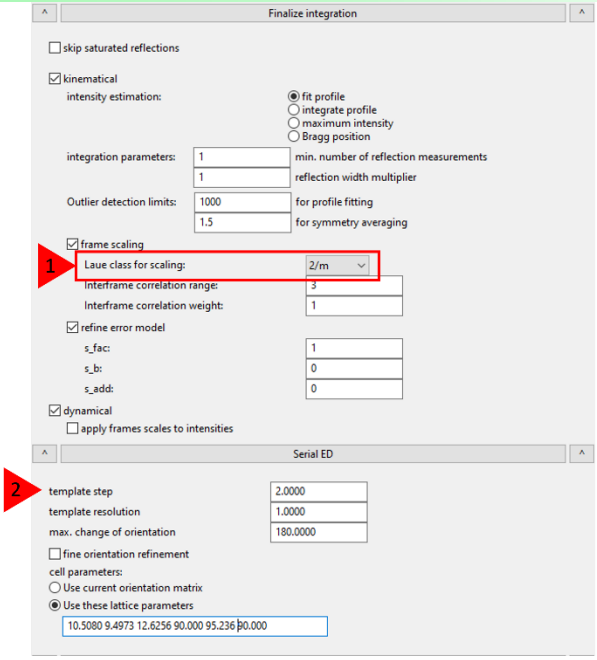


Figure B-47

The indexing of Serial ED data is based on template matching. This is why a Laue class, and a unit cell are needed to simulate frames. The simulation uses "uniform" intensities with a decay relative to the resolution.

- ***Template step***: by default, the template step is 2 degrees. This value should be enough for most experiments. For very sharp reflections where a small change in orientation brings a lot of change in the pattern the value can be decreased. For precession data, 2 degrees by default are usually sufficient, for small semi angle and continuous rotation data, a 1-degree step might be more reasonable. Note that the simulation will be more time-consuming with a smaller angle.
- ***Template resolution:*** in reciprocal angstrom (d*). The default value is 1 $Å^{-1}$. Increasing the resolution of the simulation will also slow down the process.
- ***Maximum change of orientation:*** 180° (default value) will cover all the diffraction sphere. For higher symmetry, a smaller part of the simulated data can be used.
- ***Fine orientation refinement***: when the box is checked, the frame orientation is refined directly (20 000 templates are generated). It is useful in the case of pseudo symmetry, with several closely matching orientations (set of best templates).
- ***Unit cell***: radio buttons are used to select either you already have an orientation matrix with "use current orientation matrix" (an existing celllist file from an older data processing), or if you start from scratch "use these lattice parameters". In the latest case, indicate the expected lattice parameters.

Note: those lattice parameters are not saved!

B.3.10.c. How to proceed in PETS2

To start a new pets2 project with serial ED data, store all the collected frames in one directory even though they come from different crystals.

Open Pets2 and create a New project (File/New).
In the new window, name the project, indicate the project directory, and the project frames' directory.
NEXT;
Fill information on the binning to apply, the wavelength, the calibration, the geometry, and the precession or tilt semi angle if any.
NEXT;

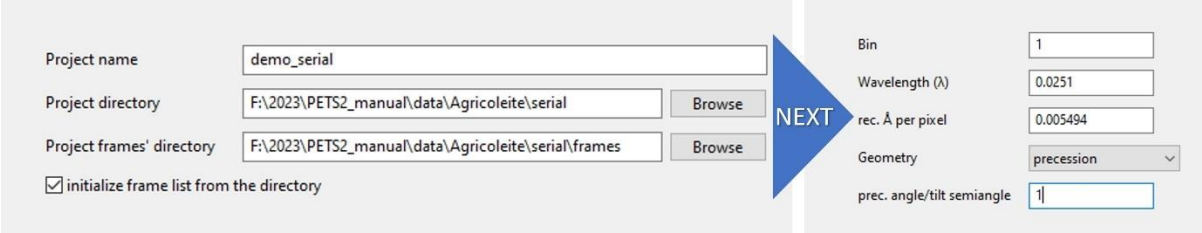


Figure B-48

Another window opens showing the list of frames, press OK. Frames are not expected to be a tilt series, so, we don't set angles.
As this pets2 project is created from scratch, once you are back to the main window, go into parameters and manually set a reasonable reflection diameter, the noise parameters, and the detector saturation limit. If you need to change the detector type (to remove the cross between the four quadrants of the cheetah ASI detector for example), you must save, close the pets2 project and change the detector type directly in the pts2 input file.
Open PETS2 again.

Go into peak search parameters to adjust settings according to your experiment and run "peak search". All peaks are collected as well as the d*. At the end of this procedure, the graph showing the maximum resolution peak for each frame, as well as the graph showing the pattern center are given in the Graph tab of the Main panel (as usual).

Before going to the Serial ED command, set the Laue class in the parameters below the command "Finalize integration". Here we set 2/m.
Pull down the menu of parameters of the "Serial ED" command.

Set the parameters for the serial ED according to your data. In our example, we kept all parameters by default (Figure B-47) and we indicate the unit cell parameters as we start a new project.

Hit the command "Serial ED". The console gives the evolution of the simulation (Figure B-49).

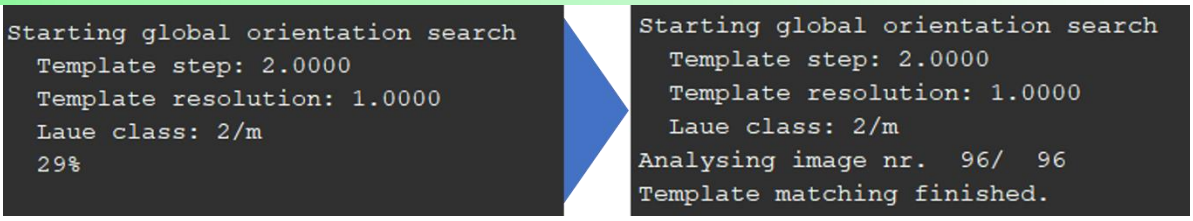


Figure B-49

Do “Peak analysis” and go to “Find unit cell and orientation matrix” to check the 3D reconstruction and the success of the matching process. Add the centering if present and indicate the Bravais class.

Do not refine the cell now as the frames’ orientation might not be accurate at this stage.

Finish.

Go to the options in “Process frame for orientation”. Choose the intensity determination method (sum count of fit profile), set reasonable Rocking curve parameters for a first run (*RC width* ≈ 0.003 $Å^{-1}$; *apparent mosaicity* ≈ 0.1 deg.) and the possibility to shift or not the integration masks.

At this stage, go to the Image data tab of the main panel, and select “si” to visualize the positions of the integration masks on the frames. The serial ED procedure might have failed to determine the orientation of some frames (usually when there are not enough reflections on the frame). In that case, remove those frames from the data reduction. To discard a frame, use the Image option panel and select “frame dialog”. Uncheck the box in front of the frames to remove from the data reduction as shown in Figure B-50.

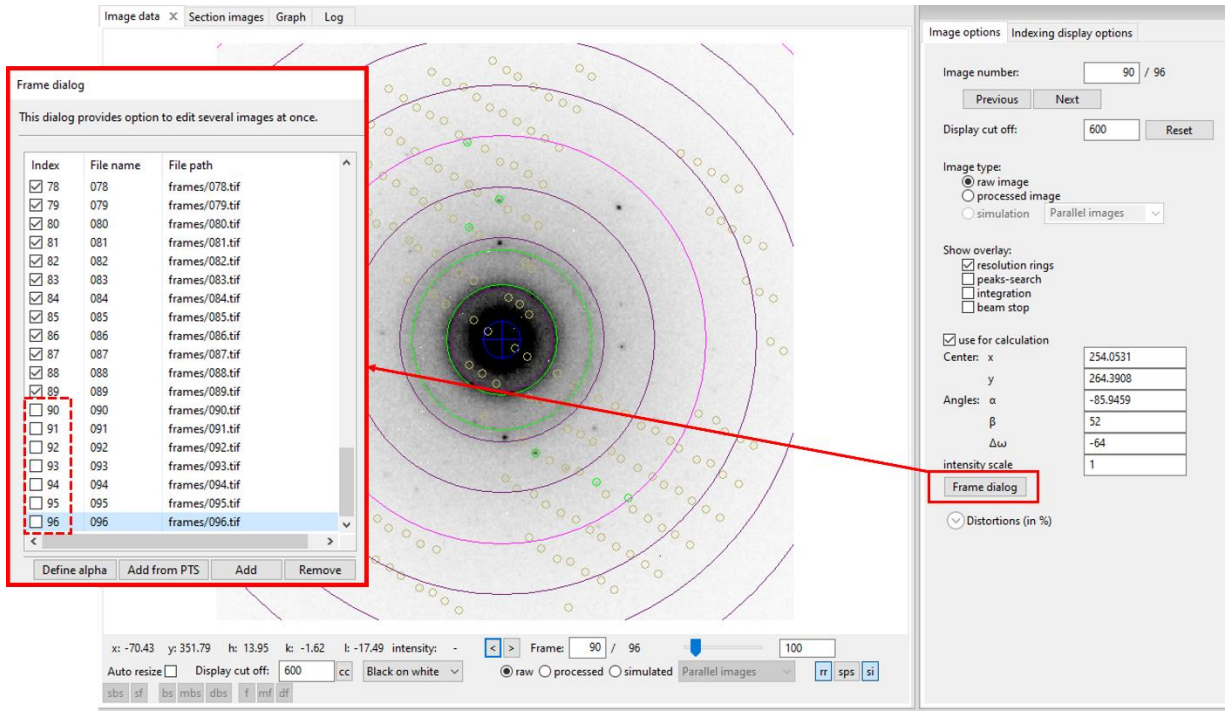


Figure B-50

Rerun “process frame for integration” without the badly indexed frames.

Adjust the reflection profile parameters as much as possible.

Pull down the menu of the command “Optimize frame geometry” and select “uniform intensities” as a simulation method and check the boxes “frame orientation angles” and “center of diffraction patterns” only. As the frames are not expected to be a tilt-series, do not use any smoothing method. The graph representing the correction of the correction angles can be chaotic and without trend.

Once the geometry is refined, process once more the frame for integration. The Rocking curves should get sharper as in the example below (Figure B-51).

To “Finalize integration” with the frame scaling, the interframe correlation range must be 0 as the frames are not a tilt-series. The scales are not correlated (see insert in Figure B-51).

The data set can be now processed as a normal tilt series data set. You can do a finer analysis, refine distortions, the unit cell or the geometry further.

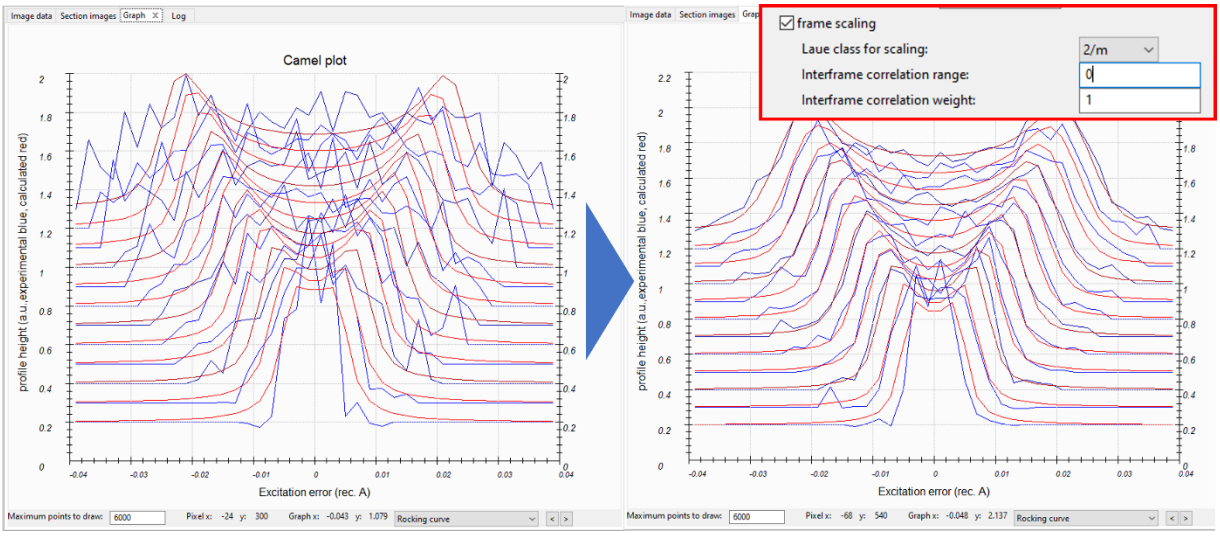


Figure B-51

B.3.11 Generate a 3D map

Hit the command to create a 3D map of the reflections with their associated intensities.

Pull down the menu to adjust the parameters:

- The **pixel size** (rec. Å): default value = 0.01 rec. Å
- Select one of the three available **output file formats** (Figure B-52): xplor, ccp4, and hdf5.
- The **symmetry can be considered in the reconstruction** according to the Laue class used for the frame scaling by checking the box.
- By default, the **background is subtracted** from the 3D reconstruction but can be included by unchecking the checkbox. Including the background is necessary e.g. when the reconstruction is made for the analysis of diffuse scattering.

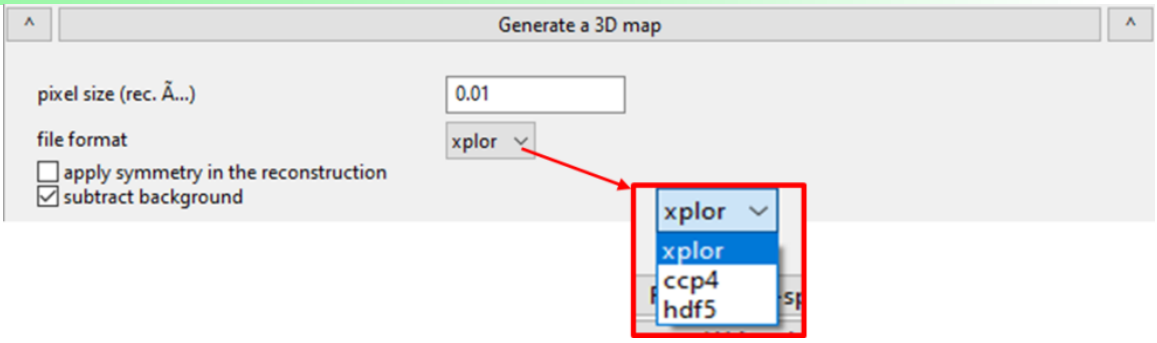


Figure B-52

B.3.12 Reciprocal-space sections

Hit the command to create the sections or pull down the menu to access the options.

In this section, radio buttons are used to select:

- The **standard set**: The standard set of reciprocal-space sections is defined according to the Bravais class previously selected in the indexing section and displayed in the Status bar at the bottom of the PETS2 window. It includes all the necessary sections to determine the symmetry for a given Bravais system.
- The **user-defined set**: this option allows the user to create or modify the set of user-defined sections. The user-defined set is either the one defined by the standard set if the sections were created previously using that option or the basic set (*hk*0, *hk*1, *hk*2, *h*0*l*, *h*1*l*, *h*2*l*, 0*kl*, 1*kl*, 2*kl*, 3*kl*) if the sections were not created beforehand with the standard option. The sections can be displayed and edited by hitting the button “Define set”.

In the picture example (Figure B-53), the sections useful for a cubic system were first created with the option “standard set” using the Bravais class c*P* (cubic, *P*-centered). The corresponding set was written in PETS2 input file, and it became the pre-defined set.

DEFINE SECTIONS MANUALLY: Each user-defined section is specified by a name and nine numbers representing three vectors. The first vector lies in the section plane and will be horizontal in the section. The second vector defines the plane orientation. The third vector specifies the origin of the section plane. For example, the line “hk3 1 0 0 0 1 0 0 0 3” defines a section named *hk0,* where the horizontal direction will be 1 0 0, the second vector in the plane will be 0 1 0, and the plane will contain point 0 0 3.

- **apply symmetry in reconstruction:** this option help to compensate for the missing wedge according to the Laue class selected in the section “Finalize integration”. Note that the Laue class will be used to create sections even if the frame scaling checkbox is unchecked to finalize the integration” (Figure B-53).

The sections are displayed in the main panel in the “Section images” tab.

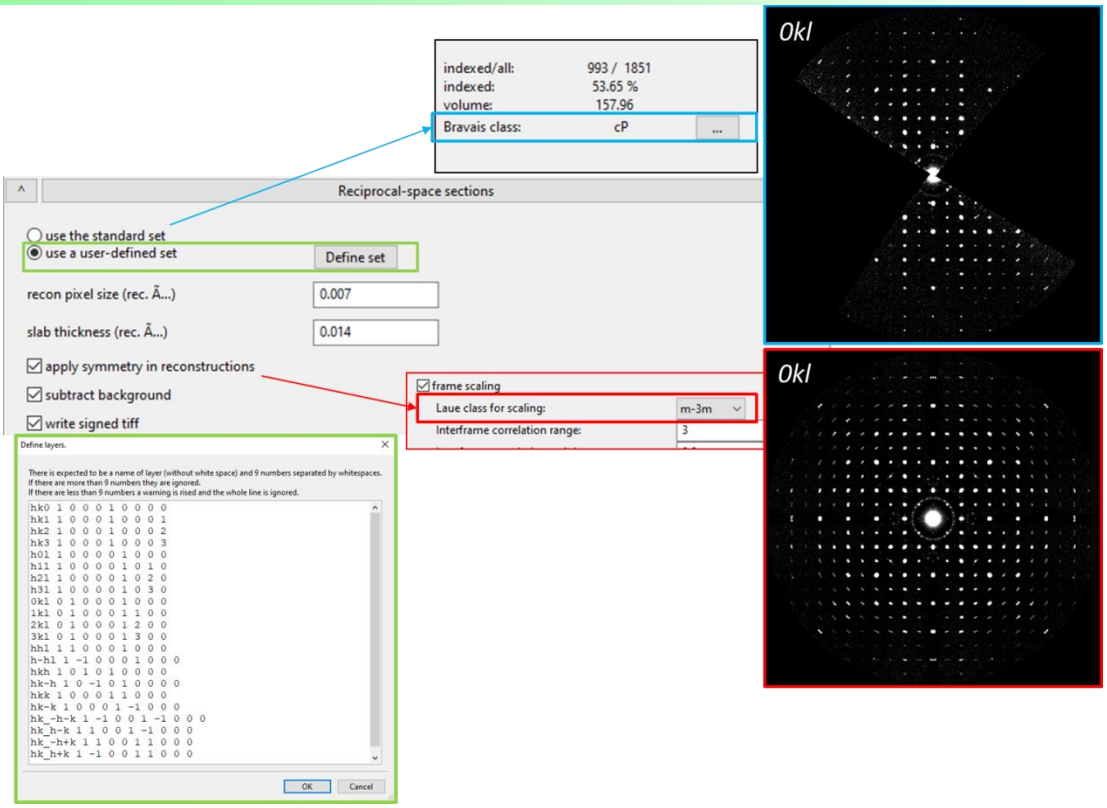


Figure B-53

Two parameters are linked to reciprocal-space layers reconstruction. The two values are expressed in reciprocal angstroms.

- **The pixel size** (recon pixel size) used in the reconstruction (rec.Å). The default value for pixel size is equal to the value of the pixelsize keyword.
- **The slab thickness**: half-thickness of the slab around the exact layer position that is projected onto the layer (rec.Å). Default value for the slab thickness = 2*keyword pixelsize.

Taking a too small pixel size and/or too small slab thickness leads to “holey” reconstructions, where some pixels do not have any intensity.

B.3.13 Write check files

This section is available only for development purposes and does not contain any useful function for the regular use of the program.

B.3.14 Auto-task control

The Autotask option was completely reworked and is now available also from the GUI (Figure B-54).

The tasks to process automatically can be manually selected from the right list and loaded in the left Auto-task list. Three predefined Auto-tasks are available as shown in Figure B-54. Once the tasks are processed, the list is deleted.

The box “keep the auto-task list” is used to keep the list after the tasks. The saved Auto-task list will automatically start when the pts2 file is opened.

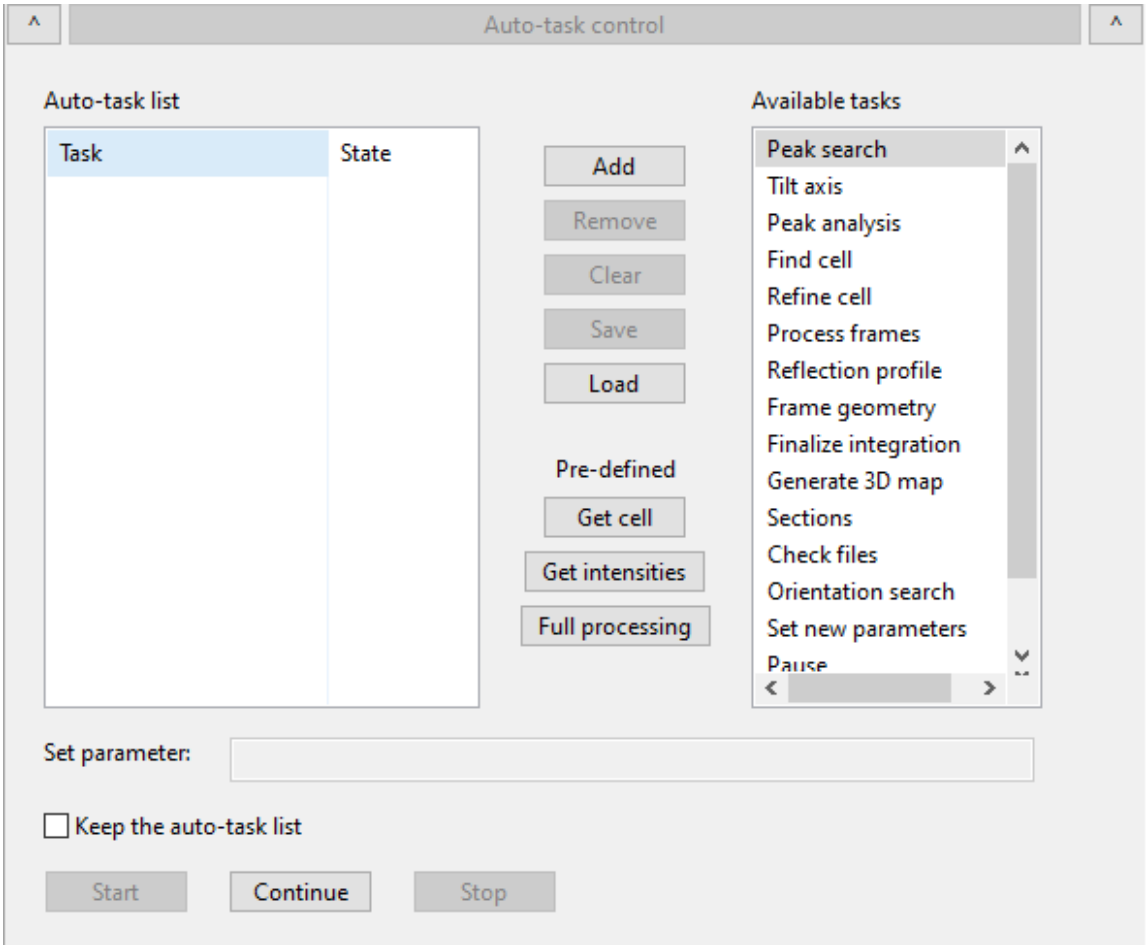


Figure B-54

B.4. Main panel

B.4.1 Image data tab: General overview

Several tabs (Figure B-55) are included in the Main panel: Image data /Section images/Graphs/Log/Cylindric projection/3D panel.

In Figure B-55, the Image tab is shown next to the Image options panel (section B.5.2). They are used together, and some tools are repeated in both panels (see blue, yellow, and pink areas in Figure B-55).

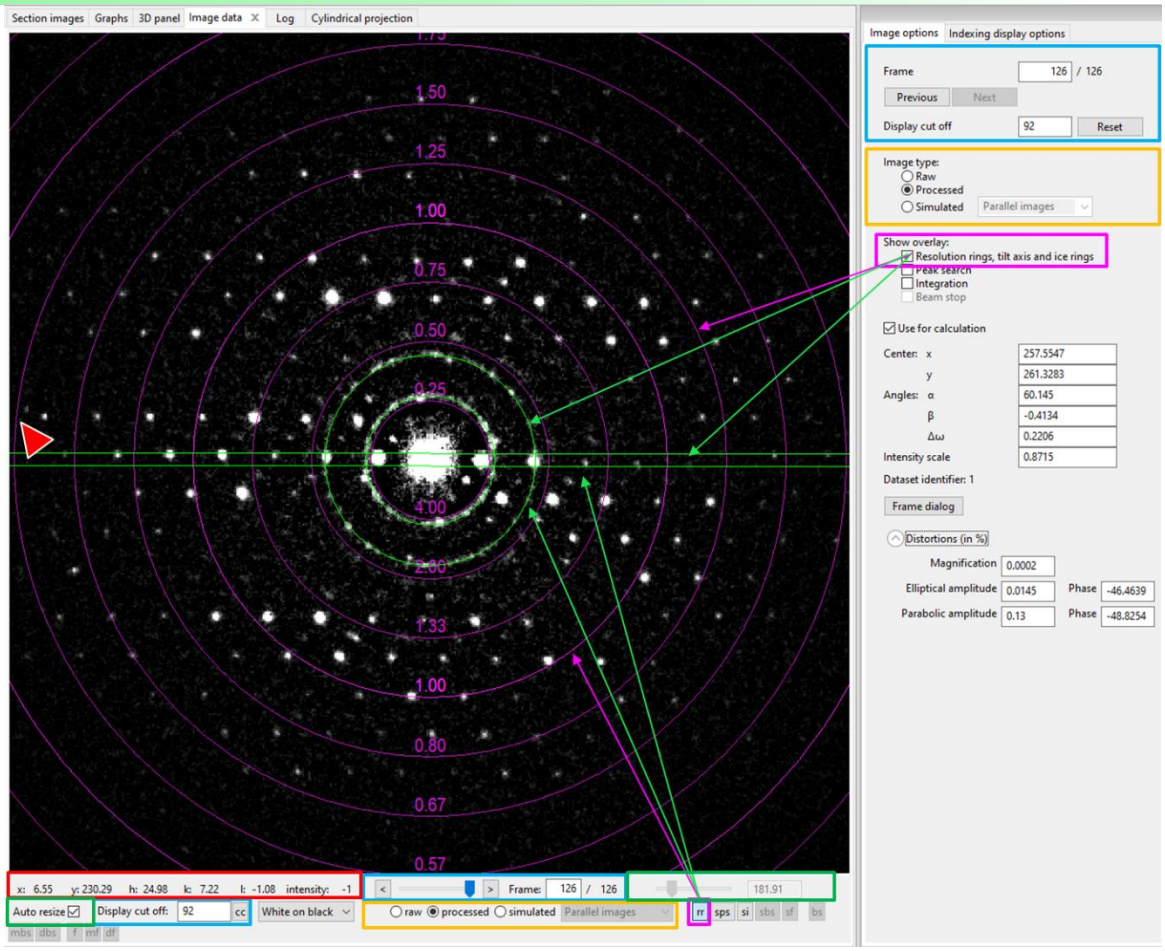


Figure B-55

The Image tab displays the data set frames and a toolbar with general information on the frame and several display options at the bottom. When an option is activated, the corresponding icon glows blue (in Figure B-55, "**rr**"= resolution ring is activated and glows blue).

B.4.1.a. Tollbar: General frames' information

From left to right:

- The ***x* and *y* coordinates** (pixels) of the point where the cursor is placed (red area in Figure B-55).
- The corresponding ***h k l* indexes** (when a unit cell has already been defined) (red area in Figure B-55).
- The **intensity of the point** where the cursor is placed (red area in Figure B-55).
- The **frame number/total number of frames:** used the arrows < and > or the slider to go through the dataset (in the blue area in Figure B-55).

B.4.1.b. Tollbar: General frames' display options

- A **zoom scale** with a slider linked to a box with the zoom value (green encircled area of the toolbar in Figure B-55). The slider and the zoom value are activated when the "auto-size" box is unchecked.

The zoom can also be changed by scrolling up or down with the cursor in the Image data tab. In that case, the "auto-size" box gets unchecked automatically.

- "**Display cut-off**": to change the contrast of the picture (Figure B-55, blue areas). To use the automatic cut off value, hit "cc" (= calculated cut-off) next to the "Display cut-off" value.
- Frames can be displayed using four **color modes** (Figure B-55 and Figure B-56): white on black, black on white, dawn and jet.

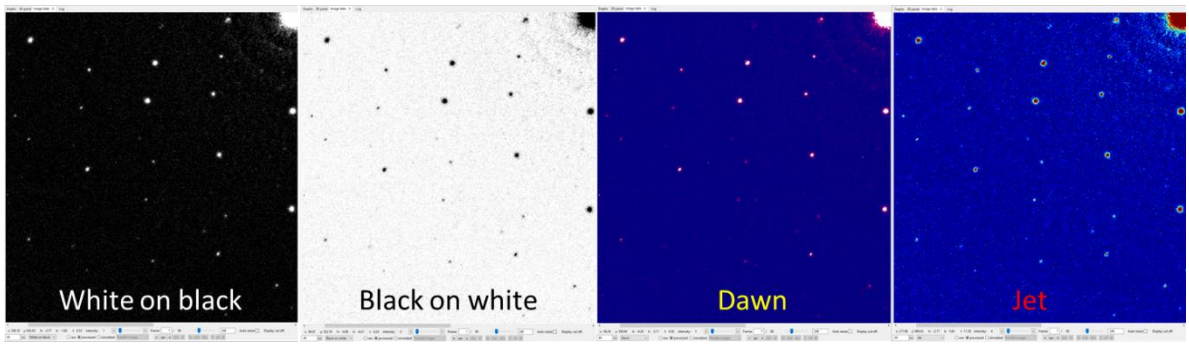


Figure B-56

- **Three image types** can be selected either with radio buttons in the toolbar of the "Image data" tab, or with radio buttons in the "Image options" tab (see yellow areas in Figure B-55). The three types of images are the *raw images* (Figure B-57 up), the *processed ones* (Figure B-57 down) and the *simulated* ones obtained after the geometrical optimizations (Figure B-58).

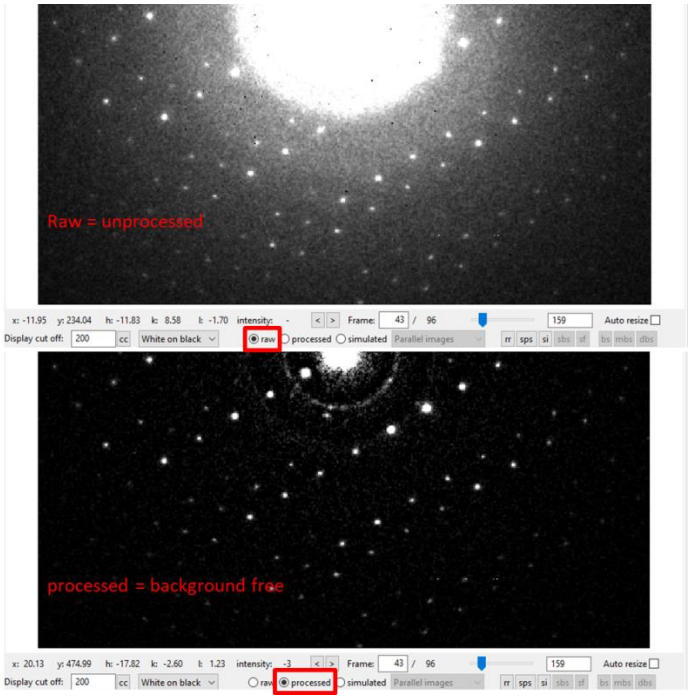


Figure B-57

- **Raw images** = unprocessed TIFF files (Figure B-57 up).
- **Processed** images = background-subtracted images (Figure B-57 down).
- **Simulated images** show the calculated images during the frame-geometry optimizations (angles and center). To visualize the simulated data, the three displaying options are presented in Figure B-58

  -Parallel images: The processed and the simulated images are displayed next to each other (see the purple area in Figure B-58),

-Simulation only: The simulated frames (see the blue area in Figure B-58),

-Combined images: The combined view shows a superposition of the calculated and the experimental images (see the green area in Figure B-58). The green color corresponds to the calculated intensities higher than the observed ones. The pink color is the observed intensity higher than calculated ones, and the white parts correspond to the perfect overlap of both.

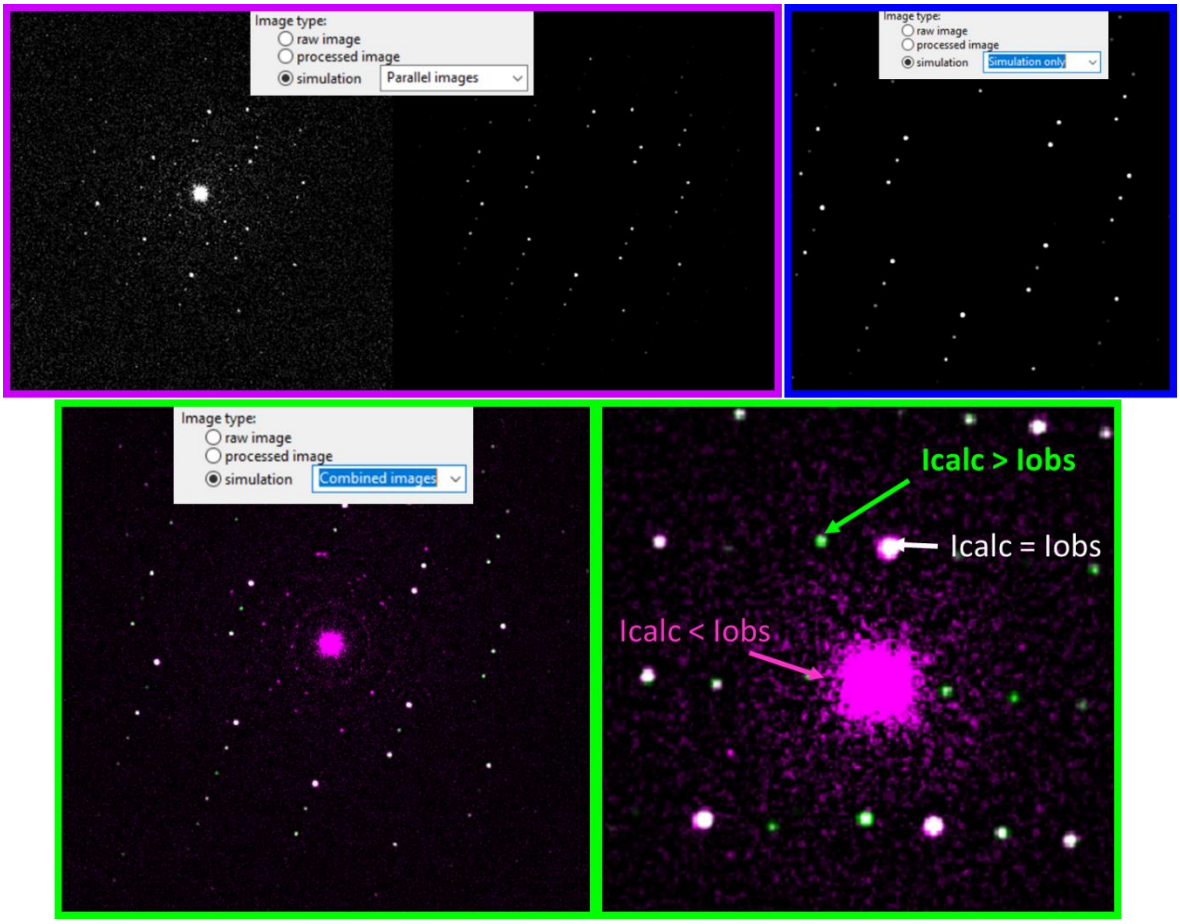


Figure B-58

B.4.1.c. Tollbar: General overlays

- **rr** = to display the **resolution rings** on the frames (pink) together with the **ice ring mask** (green circles if activated in "parameters") and the **excluded area around the rotation axis** (green rectangle) (see Figure B-55). The resolution of each ring is now displayed in reciprocal Å above the center and the corresponding value in the direct space below the center.
- **sps** = show peak search to display the points (green circles) where reflections are detected above the detection threshold I/sigma(I) during the peak search procedure (Figure B-59, left). The option sps is accessible via an icon in the toolbar of the "Image data" tab or via a check box "Peak-search" in the "Image options" tab.
- **si** = show integration to **show the integration masks** (Figure B-59, right). For each frame, the reflection positions are predicted using the current orientation matrix, the centering, and the profile parameters. Then, the intensity at each predicted reflection position is determined. During the process, PETS2 shows an image in the processing (si) of each frame with an overlay of circles

(green or brown). A circle means the reflection is integrated and the circle is green when the reflection intensity is found above I>3sigma(I). You can use the icon “si” at the bottom of the Image data tab or the check box “integration” in the “Image option tab”.

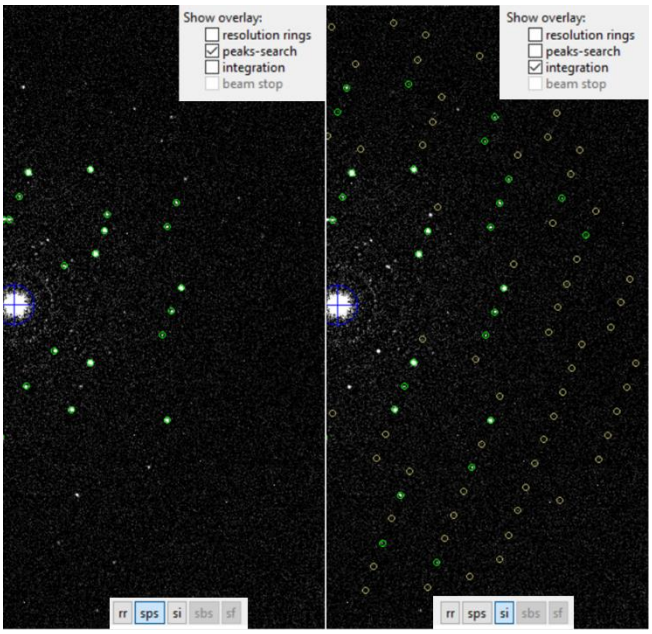


Figure B-59

B.4.1.d. Toolbar: Friedel pairs overlay for manual center determination

Option enabled in the toolbar when the “Automatic initial direct beam coordinates” box is unchecked in the Options of the “Peak search” command. Drawing Friedel pairs is a help to manually determine the center with reasonable accuracy when PETS2 fails to determine automatically it.

- **sf** = show defined Friedel pairs: activated when the automatic detection of the center is unchecked.
- **f** = define Friedel pairs. Hit “f”, and then use the cursor to select pairs of reflections either by clicking on both or by clicking on the first one and then dragging the cursor toward the second point (Figure B-60). You can define as many pairs as wanted to make the center determination more accurate.
- **mf** = move one Friedel point. This option helps to correct the position of the points (see Figure B-60). Click on the point to move on and drag it to its new position.
- **df** = delete Friedel point. To delete one or several Friedel point(s), select an area to remove, as shown in Figure B-60.

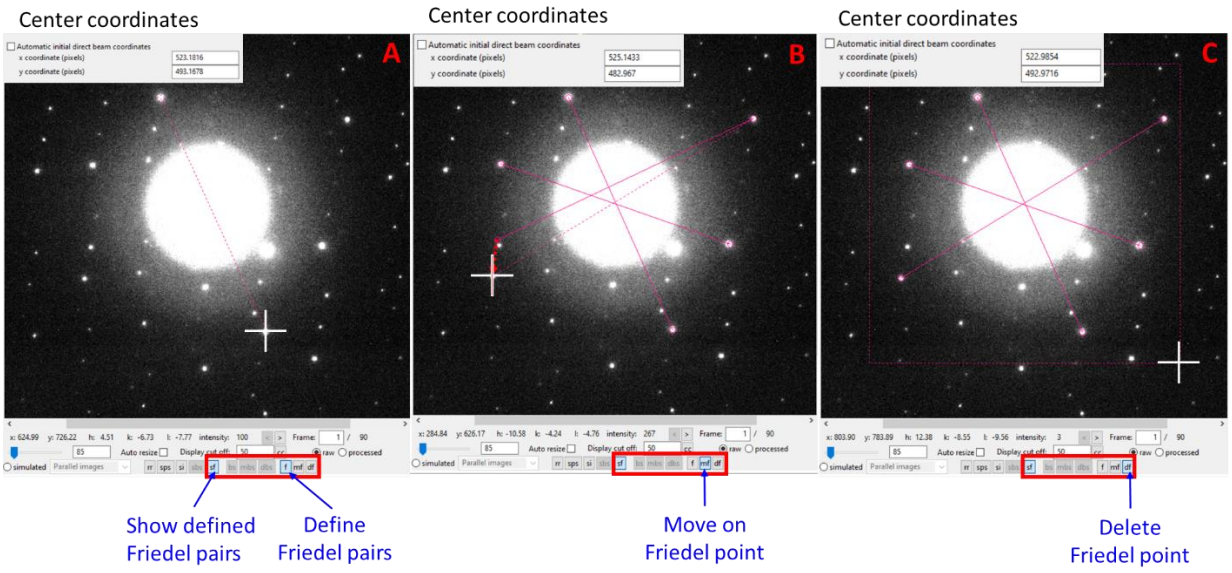


Figure B-60

B.4.1.e. Tollbar: Beam stop

Several options are specifically related to the Beam stop in the toolbar. They are enabled when "beam stop Yes" is activated in the "Parameters" options.

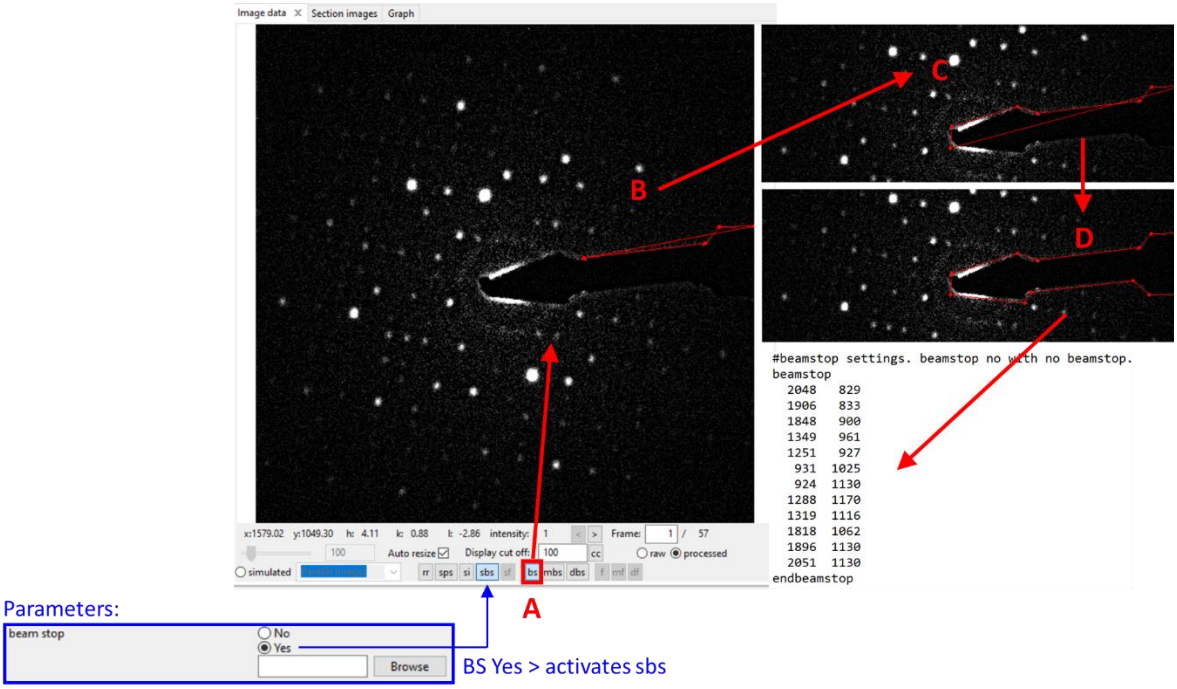


Figure B-61

- **sbs** = show beam stop overlay = to display the beam stop mask on the frame (Figure B-61, left). The sbs option is activated by default when Beam stop "yes" is selected in the parameters (see Figure B-61, left).

- **bs** = create a new beam stop. When BS "yes" is selected in the "Parameters" options (commands panel) without a loaded file with the BS coordinates, you can manually define a BS through the Image data panel options. In Fig. B40, first Press bs to activate the creation of a new BS (step A), and click on the frame to create as many points as needed to describe the mask's shape (B, C , D). The x and y coordinates of each point are stored in the pts2 file.
- **mbs** = move the beam stop. Hit the icon and move the beam stop mask by clicking directly on frame and dragging the BS to its new position (Figure B-62, left).
- **dbs** = delete beam stop. Delete a few points or all the beam stop mask by selecting the area to delete (Figure B-62, right).

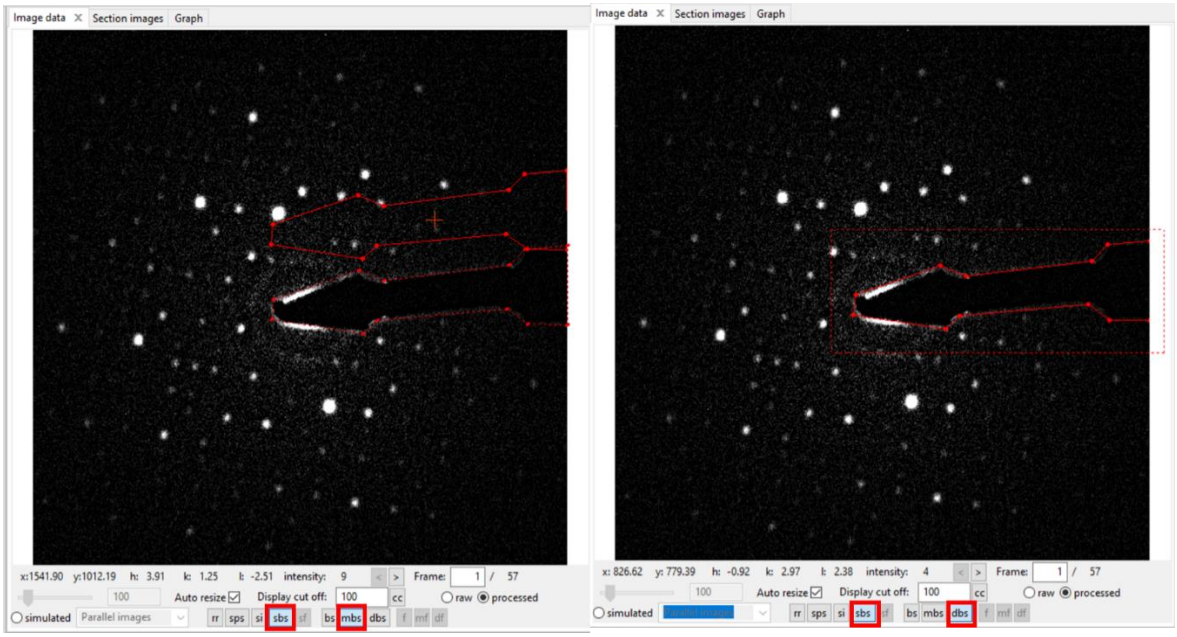


Figure B-62

B.4.2 Section images

The "Section images" tab contains sections of the reciprocal-space reconstructed after hitting the command "reciprocal-space" sections.

- Select the section to display using pull-down menu at the bottom of the tab (Figure B-63),
- Change the contrast with the **cut-off**. As for the main panel, the cut-off can be adjusted and applied to all sections by hitting "apply to all sections".
- **zoom** in-out (automatic or not) using the slider (uncheck "Auto resize") or by scrolling directly on the section.
- The **coordinates** of the cursor position are expressed either with the **x and y** (pixel) or with the ***h k l*** indexes. By placing the cursor on one reflection, its hkl indices and its corresponding intensity are given in the information bar.
- OVERLAY 1: **gr** = **grid**, displays the lattice as an overlay on the sections.
- OVERLAY 2: **f** = displays **lines** approximatively corresponding to the first (1 green) and the last (1-red) frame of the dataset. The two lines are labeled 1 for dataset1. When several datasets are merged, the pairs of lines will be labeled 1, 2, 3 etc… to indicate the corresponding dataset number (Figure B-63).
- OVERLAY 3: **rr** = **resolution rings** with their labels in rec. Å (up) and Å (down) (Figure B-63).

- The command "**save sections**" will save all sections simultaneously in png format considering the current cut-off value and the frame color theme. Those sections are stored in a folder jobname_petsdata\sections\png.

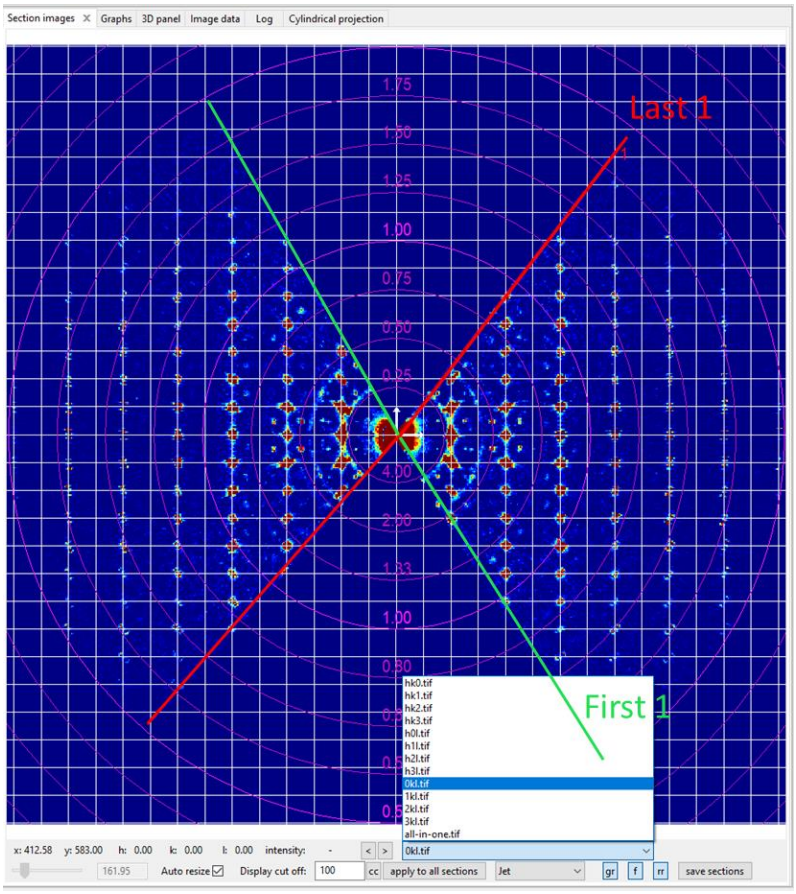


Figure B-63

B.4.3 Graphs tab

All the Graphs produced during the data processing are displayed in the Graphs tab.

Below is a list of all graphs generated by PETS2 during the data reduction. To go from one graph to another, use the drop-down menu located at the bottom of the Graph tab or the arrow on the right of the menu (see Figure B-64).

Graph	Corresponding step
Peak search: pattern centers	Peak search
Peak search: Maximum resolution	Peak search
Peak analysis: In-plane distances	Peak analysis
Peak analysis: 3D distances	Peak analysis
I/sigma in two shells	Process frames for integration
Integration: Resolution	Process frames for integration
Rocking curve	Process frames for integration
Frame scales	Finalize integration
Normal probability plot	Finalyze integration

Tilt corrections	Optimize reflection profile and geometry
Origin corrections	Optimize reflection profile and geometry
Frame-by-frame distortions	Optimize reflection profile and geometry
Profile parameters	Optimize reflection profile and geometry

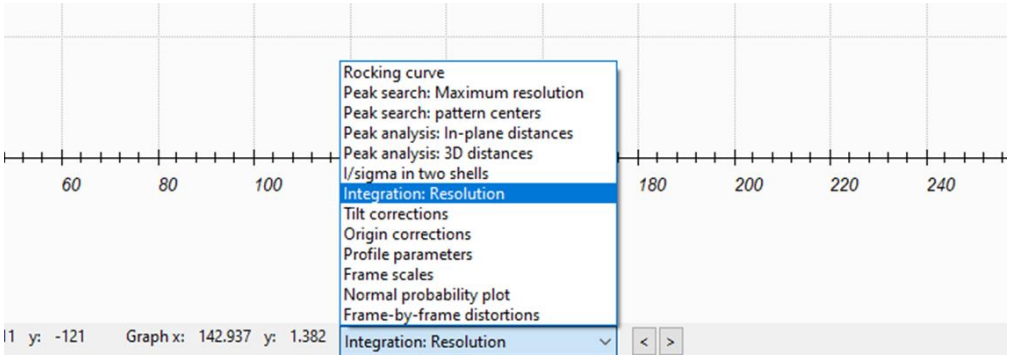


Figure B-64

Note: At this stage of software development, graphs are available in the Graph tab as long as the PETS2 project is open. When an older PETS2 project is re-opened, some graphs can easily be regenerated (the RC curves, the cylindrical projection with the "replot" button). However, most of them are lost until the corresponding step is re-run. The details of the previous data processing with the corresponding graphs are stored in the log files.

Options available on the graphs:

- **Save an image of the graphs**: click right on the Graphs tab you want to save and select "**Save graph as**….".
- **To report a bug related to the display of the graphs**: click right on the graph and select **"Print debug info to console**". The information generated in the console panel will only be useful for the developers.
- **Access graphs settings**: click right on the graph and select "properties"
- **Zoom in/out**: put the cursor on the area of interest on the graph. The mouse wheel rotation in the graph zooms both dimensions. The mouse wheel on an axis zooms only that axis.

B.4.3.a. Peak search: Pattern center

This graph shows the *x* and *y* shift of the direct beam frame-by-frame with respect to the initial value of the center (determined automatically or manually) (Figure B-65).

The *x* (red) and the *y* (green) shifts are represented in pixels.

This graph is produced during the Peak Search procedure.

To see to what frame and or value corresponds to one point of the graph: place the cursor on the point of the graph; its *x* = frame number and *y* = shift value are given below the graph in the information bar (see example in Figure B-65, left).

B.4.3.b. Peak search: Maximum resolution

This graph (see Figure B-65, right) reports for each frame, the peak with the maximum resolution found above the detection threshold during the peak search procedure (I/sigma). This graph helps to have a quick overview of data resolution frame-by-frame, the possible decay of the crystal, the thickness evolution, etc. It depends on:

- The data resolution.
- The d* max peak search (here the value = 2 $Å^{-1}$).
- The I/sigma value (5 in that example).

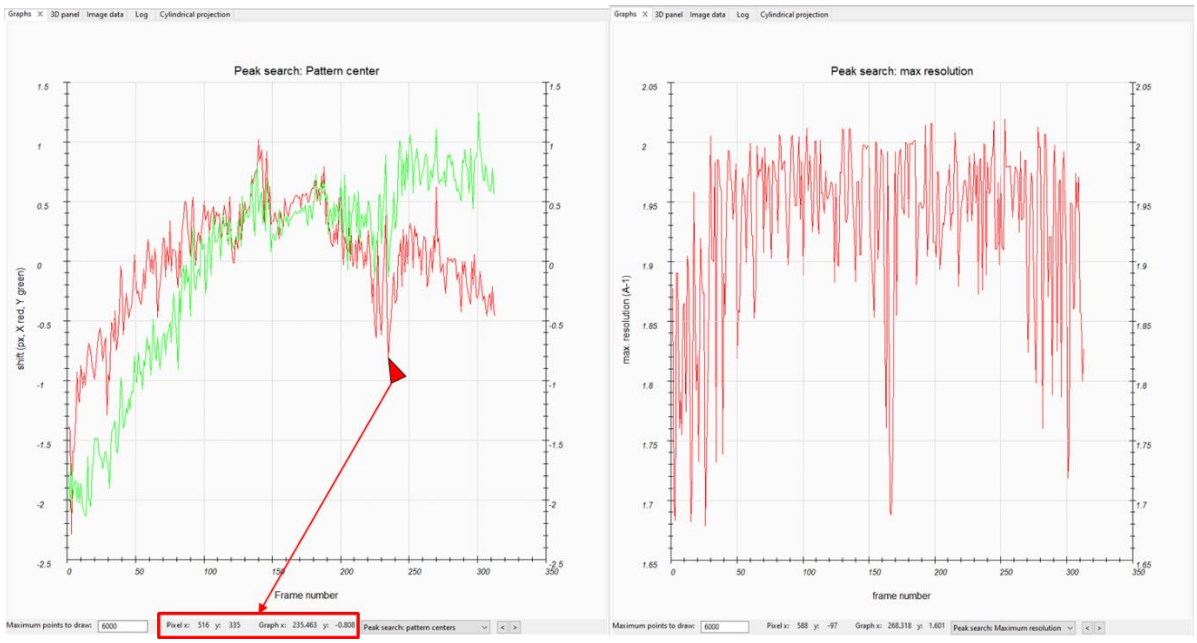


Figure B-65

B.4.3.c. Peak analysis: In-plane-distances/3D distances

See section B.3.4**.**

In the first step, all reflections measured on subsequent frames are clustered, and the centers of the clusters are used instead of individual peak positions. For this step, the limit for the clusters must be determined. Next, the program shows a plot of inter-peak distances sorted from the shortest to the longest to check that the program did it correctly (Figure B-66, left). If everything works fine, you should see a clear jump in the distances (red curve in the plot) accompanied by a peak in the derivative (green curve in the plot).

In the next step, PETS2 calculates the difference vectors between the peaks. Then the same clustering procedure is applied to the group of difference vectors, and again a plot of distances is shown for checking the correctness of the choice. The jump in the second plot is usually even sharper than in the first plot (Figure B-66, right), unless the data contain signal from more than crystal, in which case the second plot may be rather fuzzy without clear sharp peaks.

On each graph the blue line is the limit for detecting a significant increase in the derivative line. Thus, the first intersection of the blue line with the green line is the point from which the algorithm detects the onset of the first jump. The light blue vertical line is the position in the distance ranking then defines the limit for the clustering - this is the most important line that really defines the limit for the clusters. It is

this line that you can change by clicking in the plot. The horizontal purple line is placed at the intersection of the vertical light blue, and the red curve, and it shows on the *y* axis the distance threshold that is used for the clustering.

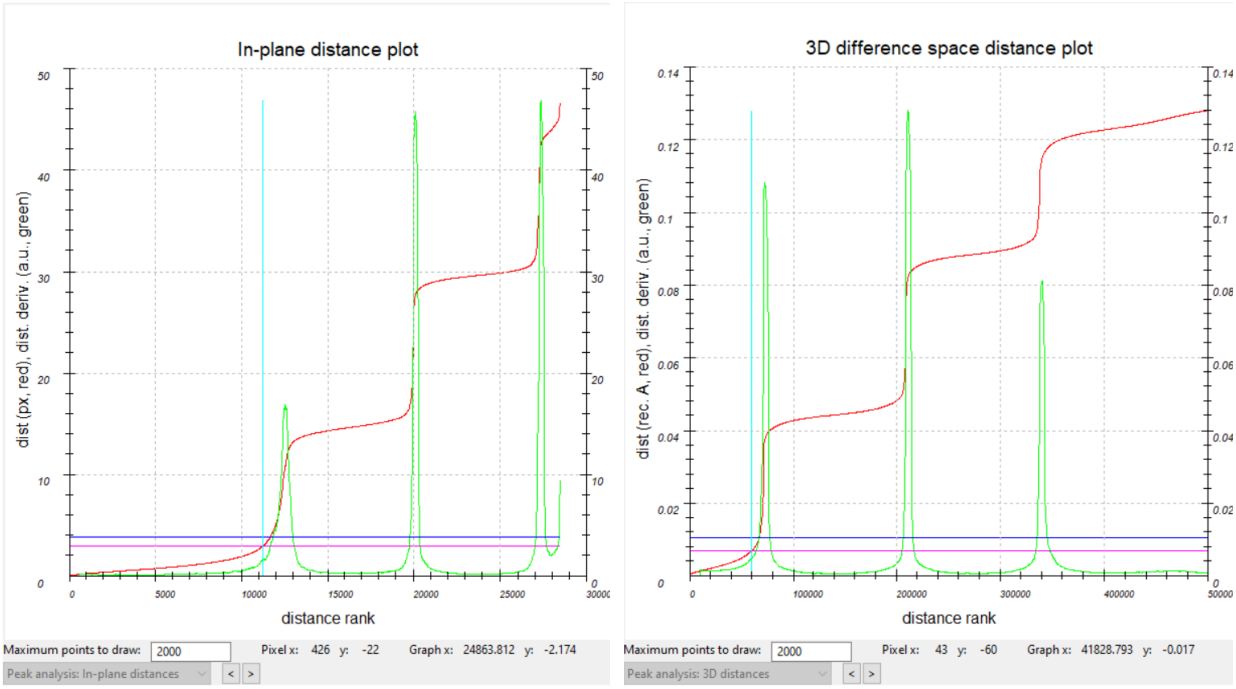


Figure B-66

B.4.3.d. Integration resolution graphs

- **I/sigma in two shells graph**: For each frame, this graph shows the mean I/sigma(I) of what are called the inner (red curve) and the outer shells (green curve) (Figure B-67). The shell below d* integration*threshold is considered the inner shell (by default, threshold = 0.5 $Å^{-1}$). The default limit between inner and outer shell for the calculation of average I/sigma can be changed in the input PETS2 file after the keyword “resshellfraction”. It does not influence the integration steps but is a way to visualize the attenuation of the resolution. The graph is created during the “process frame for integration”.
- “**Integration: Resolution” graph**: for each frame the length of the maximum diffraction vector (d* in reciprocal Å) is given for 2σ (red in Figure B-67), 3σ (green in Figure B-67) and 5σ (blue in Figure B-67). This graph is the easier way to follow the resolution decay in the data set. The graph is created during the “process frame for integration”.

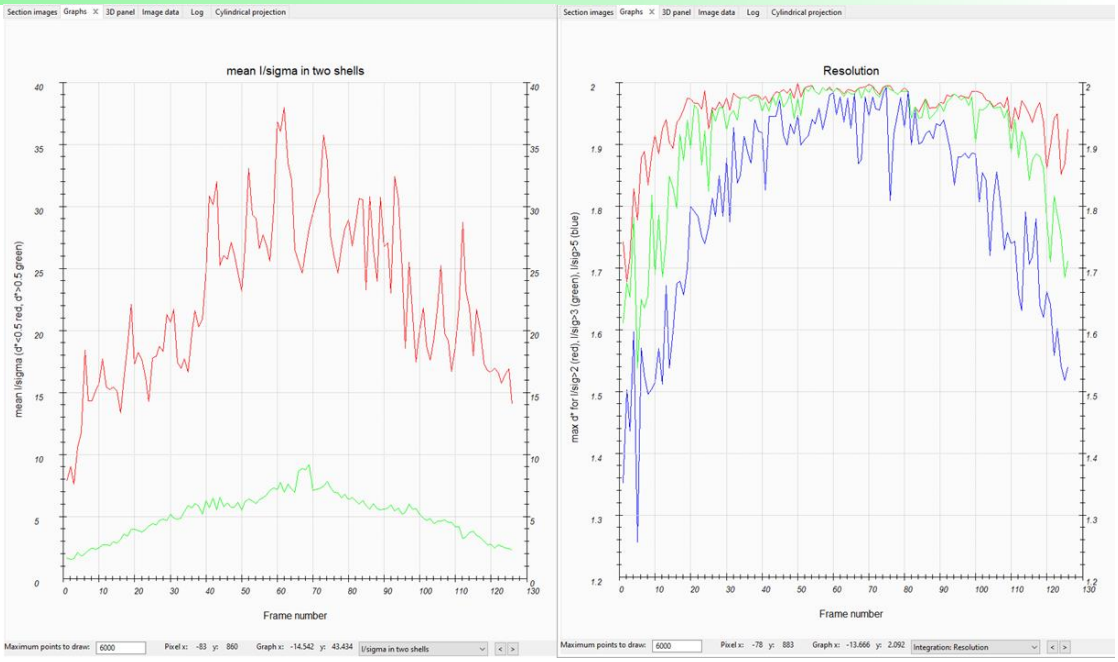


Figure B-67

B.4.3.e. Rocking curves-Camel plot

At the end of the integration process, the Camel plot (rocking curves profiles) is generated (data/ experimental - blue, fit/ calculated - red) (Figure B-68). Because of the specific shape (one or two humps), the curve is called the "the camel" and the plot of rocking curves is "the camel plot". On the horizontal axis of the plot is the excitation error of the reflections (i.e., the deviation from perfect Bragg condition). Each curve shows an average rocking curve of reflections in one resolution shell, starting from 0.2-0.3 $Å^{-1}$ in steps of 0.1 $Å^{-1}$. Notice the splitting of the humps for precession data ((Figure B-68, right). The splitting will increase with the increase of the precession angle. For continuous rotation or static frame experiments, the camel plot shows a single hump ((Figure B-68, left).

Three parameters found in the section "**Optimize reflection profile and geometry**" (inset in the figure B47) will directly influence the quality of the fit (red) or the display:

- **The rocking curve width**,
- **The apparent mosaicity**,
- **The precession or tilt semi-angle**.
- **The minimum I/sigma(I)** for display (10 by default). This display parameter won't influence the quality of the fit and the integration result. Note: for further geometrical optimizations, I/sigma(I) = 10 is used whatever the set value for the display.
- **The step size in the reflection profile**. Display parameters that won't influence the quality of the fit and the integration result. A too small value will make the observe RC (blue) very noisy.

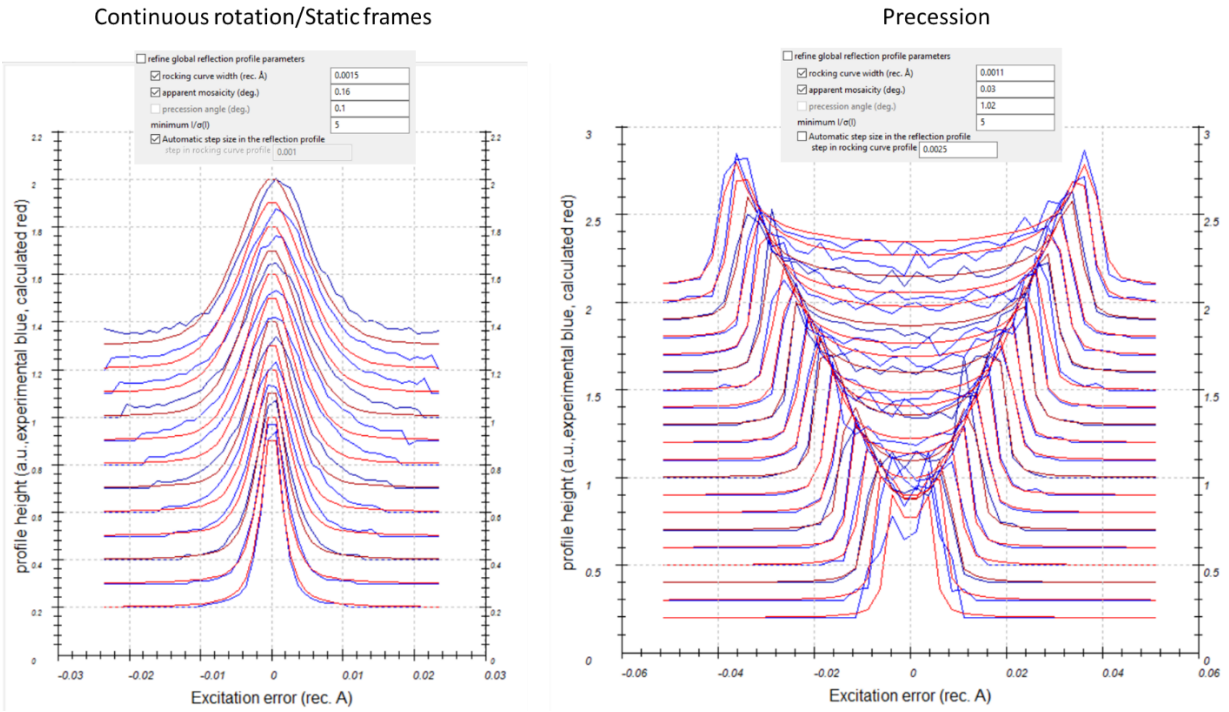


Figure B-68

B.4.3.f. Frame scales

When the integration is "Finalized", a scaling can be applied for the kinematical *hkl* files. The graph represents the scale applied for each frame and will appear only when the checkbox "frame scaling" is selected in the section "Finalize integration" (Figure B-69).

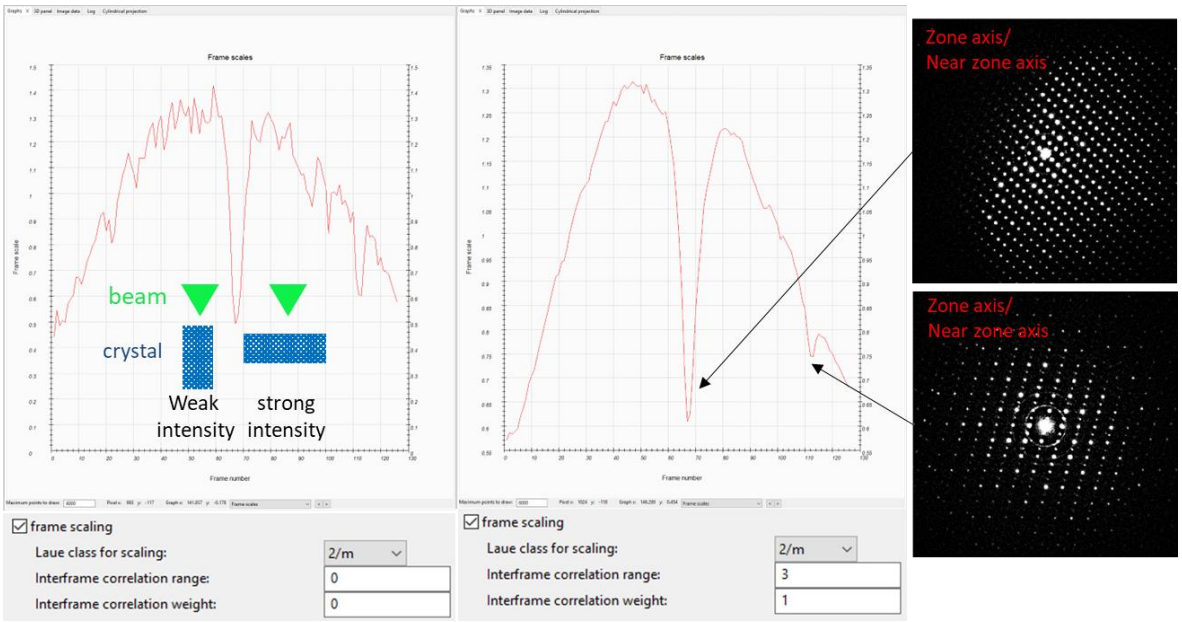


Figure B-69

B.4.3.g. Geometrical corrections: Tilt and origin corrections graphs

These two graphs are produced during the frame geometry optimization (Figure B-70).

- **Tilt corrections graph**: Correction of the orientation angle alpha-tilt, beta-tilt and omega-rotation in degree. (Figure B-70 left). The graph shows the Δalpha (red), Δbeta (blue) and Δomega (green) for all frames of the dataset(s). When a smoothing option is activated, for each tilt angle, the smoothed curve appears in bright colors, the initial calculated one is in lighter color and noisier. The graph shows the difference between the new values and the very initial ones (not the previous one if several optimizations are performed).
- **Origin corrections graph**: The frame-by-frame corrections of the center of the pattern is expressed with its two Δx and Δy coordinates in pixels (Figure B-70, right). No smoothing available for this optimization. Origin corrections in Optimize orientations shown as a difference to the average position, not to previous position on the frame.

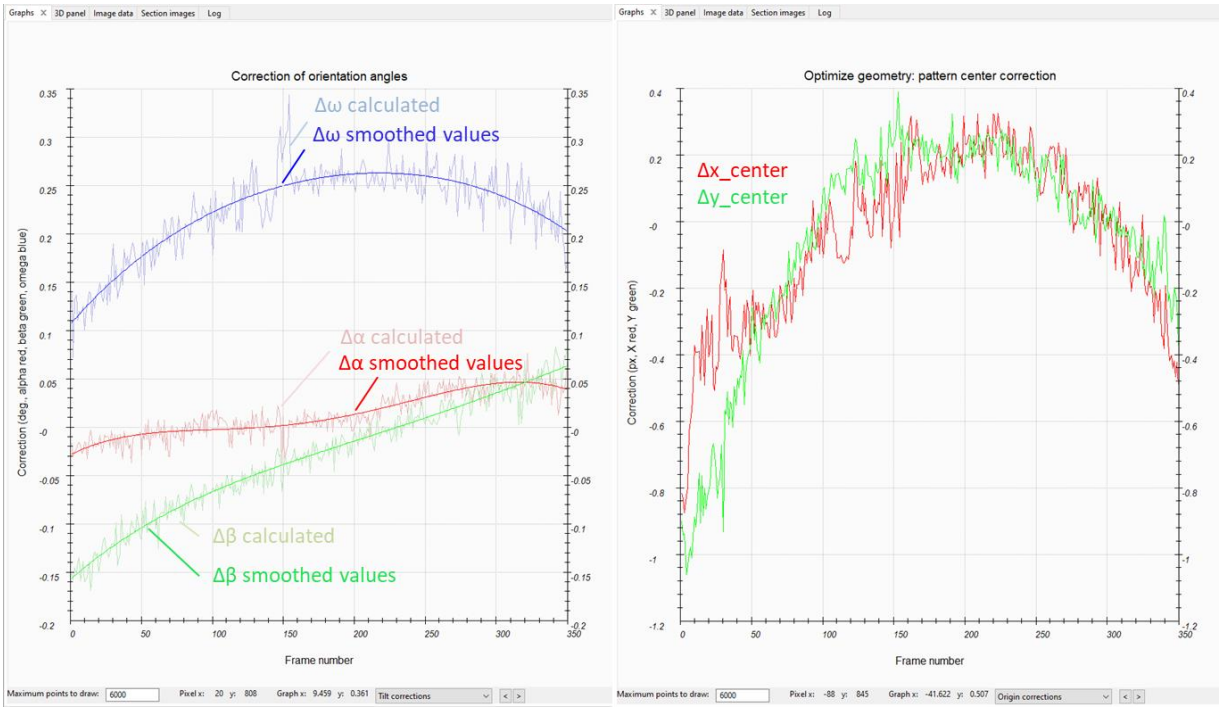


Figure B-70

B.4.3.h. Geometrical corrections: Frame-by-frame distortions graph

The distortions that are refined frame-by-frame are summarized in a graph where the x-axis is the frame number, and the ordinate will depend on the type of distortion. The smoothing is also applied to this optimization if selected. (see Figure B-71). The smoothed curves are displayed brighter than the initial calculated ones.

- **Magnification distortion or scale**: The red curve indicated as “scale” in percent is called “magnification” in the standard mode of the distortion.
- **Amplitude of the elliptical distortion** in percent: green curve.
- **Phase of the elliptical distortion**: The blue curve corresponds to the Phase of the elliptical distortion expressed in degrees and divided by 100 to match the graph scale.

- **Amplitude of the parabolic distortion**: The magenta curve in percent.
- **Phase of the parabolic distortion**: The blue curve corresponds to the phase of the parabolic distortion expressed in degrees and divided by 100 to match the graph scale.

Note that for each frame, the exact value of the distortions refined frame-by-frame can be found in the "Image option" tab for the corresponding frame number. The other types of distortions are not displayed in a graph as they are not refined frame-by-frame but only globally (see the "Refine" menu of "Find unit cell and orientation matrix", section B.3.5).

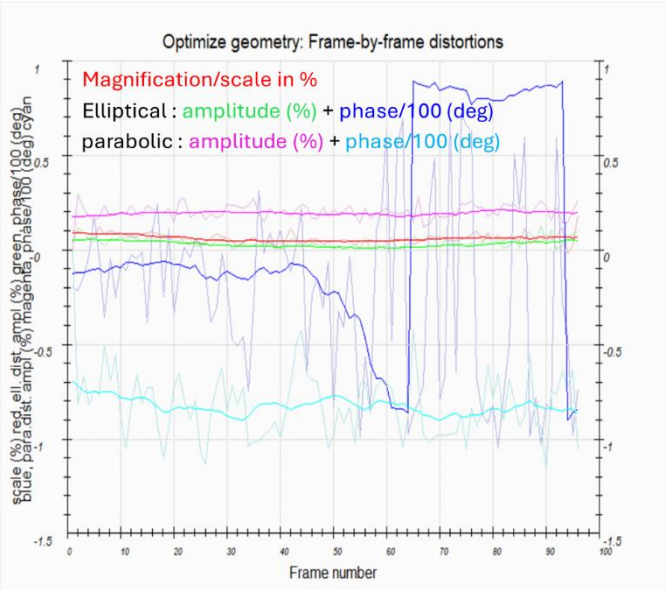


Figure B-71

B.4.3.i. Profile parameters graph

The rocking curve profile parameters (RC width ($Å^{-1}$) or mosaicity (deg)) and the variance of the spot size (pixel) refined frame-by-frame are given in the graph below (Figure B-72). As RC width and apparent mosaicity are correlated, only one of them is usually involved in the refinement.

To put those three values on the same graph, RC width and apparent mosaicity are rescaled as shown in the picture below.

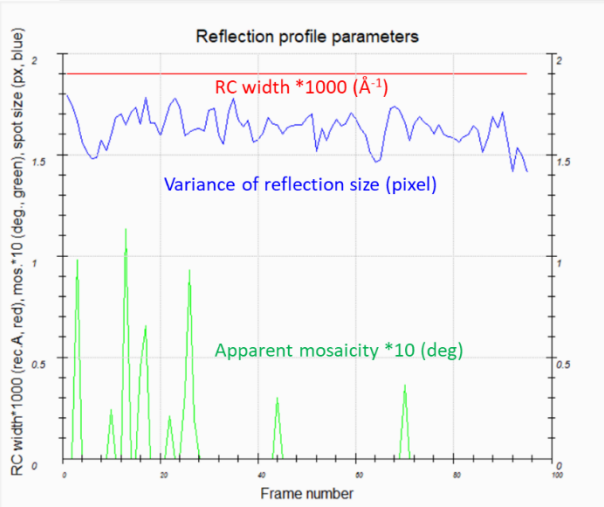


Figure B-72

B.4.3.j. Normal probability plot

This graph is produced during the "finalize integration" step. It shows the distribution of sigma(I) with a histogram and a normal probability plot. On the histogram and the plot, the pink Gaussian and the red line represent a perfect Gaussian distribution of the sigma(I). The blue histogram and the green dots are the sigma distribution observed in the data (Figure B-73). Dark red dots in the normal probability plot indicate points labeled as outliers by the outlier rejection algorithm.

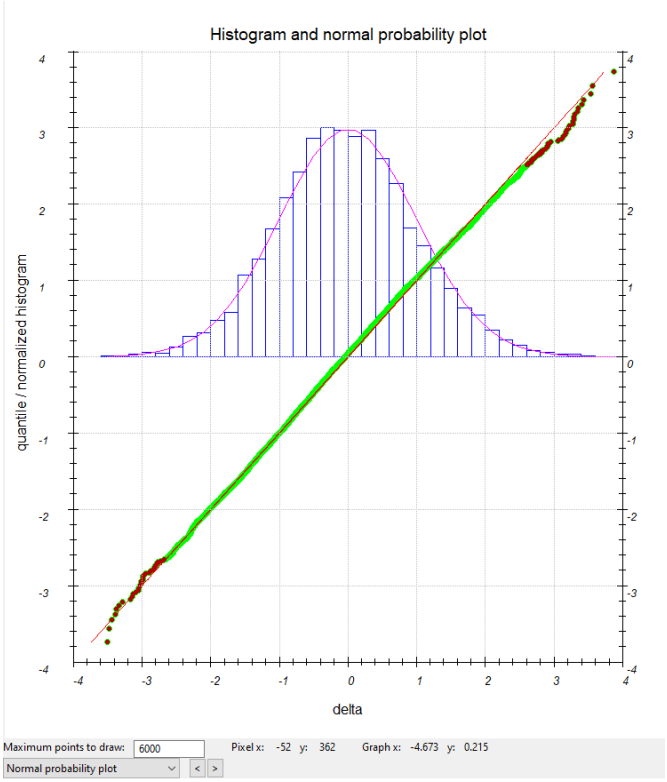


Figure B-73

B.4.4 Cylindrical projection

This tab of the main panel shows only the cylindrical projection that should be the sharpest for the right tilt axis position or omega angle (degree) (Figure B-74). The value of the omega angle can be changed, and the cylindrical projection replot in the “Tilt axis” section of the command menu. Below are two examples of such plots. The first one is made with the refined omega and the second with an omega off by 10 deg.

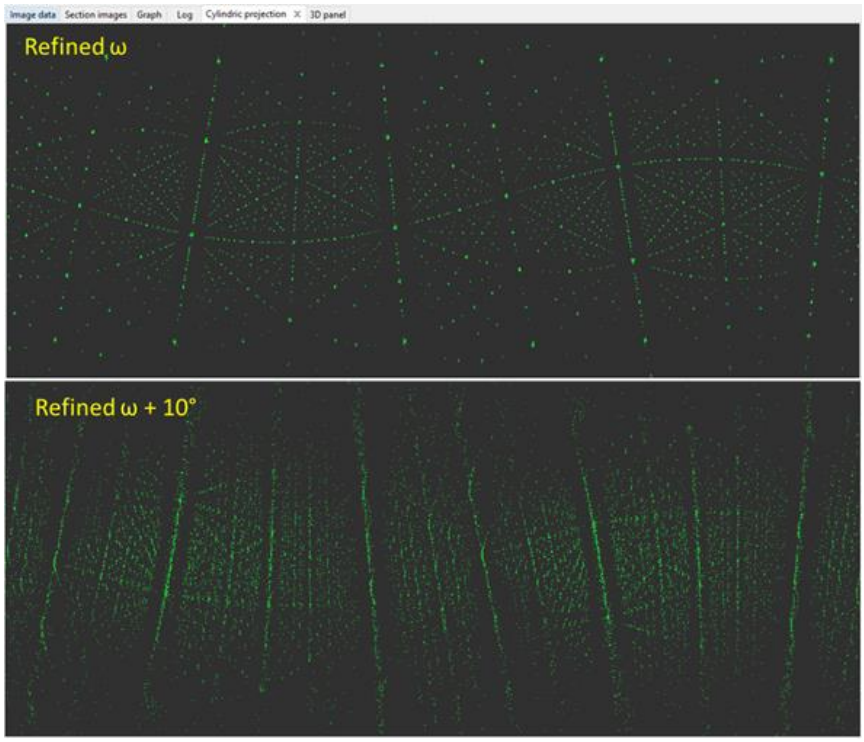


Figure B-74

B.4.5 Log files

Several report files are produced in the html format and can be displayed in the log tab of the main panel (Figure B-75). They usually merge information given in the console with the related graphs and settings. A drop-down menu and the arrows < and > are used to select the log. They are stored in the subdirectory jobname_petsdata/logs. Two versions of each file are saved. Pure text file, and a html version with embedded images. The graphs used in the logs are also saved in subfolder jobname_petsdata/plots. The table below contains a brief description of each log file.

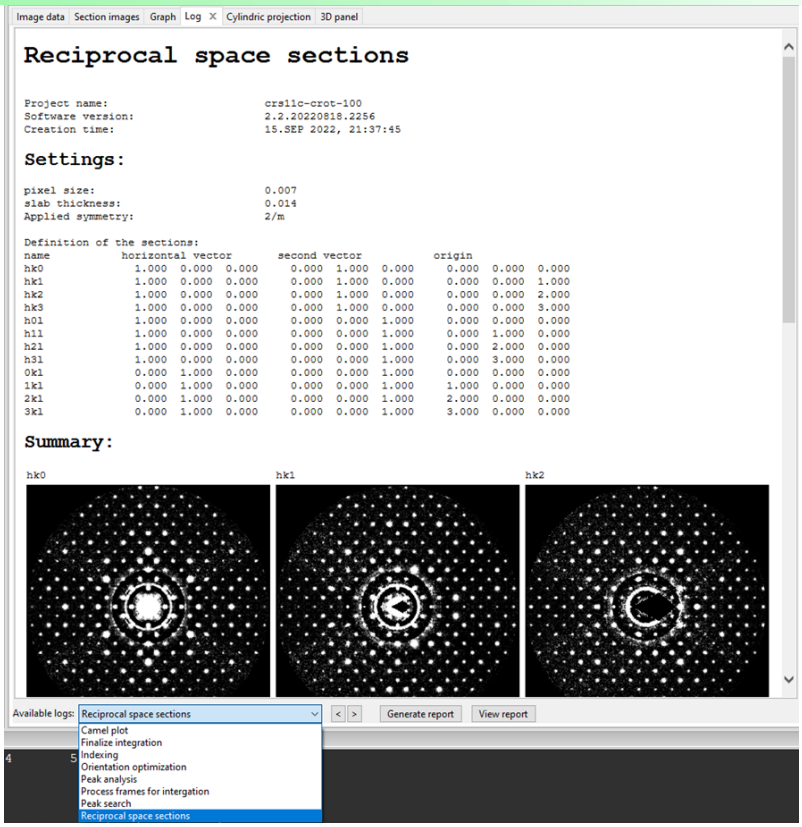


Figure B-75

Log file extension	description
logcml	**Camel plot profile** + relevant parameters
logfi	**Process frames for integration**'s report: -settings with the cell dimensions, Rocking curve profile parameters, intensity determination method, -a summary with the number of frames used, the min/mean/max resolution etc., number of integrated spots and how many could be fitted with the method. -the resolution plot after integration. -list of the frames with the number of peaks and the max resolution.
logoo	**Frame orientation optimization** -settings list **-**Four graphs related to the geometry optimization of the frame: tilt angles, pattern center, frame-by-frame distortions, and frame-by-frame profile parameters. -Details for each frame with the values of all refined parameters
logpa	**Repot for the Peak analysis** -Mostly the two graphs and values obtained during the clustering procedure.
logpfi	Report on the **Finalize integration** step: -the settings used -Graphs for frame scales and histogram and normal probability plot.

-the statistics for each Laue class (*Rint(obs)* and *Rint(all)*, *Rmeas(obs)* and *Rmeas(all)*, *Nobs/Nall* and redundancy).
-the statistics for the selected Laue class by resolution range (*d** and *d*-ranges, *Nobs/Nall/Nthr*, completeness, redundancy, *I/σ(I)*, *Rint(obs)*, *Rint(all) and CC1/2*)
-the frame scaling frame-by-frame (graph and list).

cwl	Laue class	Rint(obs)	Rint(all)	Rmeas(obs)	Rmeas(all)	Nobs/Nall	redundancy
*	-1	17.66	21.10	24.88	29.69	2622/ 8802	1.897
*	2/m	21.49	28.23	25.81	33.94	1430/ 4847	3.445
	112/m	34.09	40.52	41.03	48.52	1682/ 6477	2.578
	2/m11	33.97	40.63	40.95	48.69	1681/ 6417	2.602
	mmm	35.54	44.28	39.70	49.27	883/ 3573	4.673
	4/m	41.15	54.34	46.36	60.79	917/ 4269	3.911
	4/mmm	40.43	56.07	43.71	60.08	478/ 2415	6.914
	-3	43.44	53.78	50.70	62.15	1486/ 7129	2.342
	-31m	46.38	62.64	51.53	68.48	860/ 5127	3.257
	-3m1	51.17	66.11	56.86	72.69	847/ 5138	3.250
	6/m	51.40	64.98	56.61	70.97	845/ 5119	3.262
	6/mmm	50.99	69.74	54.81	73.98	468/ 3523	4.739
	m-3	40.11	55.18	43.82	59.23	600/ 2765	6.039
	m-3m	40.73	62.10	43.70	65.15	329/ 1832	9.114

Estimated Laue class: 2/m

Statistics for Laue class 2/m

d*-range	d-range	Nobs	Nall	Nthr	compl.	rdnd.I/s(cnt)	I/s(erm)	Rint(obs)	Rint(all)	CC1/2
0.00-0.67	Inf-1.50	110	154	300	0.513	3.40	115.08	3.68	18.29	21.03	94.41
0.67-0.94	1.50-1.06	185	388	530	0.732	3.53	60.49	3.34	20.20	26.95	73.82
0.94-1.15	1.06-0.87	218	510	684	0.746	3.53	35.08	3.17	23.07	28.82	74.65
1.15-1.33	0.87-0.75	207	590	794	0.743	3.59	22.73	3.03	24.90	32.31	69.10
1.33-1.49	0.75-0.67	210	691	911	0.759	3.54	15.41	2.89	25.23	32.12	77.41
1.49-1.63	0.67-0.61	183	751	993	0.756	3.56	11.50	2.72	25.98	35.04	64.63
1.63-1.76	0.61-0.57	175	791	1072	0.738	3.27	8.57	2.62	25.86	35.74	63.47
1.76-1.89	0.57-0.53	95	559	1178	0.475	3.26	7.57	2.40	25.11	38.75	51.30
1.89-2.00	0.53-0.50	47	413	1224	0.337	3.28	6.10	2.35	27.48	39.28	59.03
0.00-2.00	Inf.-0.50	1430	4847	7686	0.631	3.44	27.41	3.18	21.49	28.23	87.87

logps	**Peak search report:** -settings used and summary -graphs: pattern center from the peak search and maximum resolution graph -list frame-by-frame of the peak search results.
logrss	It corresponds to the report for the **reciprocal space sections** with: -parameters used to build sections -list of the sections with their vectoral definition -pictures
logindex	**Indexing report** -settings -Details for all define unit cells -Global distortion values

B.4.6 3D panel

The 3D panel displays the 3D reconstructions of the reciprocal space (cor, xyz, clust, or diff files). It is activated with the “Find unit cell and orientation matrix” menu together with the “Indexation display options” tab of the Option panel (see description in the next section B.5).

- The type of reconstruction displayed (the cloud) is selected from the header of the Indexing panel (clust in the example of Figure B-76). On the bottom and the left side of the 3D panel are the intensity histograms (blue in Figure B-76) that can help to set a specific direction.
- To do an in-plane rotation of the cloud, put the cursor out of the cloud area and click + rotate (with the cursor out of the area defined with the green circle in Figure B-76).

- To rotate in the 3 dimensions, click with the cursor inside the cloud area (red cursor, inside the circle in Figure B-76).
- The axis-base at the bottom left of the 3D panel can be changed using the Options panel (laboratory axes, reciprocal axes, or direct axes).
- To zoom in/out, use the mouse wheel in the 3D panel or use the +/- buttons at the bottom left of the 3D panel tab.
- To change the color settings, go to Edit\Settings in the main toolbar.

All other possible actions influencing the 3D reconstruction are presented either in the section related to indexing or the section about the Option panel\Indexation display options.

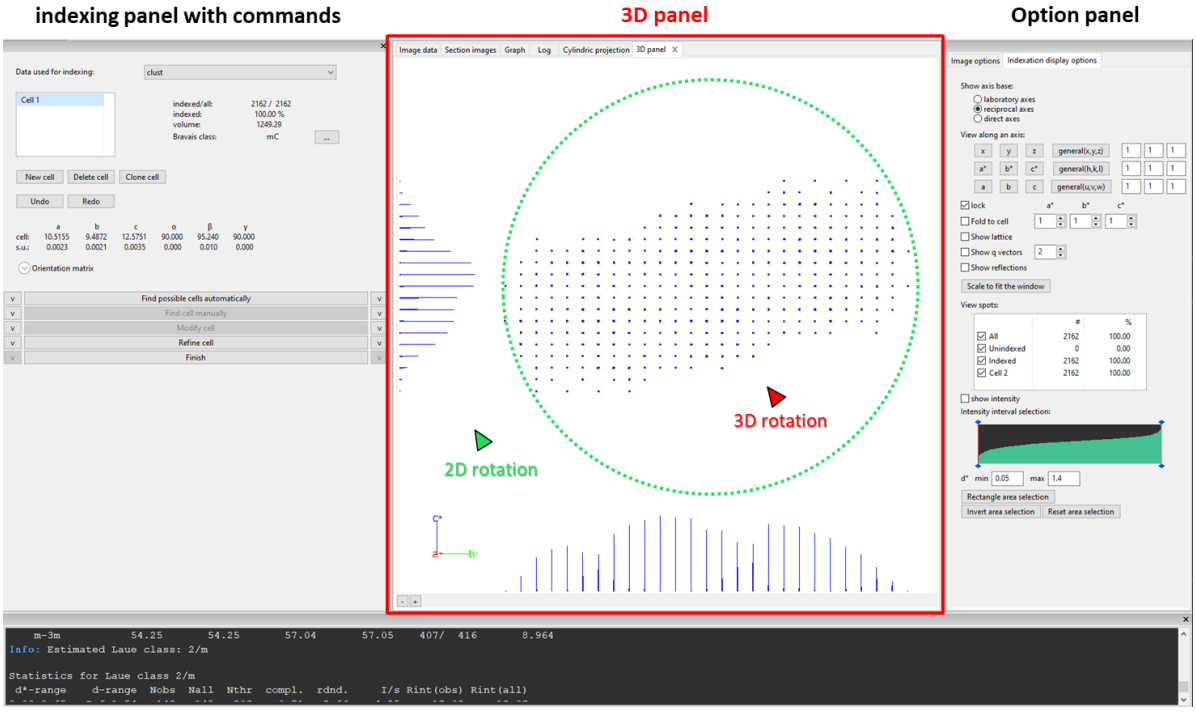


Figure B-76

On the left and the bottom of the 3D panel are 2D projections of the peak distribution, used as a help to see periodicity in the 3D projection.

B.5. Options panels

With two tabs: Image options and Indexation display options.

B.5.1 Indexing display options tab

The "**Indexing display options**" tab is activated when the menu "Find unit cell and orientation matrix" is opened and it influences the display of the 3D reconstruction of the 3D panel (Figure B-76).

B.5.1.a. Axis bases

Select one of the options to display the axis bases (laboratory axes **xyz**, reciprocal axes **a*b*c***, or direct axes **abc**) at the bottom left of the 3D panel (Figure B-77).

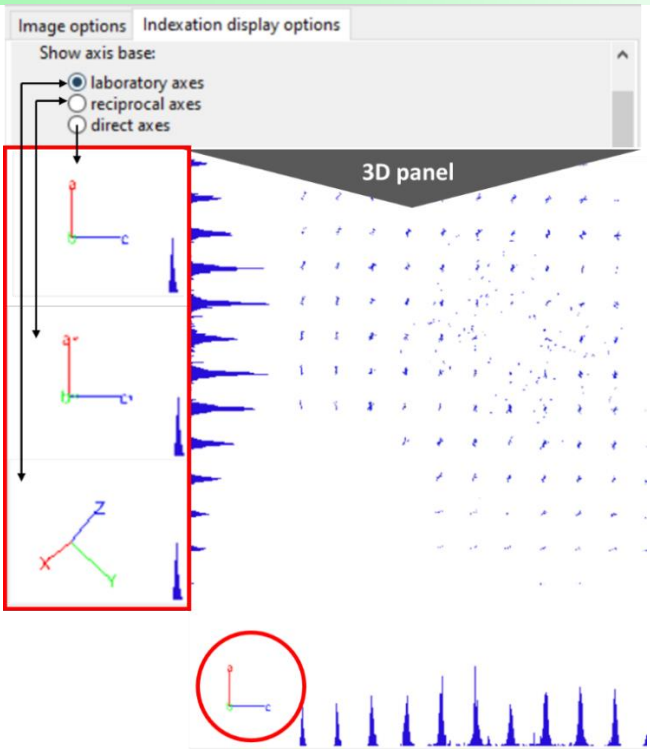


Figure B-77

B.5.1.b. View along an axis

To change the direction of the 3D reconstruction (in the 3D panel), use the buttons of the Options panel as shown below in Figure B-78. You can select the main directions or define a general one.

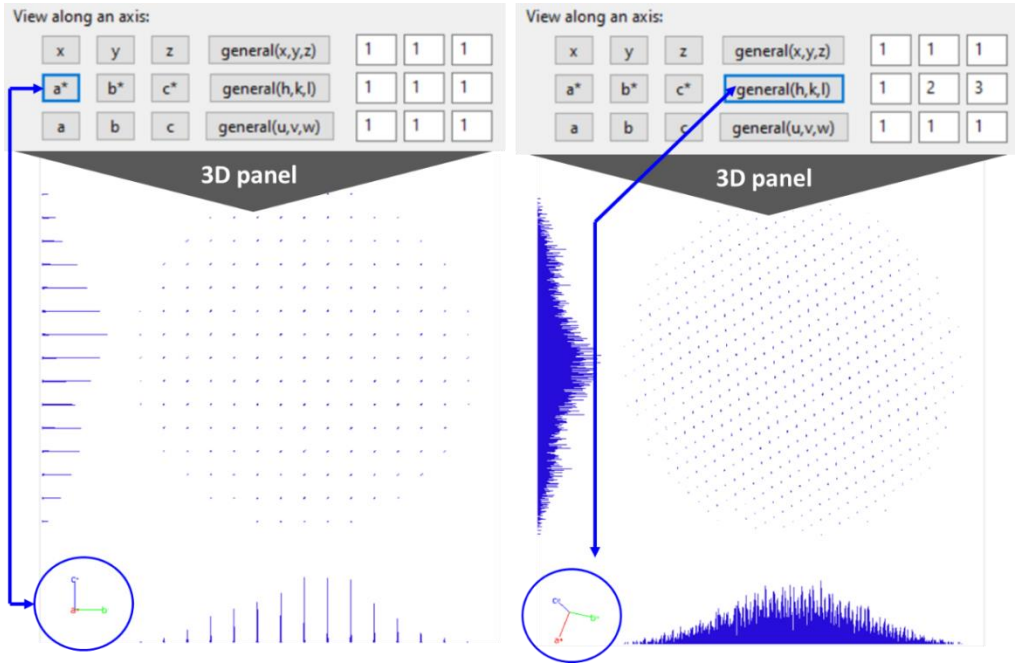


Figure B-78

B.5.1.c. Unit cells display options

- **Show lattice**: check “Show lattice” box to display the lattice as an overlay on the reconstruction. The unit cell overlay can be manually extended along **a***, **b*** and **c*** by changing the value as shown in Figure B-79 (right).
- **Fold to cell**: check the option “Fold to cell” to project all the 3D reconstruction into a certain number of unit cell units. The number of unit cells in which the 3D reconstruction is folded along **a***, **b*** and **c*** can be changed manually by changing the value as shown in Figure B-79 (right).
- **Lock**: Check the lock box to apply the same parameters to fold the cell and display the lattice overlay (see Figure B-79, left).

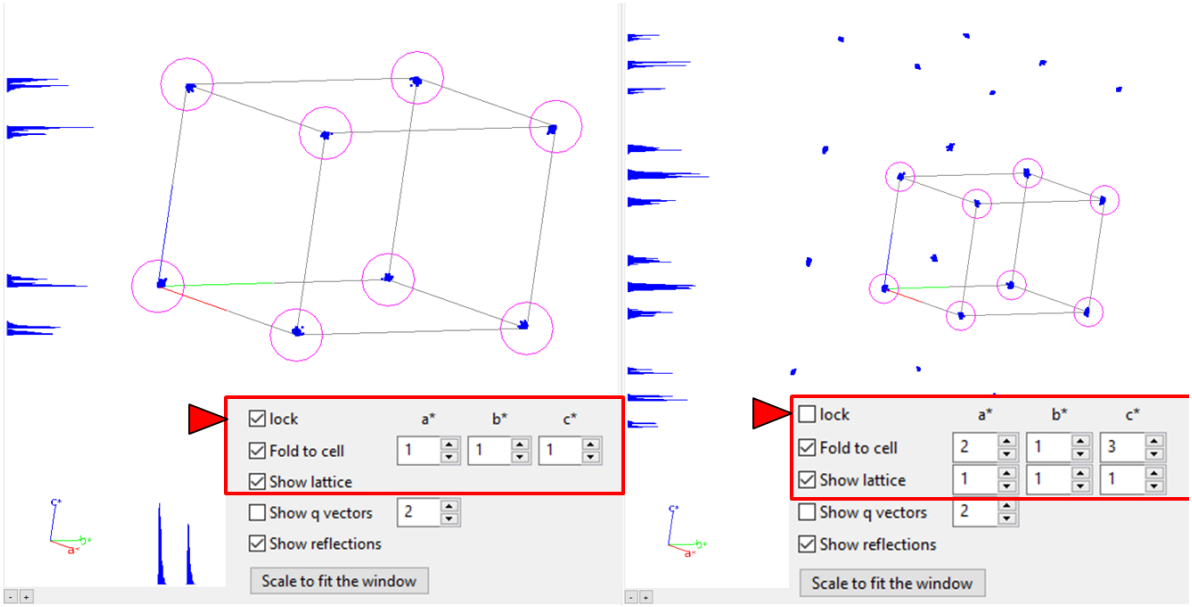


Figure B-79

- **Show reflections**: **main and satellite reflections**:
  - Check the option “**show reflections**” to display circles (purple in Figure B-79) on the main reflections. The diameter of the circles depends on the value of the maximal d* difference for indexing ($Å^{-1}$).
  - For modulated structures (Figure B-80), once one or more modulation vectors are defined, the satellite reflections can be displayed up to a given order (order = value next to the option) by checking the option “**Show q vectors**”. For more than one defined modulation wave vector, the combined **q** wave vectors will also be displayed (**q1**+**q2**, **q1**-**q2**, etc..).

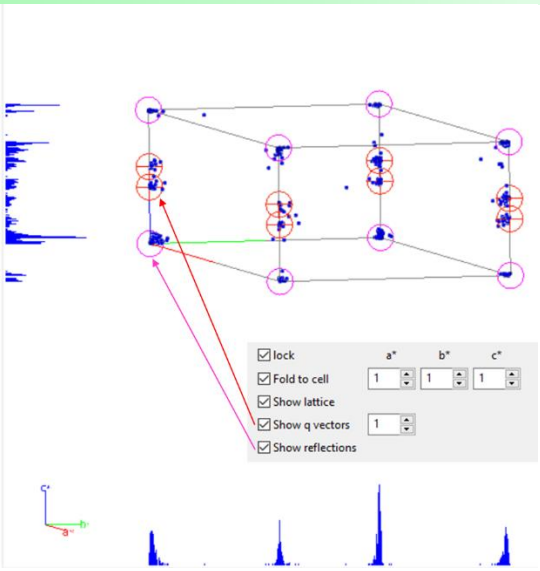


Figure B-80

- Scale to fit the window: rescale the view to match the window size (Figure B-81).

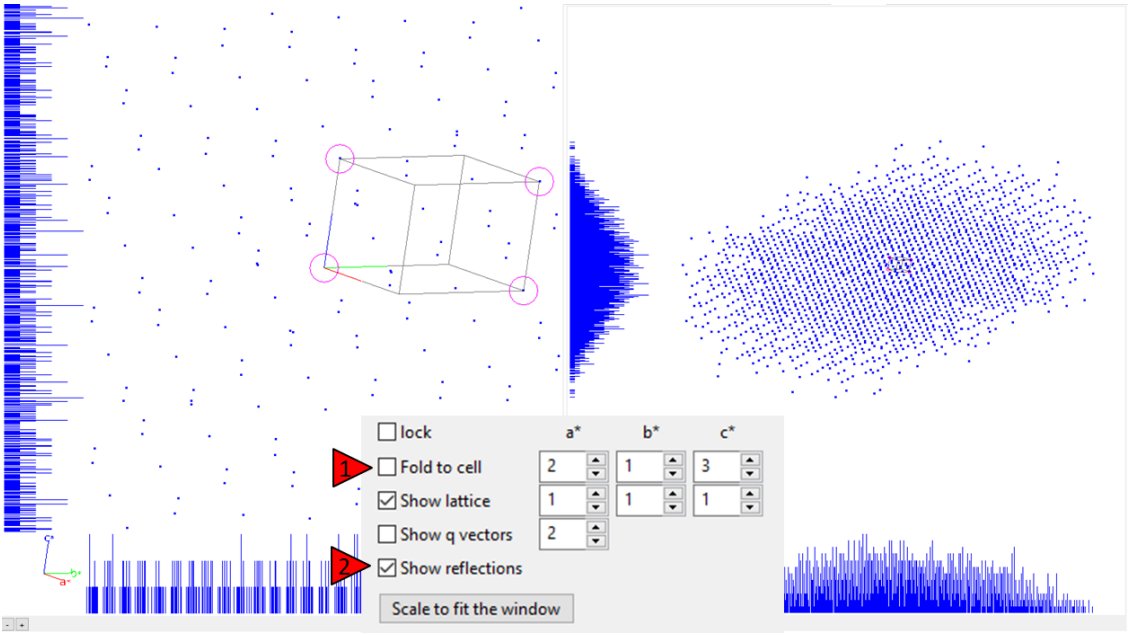


Figure B-81

B.5.1.d. Cloud display options

- **View spots: Selection of the visible spots**

By default, all the checkboxes in the section “View spots” are checked (see the middle of Figure B-82), meaning that all the reflections for a specific 3D reconstruction are displayed. To apply a selection to the reflections depending on their indexing, use the checkboxes as shown below (Figure B-82). In that example, only one cell (Cell1) is present because only one cell was defined. The list will contain as many cells as the user specifies.

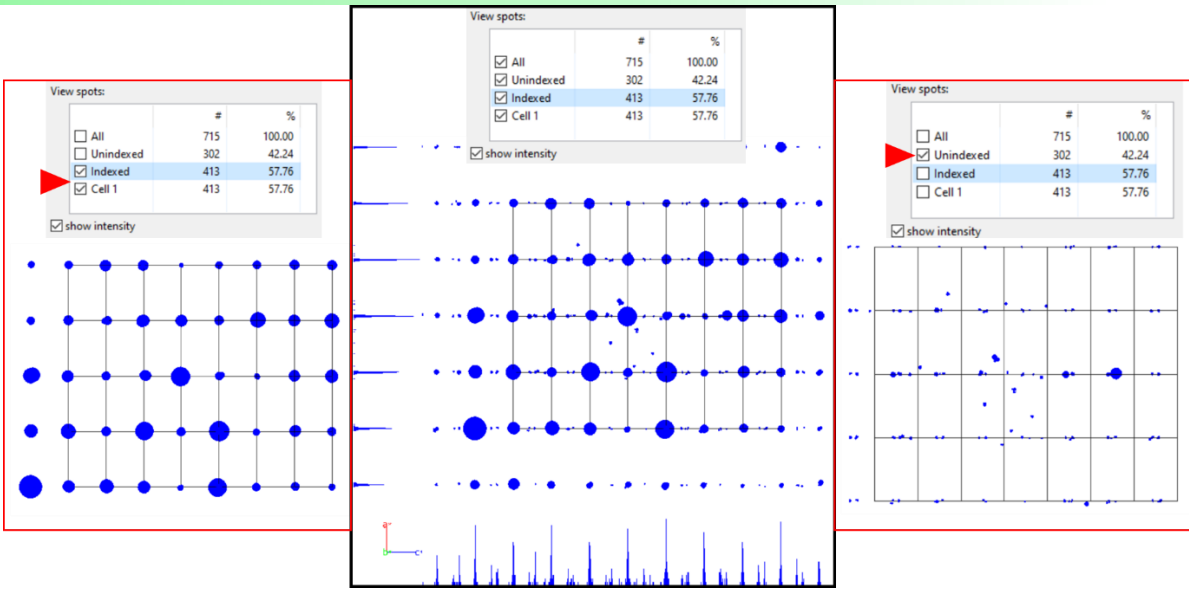


Figure B-82

➢ **Show intensity of the reflections**

Check the box "show intensity" to display the reflection with variable radius corresponding to their intensity (Figure B-83).

The intensity pattern might look different on the cor/xyz 3D reconstruction and the clustered files (clust and diff).

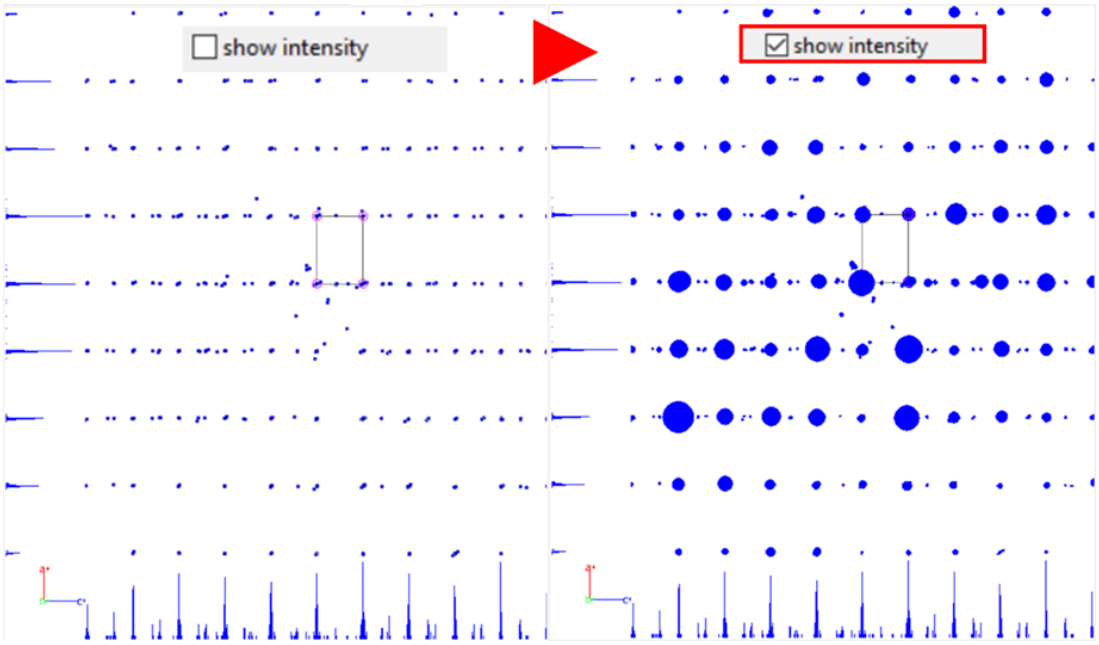


Figure B-83

➢ **Intensity interval selection**

In Figure B-84, the middle square was obtained with the default values for the intensity interval selection and the resolution range (**d***).

To skip the weakest/strongest reflections, move the left/right slider on the intensity distribution histogram (see Figure B-84, left).

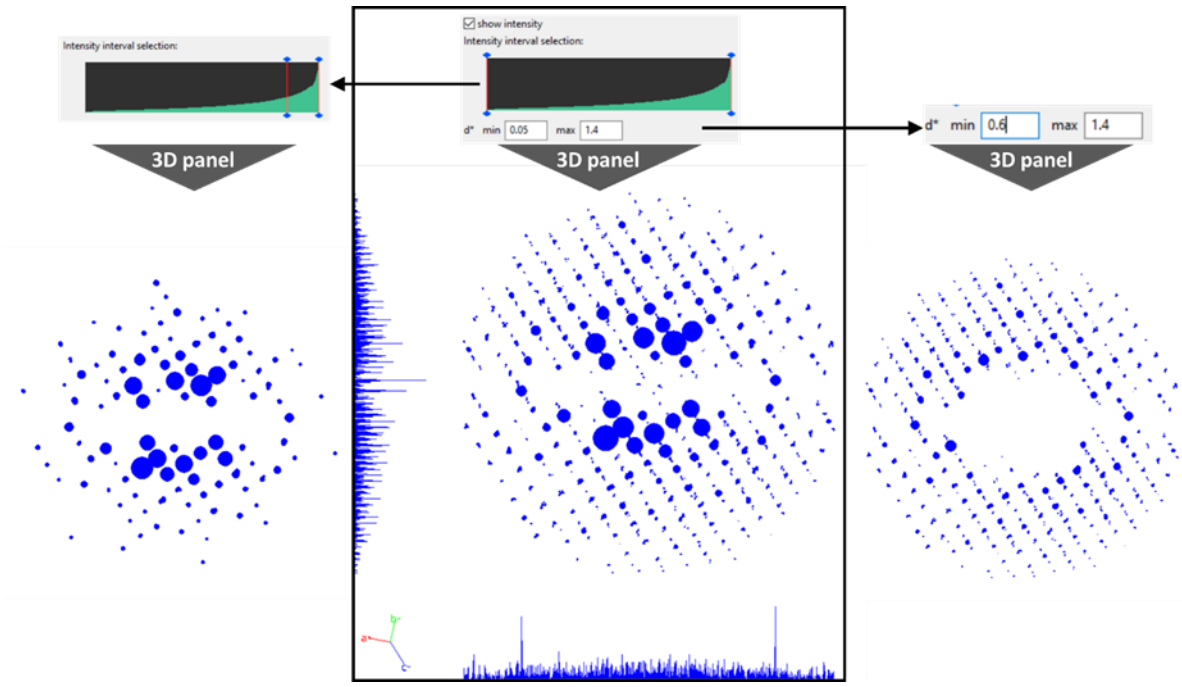


Figure B-84

➢ **Resolution area selection: d* min and max**

By default, the resolution range is defined from the d* min and max values used for the peak search in the "Parameters" of the commands panel (by default: from 0.05 to 1.4 $Å^{-1}$). To remove low-resolution or high-resolution reflections in the 3D cloud, change the values for d*min and d*max as shown in the right panel of Figure B-84.

➢ **Rectangle area selection**

- **Rectangle area selection**: to select reflections within a rectangular area of the 3D cloud (e.g., to select only one reciprocal-space layer), hit the button "rectangle area selection" and go to the 3D panel to drag the mouse to define the area (see red area in Figure B-85). The 3D panel will only display the selection (Figure B-85, middle).
- **Invert area selection**: To invert the selection (Figure B-85, right).
- **Reset area selection**: to go back to the complete 3D cloud.

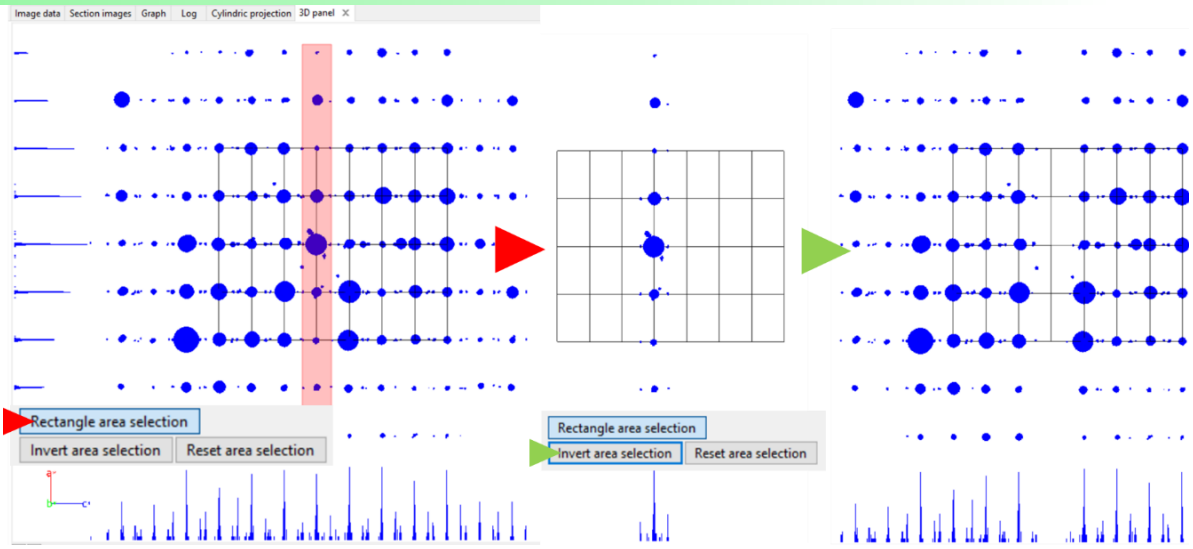


Figure B-85

B.5.2 Image options tab

All the options shown below will affect the Image tab of the Main panel, and some of them are also accessible at the bottom of the Image tab. Most of them were described previously in the "Image tab" section (section B.4.1).

B.5.2.a. General options

The general options are the image number that can be changed by hitting previous or next or changing the value of the frame (orange area in Figure B-86), the display cut-off (yellow), the image type (blue) and the possible overlays (pink) selected with radio buttons. All those options are defined previously in the Image tab section.

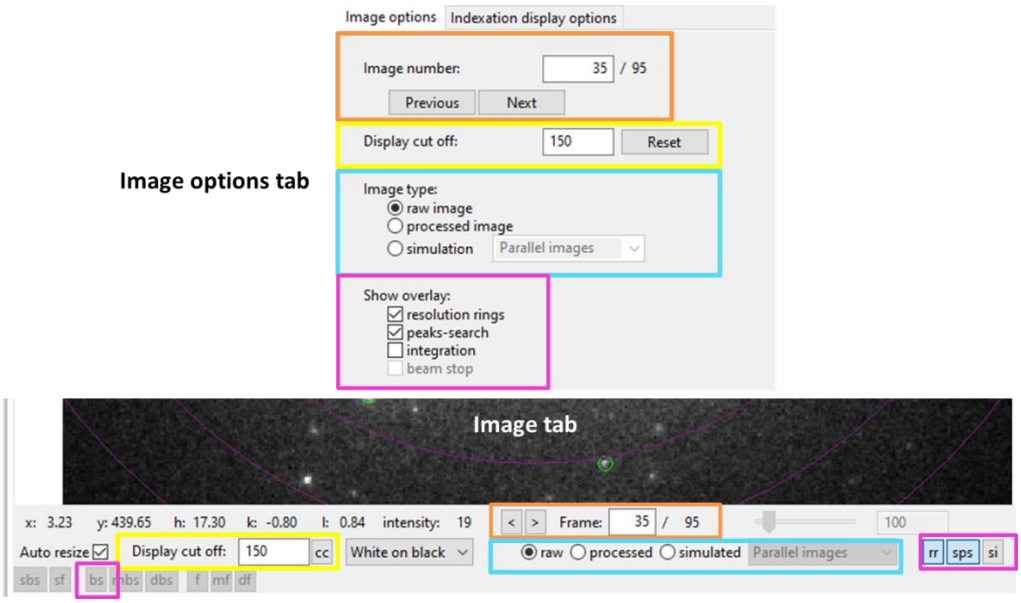


Figure B-86

B.5.2.b. Single frame options

The second part of the Image options tab is frame dependent (Figure B-87).
For each frame (= Image number selected above), the visible options are:

- A checkbox indicates if the selected frame is included in the calculation/data reduction.
- The x and y coordinates of the center for the selected frame.
- The tilt-angles (alpha, beta, Δomega).
- The intensity scale.
- The data identifier (useful for merged dataset).
- The distortions that can be defined frame-by-frame.

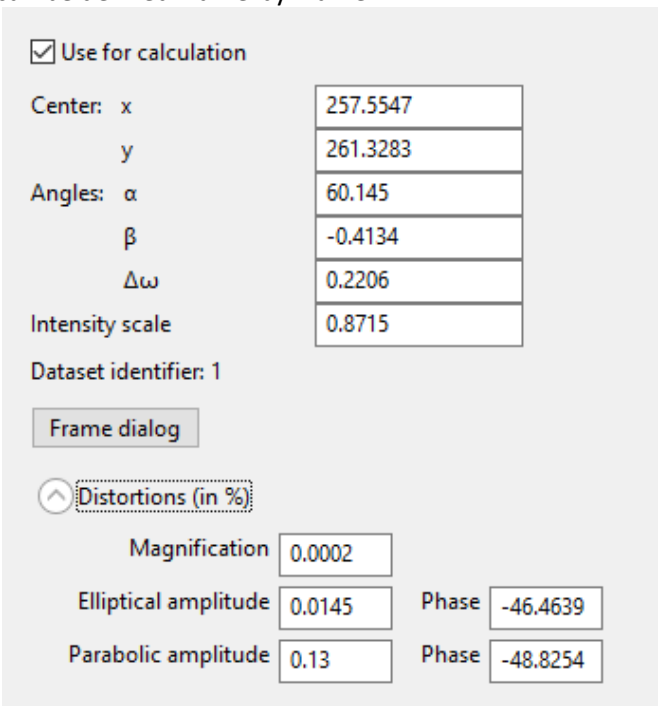


Figure B-87

- The **frame dialog button** (Figure B-87) opens the menu as shown in Figure B-88. This dialog provides options to edit several frames at once. This window was introduced in Sections A.4.1 and B.2.2.

Reminder: On the left side is a list of the frames with their number, the file name, and its path. For each frame, a check box on the left indicates if the frame is used in the data reduction or not.

To select all the frames of the list, press Ctrl+A in the window.

To select a limited number of frames: select the first frame to select, press SHIFT and select the last frame of the selection. At the bottom of the left window there are 3 buttons to define the alpha tilt from an existing list of frames (Define alpha), to add or remove single frames (Add/Remove).

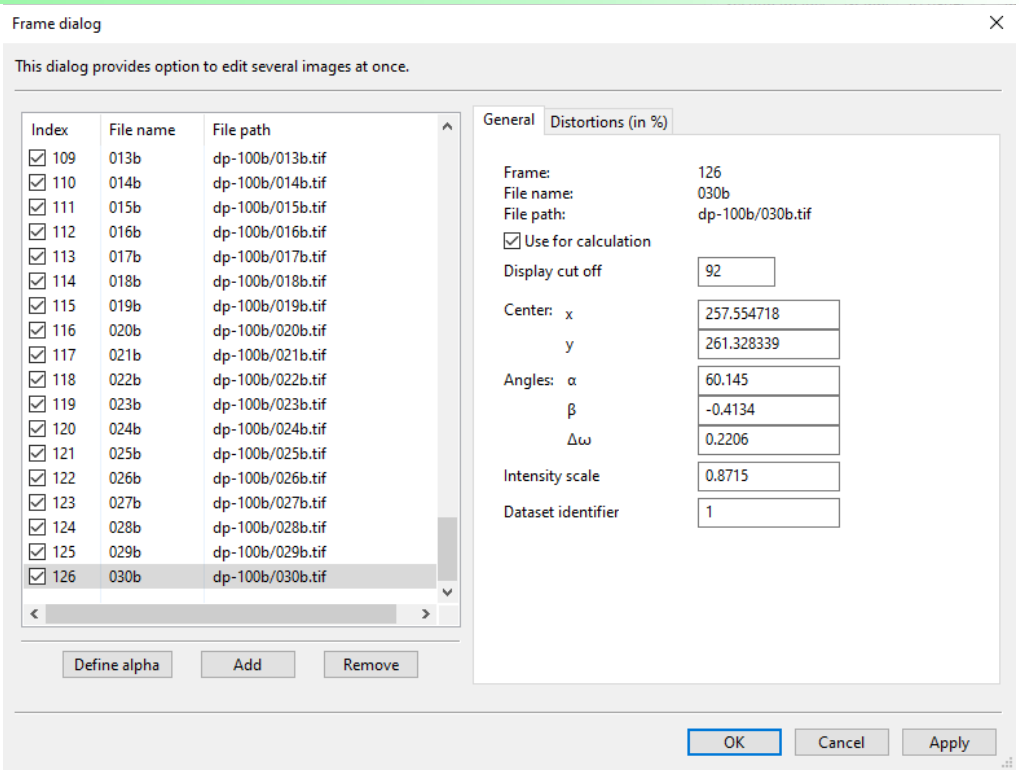


Figure B-88

To define the alpha-tilt for all frames or a selection of frames, hit “Define Alpha”. Then, Indicate the alpha-tilt (in degrees) of the first frame of your data set and the increment between two frames (α step) (Figure B-89). Press OK;

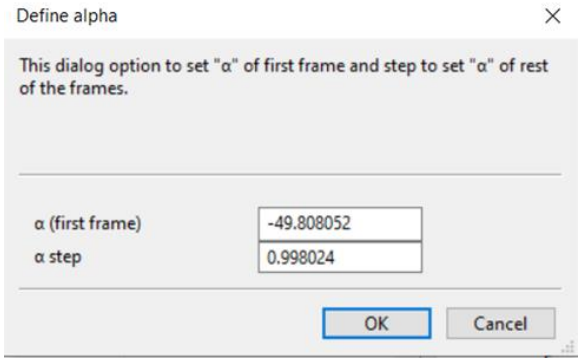


Figure B-89

The right part of the Frame dialog (Figure B-88) displays again what is already visible in the “Image options” tab for every frame (General information and distortions (in %)).

B.6. Console panel

Text window showing what is currently happening in PETS2 with log messages, info, warnings or errors. Keep an eye on that window every time a command is run.

Console search improved. Immediate search without the need to confirm with enter, "Previous" button, indication of the currently selected occurrence, always searching from the last position found, search results updated each time the console changes. Auto scroll of the console is activated when new procedure is started.

Most information given in the console panel will be later reported in the log files.

Below is an example of basic information you might get during a data reduction. When an error occurs, the console displays specific error messages. The message should be clear enough to understand the source of the problem.

- After opening an existing pts, or a pts2 file

```
Reading the input file test.pts.
The input file test.pts was succesfully read.
```

If the pts/pts2 file is invalid, an error message will appear in the console. As an example, here, one frame 097 listed in the pts file was not found in the subdirectory with all frames.

```
Reading the input file test.pts.
Error: File dp-100\097.tif does not exist.
Error: Error reading keyword imagelist.
Error: There were 1 errors in the input file.
```

- Peak search

For each frame, the console gives the beam position and the number of Friedel pairs. The last line is the total number of peaks detected above the I/sigma(I) set, and within the resolution limit.

```
>>>   96/  96 Image dp-100/096.tif: primary beam position:   254.03  264.46 from    2
Friedel pairs.

Minimum, average and maximum position of the primary beam:  253.89  254.87  255.33  |
262.81  264.53  265.09

Finished reading   2300 peaks from the file crs11p-100_petsdata/crs11p-100.rpl.
```

- Tilt axis: GLOBAL SEARCH

The console shows the list of tests performed to optimize the tilt axis. For each value of omega (10 degrees step) a score is evaluated: the higher the better. In the example below, a global search for the tilt axis orientation omega was carried out. The final omega value and the score is given, the rpl file is rebuilt with the new omega value.

```
Looking for the best omega
  Value   0.000, score   7.251
  Value  10.000, score   1.409
  Value  20.000, score   0.916
  Value  30.000, score   0.787
  …
  …
  Value 330.000, score   0.780
  Value 340.000, score   0.926
  Value 350.000, score   1.497
Final value of omega:
```

```
omega  :    0.000   7.251

Finished reading    2300 peaks from the file crs11p-100_petsdata/crs11p-
100.rpl.
```

- Tilt axis: FINE REFINEMENT

For a finer refinement of omega, the Δomega will be smaller. The refinement of omega is done first, the delta tilt axis position (if refined) is done after the omega refinement. The best omega and delta values correspond to the highest score values.

```
Refining the position of the tilt axis:
Refinement of omega:
  Value  -0.017, score  7.250745
  Value   0.003, score  7.251493
  Value   0.035, score  7.250298
  Value   0.003, score  7.251493
  ....
Refinement of delta:
  Value  -0.020, score  7.250822
  Value   0.000, score  7.251548
  Value   0.032, score  7.250340
  Value   0.000, score  7.251548
  Value   0.012, score  7.251393
  ....
Optimized angles:   0.00231  -0.00001   7.25155
Finished reading    2300 peaks from the file crs11p-100_petsdata/crs11p-
100.rpl.
```

- Peak analysis

STEP 1: In-plane distances in pixels (see Graph “in-plane distance plot” associated)

```
Finished reading    2300 peaks from the file crs11p-100_petsdata/crs11p-
100.rpl.
Number of entries in the distance table:   6277
Estimated threshold:   1.21 pixels.
Info: Press Continue, if you are happy with the result, or enter the new
threshold by clicking in the graph and then press Continue.
```

STEP 2 (Peak analysis (continue)): 3D distances in angstrom (see Graph “3D difference space distance plot” associated)

```
Thresholds for considering two peaks the same:  1.21 pixels, and  4.00 deg.

 2300 peaks merged into  796 clusters,   590 with more than one peak.

14988 difference peaks saved to crs11p-100_petsdata/crs11p-100.diff.

Number of entries in the distance table: 760075

Estimated threshold:   0.0068 A-1.
Info: Press Continue, if you are happy with the result, or enter the new
threshold by clicking in the graph and then press Continue.
```

STEP 3 (Peak analysis (continue)): result of the clustering (clust, xyz, diff reconstructions).

```
Thresholds for considering two peaks the same: 0.0068 reciprocal angstroms.
14988 peaks merged into 5666 clusters,  1332 with more than one peak.
Peak analysis successfully finished.
```

- Find unit cell and orientation matrix

FIND CELL AUTOMATICALLY:

```
Current cell was rewritten by the best found cell.
```

CHECK CENTERING:

When no new centering is found

```
Check centering: The centering aP    was confirmed.
```

When a new centering is detected (here C centering): a warning message appears to indicate that the initial centering (P) was automatically replaced by the new one (C).

```
Warning: Check centering: New lattice centering found. Centering vectors were
updated.
```

GO TO SUPERCELL:

```
Sorry, no frequent fraction vector was found.
No suitable supercell was found.
```

MODIFY CELL:

The console indicates if the modification could be performed or not and how the unit cell volume is affected.

```
Determinant of transformation matrix is  1.00, volume of cell is multiplied
by 1.00
Error: Determinant of transformation matrix is negative, please try again.
```

REFINE CELL:

The console gives information only when the unit cell is refined with distortions. The console shows the starting and the final lattice parameters with the new orientation matrix. For each cycle, the console displays the Root Mean Square Deviation (RMSD) between predicted and observed reflection positions (in pixels), the maximal parameter shift/e.s.d., and the number of indexed peaks/the total number of peaks. As the distortion parameters change, the rpl file and the cluster files of the 3D representations are recalculated after the refinement, as indicated in the two last lines. In the last line, 14990 peaks are, in fact, 14990 “difference space” peaks.

```
Finished reading     2300 peaks from the file crs11p-100_petsdata/crs11p-
100.rpl.
Starting cell parameters:  10.513   9.485  12.593  90.000  95.294  90.000
Cycle      RMSD  MaxShift  Number of good peaks/all peaks
    0     1.310      ----   2222/  2300
    1     0.832   27.7385   2246/  2300
    2     0.609   22.6593   2248/  2300
    3     0.536   13.3251   2248/  2300
    4     0.507    7.0302   2247/  2300
```

```
     5      0.502     3.4346   2247/  2300
     6      0.501     1.7174   2247/  2300
     7      0.501     0.8610   2247/  2300
     8      0.500     0.4349   2247/  2300
     9      0.500     0.2128   2247/  2300
    10      0.500     0.1077   2247/  2300
    11      0.500    -0.0700   2247/  2300
    12      0.500    -0.1498   2247/  2300
    13      0.500     0.1711   2247/  2300
    14      0.500     0.0319   2247/  2300
    15      0.500     0.0884   2247/  2300
    16      0.500    -0.2143   2247/  2300
    17      0.500    -0.0973   2247/  2300
    18      0.500     0.0236   2247/  2300
    19      0.500    -0.0116   2247/  2300
    20      0.500    -0.0943   2247/  2300
    21      0.500     0.0629   2247/  2300
    22      0.500     0.0861   2247/  2300
    23      0.500    -0.1704   2247/  2300
    24      0.500    -0.1188   2247/  2300
    25      0.500     0.1134   2247/  2300
Final orientation matrix:
  0.081640 -0.053859  0.013653
  0.049426  0.087485 -0.013047
  0.003020  0.024184  0.077602
Cell parameters:  10.518   9.475  12.575  90.000  95.298  90.000
Finished reading     2300 peaks from the file crs11p-100_petsdata/crs11p-
100.rpl.
 2300 peaks merged into  796 clusters,   590 with more than one peak.
14990 peaks merged into 5664 clusters,  1328 with more than one peak.
```

- Process frame for integration: the console only displays the frame processed, no specific information here.
- Optimize reflection profile

The combined values of RC width (rec. Å) and apparent mosaicity (in radians) are associated with a reliability factor that is minimized. After the list, the console gives the two optimized values with the RC width (rec. Å) and apparent mosaicity (in degrees). The number of reflections used to calculate the camel plot depends on the I/sigma(I) value given in the section “optimize reflection profile”, not the one used for the peak hunting.

The last sentence gives an idea of the real resolution of the data by giving the last resolution shell with more than 3 reflections per one point in the camel plot. In that example, the process frame for integration was carried out with a *d**(max) value of 1.4 rec. Å. The console indicates that not enough significant data is found above a resolution of 1.3 rec. Å to provide a reliable curve in the camel plot.

```
Rocking curve analysis:
Refinement of reflection profile parameters:
 0.001987 0.002517  1.1320
 0.001156 0.003865  1.0303
 0.000861 0.003950  1.0157
 0.000884 0.003910  1.0156
 0.000885 0.003910  1.0156

Refined values of relrodsigma and mosaicity:   0.00088   0.22401
Camel plot calculated from   1036 reflections.
```

```
Highest resolution shell with more than  3 reflections per non-zero grid
point:  1.30
```

- Optimize frame geometry

For each frame, the console will give the optimized alpha-tilt, beta-tilt, omega-tilt, the new *x* and *y* center of the frame, The RC width, mosaicity, sigma of the reflection size (in pixels), scale for the simulated frame, reliability factor (that should be minimized).

The values of the frame-by-frame distortions are not listed in the console. They are visible in the related graph and in the "Image options" tab for individual frames.

At last, the vector of the tilt axis in the microscope coordinates is given. Here 1 0 0 means that our tilt axis is almost perfectly aligned with the x-axis of the microscope. The second line reports the calculated angle that vector forms with the x-axis of the microscope (in-plane deviation) and the *xy* plane (out-of-plane deviation). Here again, in our example, the in-plane deviation is almost zero, and the out-of-plane deviation is 0.079 degrees.

```
Analysing frame nr.            1
Result:  -45.069389    0.332473    0.053814  254.777451  262.473419    0.000810
0.254500    1.257329    1.100700    0.9979
…
…
…
Analysing frame nr.           96
Result:   50.149651    0.216620   -0.051968  254.171509  264.361267    0.000810
0.254500    1.336086    2.883678    0.9812
Finished reading    2300 peaks from the file crs11p-100_petsdata/crs11p-
100.rpl.
 Optimization finished
Average orientation of the tilt axis:  1.00000  0.00002  0.00137
Angles from x-axis and from xy plane:   0.00125   0.07861
```

- Finalize integration

A log file "Finalize integration" with all helpful information is written simultaneously.

-The console gives first all the necessary parameters influencing the integration: *d**(max), the *RC width*, the *apparent mosaicity*, the integration mode, and the Laue class used for the frame scaling if applied.

-When the frame scaling is applied, the minimum and the maximum intensities found in the data set are given in the console. The integrated intensities are all rescaled using a factor that is a power of 10. This overall rescaling is a way to keep the accuracy carried by the decimals that can be otherwise lost in the data processing. The factor is set so that the largest intensity of the data set once rescaled stays below 100 000. As an example, here, the largest intensity is 204.63. The largest factor that can be used to stay below 100 000 is 100 (20463). This rescaling is an overall rescaling, not the frame-by-frame scaling that is displayed in the log files, the graph tab "frame scales", as well as in the Image option tab for individual frames.

-The three parameters used to correct the sigma(I) are given in the console (see section B.3.9).

-The following line gives the number of outliers kicked out of the final *hkl*-type files. In that example, 45 out of 3879 reflections are rejected using the parameters outlier detection limit for symmetry averaging = 1.5 (default value) (see section B.3.9).

Intensity statistics:

- Observed/Total number of unmerged integrated reflections.

-Intensity statistics for each Laue class. The table is Laue class/Rint(obs)/Rint(all) /Rmeas(obs)/Rmeas (all)/Number of observed reflections (for I/sigma(I) > 3) /Number of all reflections/ redundancy.

After the list, PETS2 evaluates the most likely Laue class. Note that this estimation and the statistics are influenced by the Laue class used to scale the frames (if used).

-Another table presents the statistic by resolution shell (in d* and d) as following:

Resolution shell in rec.Å/ Resolution shell in Å/ number of observed reflections/number of all measured reflections/ number of theoretical reflections possible given the symmetry/data completeness for the resolution shell/ redundancy/Intensity/sigma(I)/Rint(obs), Rint(all), and CC1/2.

```
*** Kinematical integration ***
Settings:
  dstarmax:  1.400
  RC width:  0.00100
  mosaicity:  0.100
  integration mode: fit profile
  frame scaling: applied, Laue class 2/m

Refinement of frame scales...
Profile-based outlier rejection:
Number of peaks considered: 13719
Number of peaks rejected  :     0
Total percentage rejected :  0.00%
Total percentage rejected :  0.00%
... successfully finished.
Minimum and maximum intensity before rescaling:       -6.92      204.63
Integrated intensities rescaled by a factor       100.00
Error model refinement...
Refined parameters of the error model: s_fac = 0.7286, s_b =  0.0932, s_add =  0.2524.
Info: 45 out of  3879 reflections identified as outliers at the rejection limit of
1.50 and rejected.

Intensity statistics:
Observed/Total number of integrated reflections:  2297/ 3834
cwl Laue class Rint(obs)   Rint(all)  Rmeas(obs)  Rmeas(all)    Nobs/Nall  redundancy
 *  -1             9.36       12.36       13.22       17.45   1172/ 1984       1.932
 *  2/m           13.62       17.29       16.74       21.44    803/ 1371       2.796
    112/m         31.37       33.76       37.06       40.34    760/ 1359       2.821
    2/m11         29.80       32.04       35.30       38.30    756/ 1335       2.872
    mmm           36.17       38.59       40.15       43.19    487/  882       4.347
    4/m           47.68       49.76       52.74       55.39    467/  875       4.382
    4/mmm         51.61       54.09       54.92       57.83    290/  549       6.984
    -3            26.85       29.72       31.96       35.90    985/ 1691       2.267
    -31m          44.64       48.09       50.21       54.39    615/ 1097       3.495
    -3m1          47.65       50.37       53.82       57.25    635/ 1142       3.357
    6/m           45.19       48.22       50.51       54.33    599/ 1106       3.467
    6/mmm         53.76       58.00       58.23       62.90    388/  743       5.160
    m-3           43.56       47.08       46.89       50.84    340/  640       5.991
    m-3m          53.21       56.95       55.96       59.98    209/  418       9.172
Info: Estimated Laue class: 2/m

Statistics for Laue class 2/m
 d*-range    d-range  Nobs  Nall  Nthr  compl.  rdnd.     I/s Rint(obs) Rint(all)   CC1/2
0.00-0.47   Inf-2.14    52    56    79   0.709   2.68    4.74      9.03      9.30   96.93
0.47-0.66  2.14-1.52    78    93   132   0.705   2.80    4.60     10.41     11.25   95.58
0.66-0.81  1.52-1.24   109   127   175   0.726   2.89    4.32     14.16     15.18   96.11
0.81-0.93  1.24-1.07   113   143   193   0.741   2.84    4.04     15.54     16.88   94.85
```

```
0.93-1.04   1.07-0.96   121   163   228   0.715   2.80    3.90     15.97     17.87    90.74
1.04-1.14   0.96-0.87   101   167   227   0.736   2.84    3.50     15.16     19.82    90.87
1.14-1.23   0.87-0.81   110   207   283   0.731   2.84    3.15     15.48     22.19    86.93
1.23-1.32   0.81-0.76    76   209   282   0.741   2.81    2.66     19.64     31.68    64.36
1.32-1.40   0.76-0.71    43   206   295   0.698   2.65    2.03     27.10     51.75    50.29
 ------------------------------------------------------------------------------------------
0.00-1.40   Inf.-0.71   803  1371  1894   0.724   2.80    3.84     13.62     17.29    96.23

*** Dynamical integration ***
Dynamical integration finished successfully.
```

B.7. Output subdirectory

The pts2 file is found in the same directory as the output hkl-type files (hkl, cif.pets, dyn_cif.pets and _sheklx.hkl files).

A subdirectory jobname_petsdata contains all the output files/directories (Figure B-90):

- Logs: folder with all the log files
- Plots (png)
- Processed: folder with the list of processed frames (TIFFs, background-free)
- Simulation: folder with the list of simulated frames during the geometry optimization (TIFFs)
- Sections: folder with the sections of the reciprocal space (tif) and the subfolder with the saved sections (cutoff and color theme in png)
- A list of output files (see section E, OUTPUT FILES)

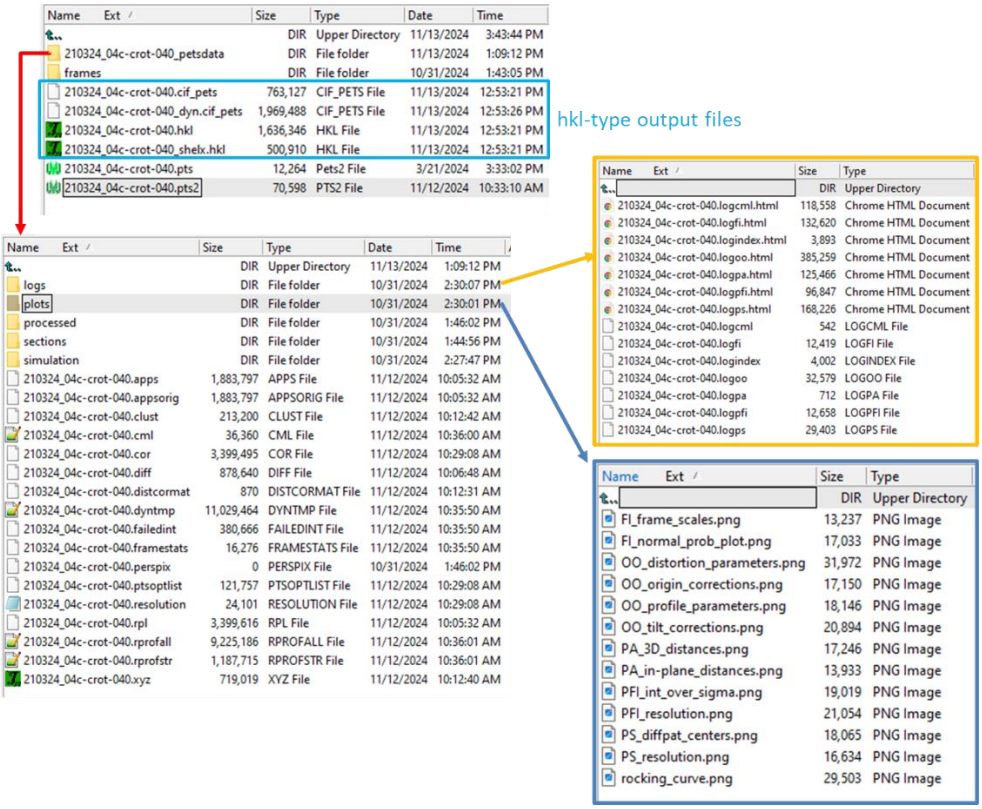
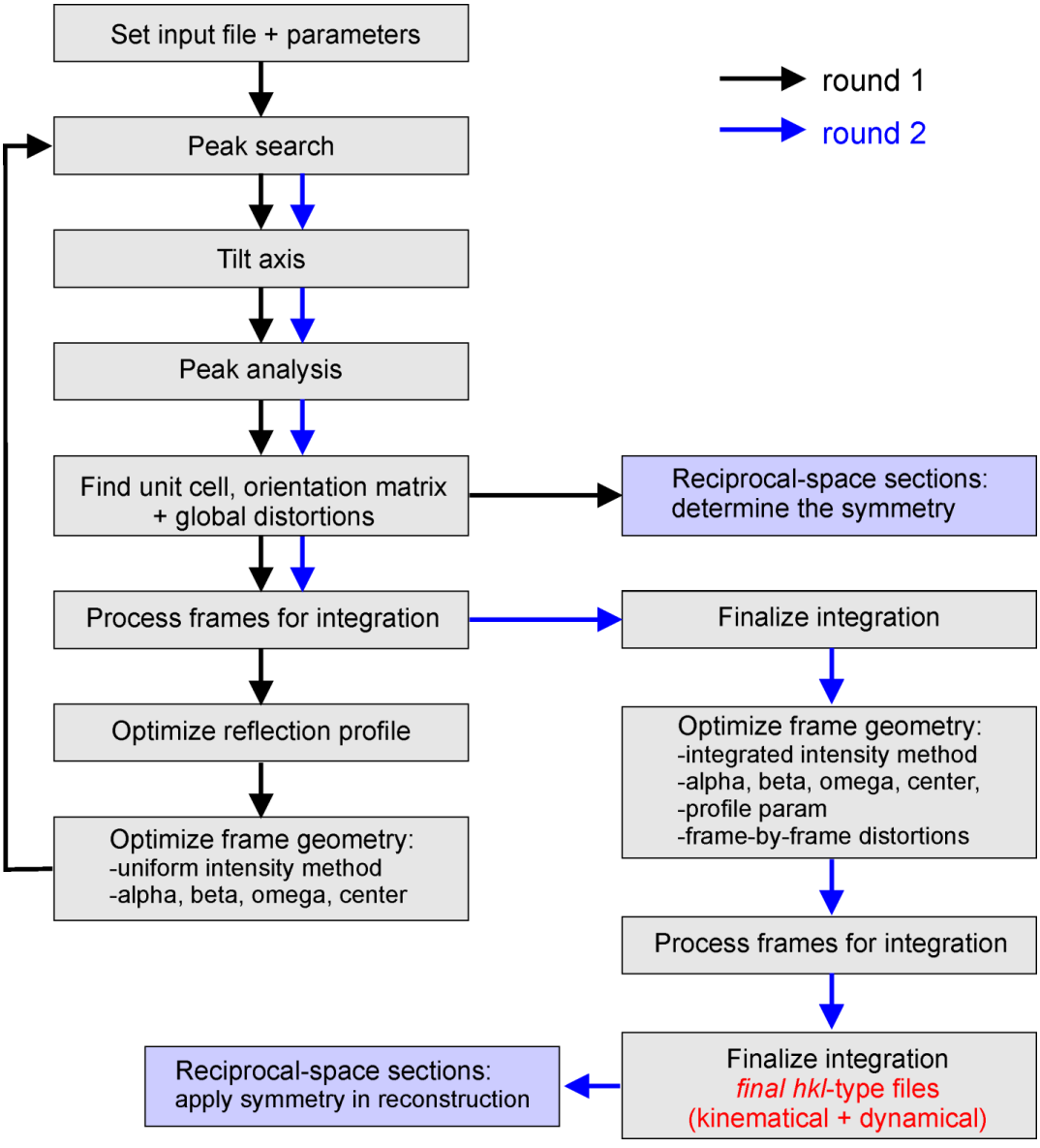


Figure B-90

C BASIC PROCEDURE

The scheme below represents a standard procedure that should lead to good results for good 3D ED data. For non-standard/difficult data, the procedure may need to be adjusted, but this is still a good start. We recommend for the second round keeping the refined centers for the new peak search.



D KEYWORDS AND OPTIONS AVAILABLE IN THE PTS2 INPUT FILE

List of tasks and options to be automatically performed after the start.

The table summarizes all the keywords/commands in a PTS2 file with their options (red). Although most are accessible from the GUI; some will be changed in the input file (GIU yes/no).

The comments (labeled #) are already in the PTS2 file above the corresponding keyword. A more extended description is given for some keywords (labeled ##).

Note that to start a basic data reduction, a simpler PTS file can be created (as already explained in section A). When the data reduction starts, a pts2 file is generated with all entries, and it needs to be saved.

Command default option/value	**#comments/options present in the input file pts2,** **##additional information**	**GUI**
autotask endautotask	# peak search, tilt axis, peak analysis, find cell, refine cell, process frames, reflection profile, frame geometry, finalize integration, generate 3d map, sections, check files, global indexing, set, quit, break. ## keyword set can be followed by any valid one-line command. The effect is the same as if this command was read from a pts2 file. E.g., "set reflectionsize 10" will change the reflection size to 10.	yes
keepautotasks no/yes	#If yes, list of tasks to be automatically performed after the start is not cleared when all tasks are finished.	yes
lambda value	#wavelength in angstroms ##lambda: relativistic wavelength of the incident electrons in angstroms. For U_{acc} = 100kV, λ_{100} = 0.0370Å, 120kV: λ_{120} = 0.0335Å, 200kV: λ_{200} = 0.0251Å, 300kV: λ_{300} = 0.0197Å.	yes
aperpixel value	#image calibration in reciprocal angstroms/pixel ## calculated as aperpixel = (pixel size for one given detector)/(λ(in Å) * camera length calibration)	yes
geometry option	#experimental geometry. Options: continuous rotation, precession, static.	yes
detector default	#detector type. Options: default, asi, olympus, cetad. More to come... ## When default is selected, nothing is done on the frames. With asi, the cross due to the 4 detector blocks is corrected, the detector noise parameters preset as well as the saturation limit.	no

	The correction is fine-tuned for the ASI Cheetah detector. Please contact us if you need a correction for another detector type.	
noiseparameters 4.2 0.00	#Parameters allowing to calculate the sigma(count) on every pixel. The first is G*gamma (gain*cascade factor), the second is sigma**2 of the dark image. ##Those two values depend on the detector and need to be adjusted for your camera.	yes
phi value	#precession or integration (semi)angle. Must be zero for static geometry.	yes
omega value 0 1	#angle between the positive horizontal axis on the image and the projection of the rotation axis on the image. Two keys define if global search and local refinement should be performed (0 = not refined, 1 = refined).	yes
delta value 1	#angle between the positive main rotation axis and the x-y plane (perpendicular to the beam). Positive, if the angle between the axis and the beam is smaller than 90 degrees. The key defines, if delta should (1) or should not (0) be refined.	
pixelsize 0.0100	#size of the pixel in the 3D map of reciprocal space.	yes
bin value	#binning of the images after reading in. Should be an even number.	yes
reflectionsize value	#Diameter of integration circle of the reflections (pixels, unbinned)	yes
dstarmax 1.4	#maximum dstar (rec.angstroms) for integration ## The default value is 1.4 rec.angstroms	yes
dstarmaxps 1.400	#maximum dstar (rec.angstroms) for peaksearch ## The default value is 1.4 rec.angstroms	yes
dstarmin 0.050	#minimum dstar (rec.angstroms) for integration and peaksearch ## 0.05 = default value	yes
centerradius 0.150	#excluded region around the central beam. Obsolete, partly overlapping with dstarmin. Will be removed/replaced soon.	no
beamstop no OR beamstop 8 223 283 210 293 263 81 250 12 236 endbeamstop	#beamstop settings. beamstop no with no beamstop. "beamstop followed by a list of beamstop points, if beamstop is used. ## With no beamstop the entry is beamstop no. ##With a beamstop the entry becomes beamstop x1 y1 x2 y2 etc. endbeamstop ##Beamstop defines a polygon that is excluded on each frame. It is a multiline list of coordinates, each line is one point of the polygon, x and y, unbinned pixels.	yes

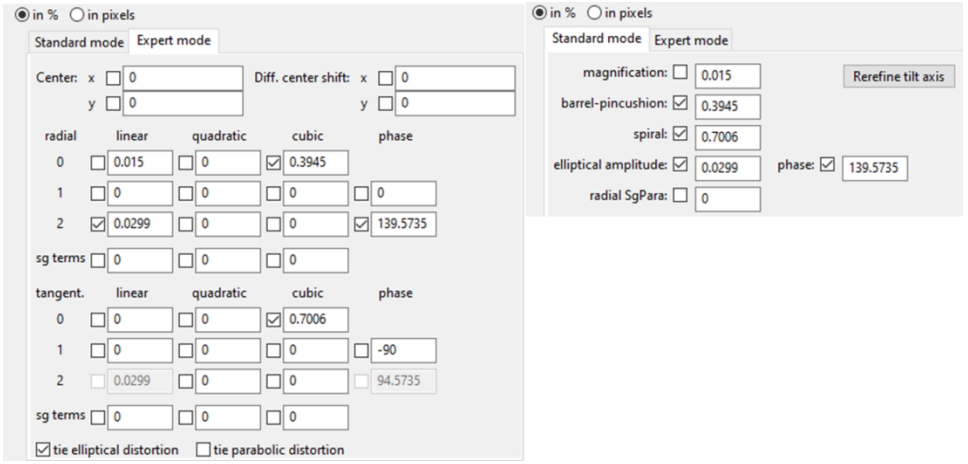
	The easiest way to define the beamstop is to use the GUI.	
Badpixels 8 223 283 210 293 263 … endbadpixels	#list of coordinates of bad pixels. These pixels are ignored, and their value is interpolated from the values of the neighboring pixels.	yes
Avoidicerings yes/no	## yes to activate the ice ring mask	yes
icerings 0.2735 0.4446 endicerings	#list of ice ring diameters. Peaks in the vicinity of these rings are ignored, if avoidicerings is yes.	yes
peaksearchmode auto	#Peak search mode - auto (default) = find automatically, manual = click in the picture to add or remove found peaks.	no
center AUTO	#center of the diffraction pattern. Options: AUTO = find center automatically; two numbers x y - position of the pattern center on the first frame in pixels. ## For a manual determination of the center, the x and y value (un-binned) can be read in the pets' GUI from the Image data tab below the frame pictures. The entry is written (as an example: center 250 240). The manual option is useful if the automatic procedure fails to determine the center. The more accurate option is to use Friedel pairs in the GUI.	yes
centermode friedelpairs 1	#Mode of center determination. centralbeam = use central beam, friedelpairs = use friedel pairs, useold = take whatever was previously set. Optional second argument: always accept center? (0/1). Applied only to Friedel pairs.	yes
i/sigma 5.00 10.00	#Intensity/sigma for peak search and for the calculation of the camel plot	yes
mask -500 -500 500 -500 500 500 -500 500 -500 -500 endmask	#mask defines a polygon that is excluded on each frame. It is a multiline list of coordinates, each line is one point of the polygon, x and y, unbinned pixels	
moreaveprofiles yes/no	#Option for average peak profile calculation in the fit profile. If yes, each group of frames will have its own calculated average peak profile.	yes

peakprofileparams 100 1 35.0	#Parameters for average peak profile (APP) calculation. First: minimum number of peaks in one APP. Second: minimum I/sigma peaks (0=fixed/1=auto). Third: value of minimum I/sigma for the case fixed.	yes
peakprofilesectors 0 1 0.57	#Parameters for specifying image sectors for APP calculation. First: distinguish image sectors (0/1). Second: radius of the central sector (0=fixed/1=auto). Third: radius value for the case fixed .	yes
background 0 1.000000	#Constant background to be subtracted from all images after reading in. May be negative if needed. Second optional argument is the factor that multiplies the raw image at reading ##The first number is a linear transformation of the input intensities (background threshold). The second number is a scaling of the background.	no
backgroundmethod median	#Method for background subtraction. median = median filter (default, the best), average = average in concentric circles, fft = background by Gaussian smearing. Median should be used in normal circumstances. #3 ways of subtraction of the Bg, median is best and other options might be removed in later versions, fft removes low frequencies from the image. median>fft>average.	no
peakanalysis 0 0 3.2262 0.0044	#Peak analysis options: estimate cluster limits automatically (0) or use preset values (1), process automatically (1) or wait for confirmation (0), xyz cluster limit (in pixels), 3D cluster limit (in rec. Angstroms).	yes
minclusterpoints 2	#Minimum number of points in a cluster (used during peak analysis). ##2 is the default value	no
indexingmode diffspace	#Algorithm for automatic cell determination. diffspace = new algorithm using difference space clustering, triplets = old "jana-style" algorithm. ##diffspace and triplets are two different algorithms of automatic indexing. diffspace procedure uses the clust reconstruction. Triplets option uses the reconstruction selected in the GUI (clust, xyz, or cor).	yes
indexingparameters 0.0140 0.6000 3.0000 1.0000	#Parameters for cell determination: indexing tolerance (rec. A), relative length tolerance (%), angular tolerance (deg.), maximum Sg for indexing (distortions only). ##The first number = indexing tolerance is used both for indexing and the refinement of the unit cell parameters. 0.6 degree and 3% are the default angular and length tolerances respectively. They are used during the indexing to guess the possible crystal system and centering. The last number is the excitation error Sg considered for the refinement. 1 is almost like infinite.	Yes
maxindex 2	#maximum satellite index used in integration. (Applies only to modulated structures.) # for 3D periodic structure, this parameter is ignored	yes

cellrefinemode cellanddistort	#Algorithm for cell refinement. cellandub = refinement from 3D peak coordinates (standard), cellfromd = refinement against lengths of the diffraction vectors, cellanddistort = cell from 2D peak positions plus, optionally, distortion parameters. ## The refinement with distortions automatically uses the cor file reconstruction. For the other refinement options, the selected reconstruction is used.	yes
cellrefineparameters 2 1 1	#Parameters for cell refinement: crystal system index, key to refine cell in cellanddistort (0/1), key to refine distortions in cellanddistort (0/1). # crystal system index: 2 = monoclinic, refine cell or not = 1/0, refine distortions/not = 1/0). This part will be filled through the GUI.	yes
referencecell 10.00000 10.00000 10.00000 90.00000 90.00000 90.00000 0 0	#Cell reference to be used in cell refinement (a b c alpha beta gamma). The 7th parameter says if lattice scaling is allowed (0/1). The 8th parameter specifies if the cell should be used also as a source for orientation matrix in global orientation search. ##When the 7th number = 1, the lattice scaling is fixed up to a scale factor keeping the *a/b, b/c, a/c* ratios. If 0, the lattice parameters will be refined to be exactly what is expected. This last option is only accessible for the refinement with UB cell.	yes
intensitymethod profilefit 0	#Method for peak intensity determination: profilesum or profilefit. Second parameter: if 1, a two-pass integration is performed ## a two-pass integration = It performs the integration two times, first time with allowing shift of the peak's positions, then it calculates the aver age shift for each reflection on all frames, and then the second integration uses that shift of integration of the reflection on all frames. It is supposed to give a better result for weak reflections.	yes
adjustreflbox yes	#If yes, integration circle will be shifted to maximum intensity during intensity extraction. Useful for distorted data, dangerous for dense patterns. ##this option is now rather robust when combined with the profilefit intensitymethod.	yes
resshellfraction 0.5000	#Limit between inner and outer shell for the calculation of average I/sigma (shown in the plot made during integration + in the file framestats). ## Multiplier of the dstart value used to process frame for integration. The shell below dstar*threshold is considered as the inner shell. It does not influence the integration steps but is a way to visualize some part of the resolution range when needed.	no
saturationlimit value	#Above this number the pixel is considered saturated.	yes

	## 13000 is the default value for our CCD camera, 64000 for asi, etc.	
skipsaturated no	#If yes, reflections containing saturated pixels are not written in the output reflection list. # For structure solution, it is often best to keep all reflections, even saturated. For the refinement, you might consider removing them.	yes
minrotaxisdist 0.0250	#minimum distance from rotation axis in reciprocal angstroms.	yes
minreflpartiality value	#minimum reflection partiality in percentage. Default = 70%	yes
rcshape pv	#Shape of the reflection profile. gaussian, pv (pseudovoigt: RCwidth Lorentzian, mosaicity Gaussian), lorentzian.	no
integrationmode fit 1 5 3 0.800	#Integration settings: first the mode: fit, integrate, bestpattern or maximum. Second value: do framescaling? (0/1). Third value: Laue class number for frame scaling. Fourth value: range for frame scale restraints. Fifth value: weight of the frame scale restraint. ## The third number is called interframe correlation range and represents how many frames are considered with close scaling, the fourth number = interframe correlation weight and defines how strongly the scales of consecutive frames are forced to be close, higher is the number, stronger is the restriction. By default, those two numbers are set to 0. A good starting point is 3 and 0.8.	yes
integrationparams 0.0010 0.1000 1 1.00	#parameters of the profile - rocking curve width (rec. angstroms), mosaicity (deg.), minimum number of measurements per reflection, range of integration (as a multiple of the sigma of reflection profile).	yes
intkinematical yes/no	#If yes, kinematical intensity integration will be performed in Finalize Integration.	yes
intdynamical yes/no	#If yes, dynamical intensity integration will be performed in Finalize Integration	yes
dynamicalscales yes/no	#If yes, dynamical intensities for dynamical refinement are divided by the frame scales (= corrected like the kinematical intensities).	yes
dynamicalerrormodel no	#If yes, sigma(I) for dynamical processing is modified by the error model coefficients (= the same treatment as sigmas in the kinematical processing).	no
errormodel 1.0000 0.0000 0.0000 1 1.50	#Error model parameters. - s_fac, - s_b, -s_add. $sigma^2 = s_{fac}^2*(sigmacnt^2+s_b^2*int+s_{add}^2*int^2)$, with int the maximum intensity of symmetry related reflections. -the refinement flag (1= refine error model).	yes

	-level for outlier rejection.	
outliers 1.50 1000.00	#Outlier thresholds. First: threshold for outliers in symmetry averaging. Second: threshold for profile fitting.	yes
refinecamelparams 1 1 0 1 0.0020	#Keys of the camel refinement. -1=apply, 0= don't apply: -refine RC width, -refine mosaicity, -refine precession angle. -Automatic/User defined camel plot step (0/1), -camel plot step. (1: RC, 1: mosaicity, precession angle, step size in the RP, step value) 1 means checked not refined.	yes
orientationparams 1 1 0 1 -3 0 2	#Keys for the orientation optimization. -1=apply, 0= don't apply: -opt. of tilt angles, -opt. of frame centers, -RC width, -mosaicity, -frame-by-frame distortions (binary code - 1st bit magnification, second elliptical, third parabolic), negative means not optimized. -transfer average distortions to global distortions. -Last key selects integrated intensities (=1) or uniform (=2).	yes
simulationpower 0.500	#An exponent applied to experimental and simulated counts in orientation refinement. Typically, <1 to suppress overweighting of the strong reflections in the fit. (Applied in the orientation refinement, it fits pixels. If very strong reflections, they tend to dominate the fit. If they are also off, the fit is not successful. We can take all intensities to one power (0.5) to reduce the sensitivity to strong reflections).	no
interpolationparams 0 0 0.00	#Keys for interpolation of orientation optimization values. -First: mode of interpolation (0 = none, 1 = polynomial, 2 = moving average). -Second: order (polynomial order for poly fit, half-range for moving average). -Third: level for outlier rejection (in sigma).	yes
distcenterasoffset yes	#If yes, the center of distortions is assumed to be a constant offset from the center of the diffraction pattern. Otherwise, constant coordinates of the distortion centers are assumed. ## yes/no	yes

distortunits percent	#Units in which distortions are displayed and stored. pixels or percent.	yes
distortions 0.0000 0.0000 0.0000 0.0000 0.015000 0.000000 0.394500 0.000000 0.000000 0.000000 0.000000 0.000000 0.029900 0.000000 0.000000 139.573502 0.000000 0.000000 0.700600 0.000000 0.000000 0.000000 0.000000 -90.000000 0.029900 0.000000 0.000000 94.573502 0.000000 0.000000 0.000000 0.000000 0.000000 0.000000 enddistortions	#Distortion parameters.  ## By default all values are 0 excepted for the phases. In the example, the refined values in the PTS2 files are given as a comparison with where they are found in the GUI, in the standard and the expert modes. For more details, see the section related to distortions.	yes
distortionskeys 0 0 0 0 0 0 1 0 0 0 0 0 1 0 0 1 0 0 1 0 0 0 0 0 2 0 0 2 0 0 0 0 0 0 enddistortionskeys	#Refinement keys for the distortion parameters. ## 1 = refined, 0 = not refined, 2 = all elliptical distortions are tied together: i.e., the second order of the tangential distortions (elliptical distortions) are tied to the radial component of the second order distortions (elliptical too).	yes
mapformat xplor no	#Format of the 3D map of reciprocal space. May be xplor, ccp4 or hdf5. Second argument: apply symmetry in the reconstruction (no/yes)	yes
reconstruction hk0 1.000 0.000 0.000 0.000 1.000 0.000 0.000 0.000 0.000 hk1 1.000 0.000 0.000 0.000 1.000 0.000 0.000 0.000 1.000 h0l 1.000 0.000 0.000 0.000 0.000 1.000 0.000 0.000 0.000	Sections of reciprocal space. Each line contains name, first vector (3 coords), second vector (3 coords) and origin (3 coords).	yes

0kl 0.000 1.000 0.000 0.000 0.000 1.000 0.000 0.000 0.00…. Endreconstruction		
reconstructionparams 0.0070 0.0140 1	#Parameters for the sections of rec. space. Pixel size (rec. angstroms) and thickness of the summed region perpendicular to the section. Switch from 0 to 1 to apply symmetry in the reconstruction. # The thickness is in Å, and 1/0 = apply/don't apply the symmetry in the reconstructions.	yes
removebackground yes yes	#If yes, background subtraction is done on reconstructions. If no, raw images are used. -First argument: 2D reconstructions (rec. space sections), -2nd argument: 3D map reconstruction.	no
serialed 2.000 1.000 180.00 0 0	#Settings for global orientation search by template matching. Three reals: step between templates (deg.), max resolution of templates (rec.A), maximum deviation from the original orientation. Then a switch to use reference cell (1) or current orientation matrix (0). Finally, a switch to perform or not fine refinements of the result (0/1).	yes
virtualframes 7 4 -1 1	#Virtual frames -first the number of summed frames, -second the step between frames, -third 1 to sum all intensities, only intensities on the virtual frame, -1 - decide based on geometry. -fourth: determine the first and second number automatically/manually: 1/0.	yes
Cifentries ….. endcifentries	#List of entries to be propagated further to the structure analysis pipeline. May contain experimental info, PETS augments it with data-processing related entries.	no
imagelistheader imgname alpha beta domega alphaorig betaorig domegaorig xcenter ycenter intscale diffbfac magcorr elliamp elliph paraamp paraph useforcalc dataset	# Order of items in each line of the image list ## Only the first three columns are needed to start a basic run from a simpler version of the pts input file done manually or generated from in-house software. The additional columns will appear when the project is saved in a pts2 file during the procedure.	no
imagelist "framefolder\name001.tif" alpha beta "framefolder\name002.tif" alpha2 beta2 etc. endimagelist	#List of images ## Only the first three columns are needed to start a basic run from a simpler version of the pts input file done manually or generated from in-house software. Additional columns will appear when the project is saved in a pts2 file during the procedure.	yes
Celllist	#Orientation matrix and cell parameters	YES

cellItem active .... endCellItem cellItem .... endCellItem endcelllist	## The old content of the celllist file is now found after the imagelist. The structure remains the same. A list of cellItem/endCellItem. In each cekkItem: the name, the ub matrix, celldir, cell parameters, cell volume, cell s.u., cell symmetry, cell centering, cellHistoryCodes, cellHistoryParams. Active in front of cellItem means that unit cell is used in the data reduction. celllist cellItem name Fortho ubmatrix 0.046632 -0.000519 0.021335 0.052664 0.001861 -0.018865 -0.001037 0.070871 0.000658 celldir 0.662595 -0.007530 0.748709 0.748832 0.026351 -0.662504 -0.014811 0.999624 0.022880 cell 14.2147 14.1049 35.1039 90.013 90.020 90.004 cellvol 7038.19 cellsu 0.0000 0.0000 0.0000 0.000 0.000 0.000 cellSymmetry t cellCentering F cellHistoryCodes 3 1 2 0 cellHistoryParams 0.014 2.000 5000.000 3.000 0.600 0.014 3526.000 7119.000 0.074 1.425 endCellItem cellItem active name mono1 ubmatrix -0.026122 0.000581 0.083012 -0.071042 -0.001872 -0.074182 0.001650 -0.071398 0.002621 celldir -0.666518 0.008140 0.938558 -0.745355 -0.026210 -0.344719 0.014119 -0.999623 0.016676 cell 14.2074 14.0007 9.6593 90.000 111.616 90.000 cellvol 1786.24 cellsu 0.0004 0.0012 0.0003 0.000 0.002 0.000 cellSymmetry a cellCentering C cellHistoryCodes 3 1 1 2 cellHistoryParams 0.014 2.000 5000.000 3.000 0.600 0.012 4743.000 7119.000 0.074 1.426	

	endCellItem endcelllist	

E OUTPUT FILES

List of all output files created during the data reduction and given either in the same directory as the PTS2 file or in subdirectories.

Folder\File	**When produced**	**Description**
Jobname.pts2	Whenever a pts/pts2 file is saved	A pts2 file is the main input file with all necessary commands to run the data reduction from a tilt series of frames. See **Section C**
Jobname_petsdata\ Jobname.rpl	Peak search	rpl stands for raw peak list. 12 columns: Frame-by-frame peak list, 1-3: raw 3d coordinates in the Cartesian reference coordinate system (microscope settings labelled xyz in the GUI). The coordinates in the raw file are not corrected for effects like tilt-axis refinement etc. and should not be used for any other than internal purposes. The corrections are applied to the coordinates in the Jobname.cor file. 4: integrated intensity, 5: sigma(I) 6-7: x and y positions of the intensity maximum on the frame (integer, unbinned) 8-9: x and y refined positions of the peak when the distribution of the intensity is considered. 10: alpha angle 11: maximum intensity value 12: frame number where the peak is found
Jobname_petsdata\ Jobname.cor	Peak search	12 columns: For each peak detected during the peak search and sorted by their integrated intensity, 1-3: 3d coordinates (corrected with the omega rotation axis) in the Cartesian referential (microscope settings labelled xyz in the GUI)

		4: integrated intensity, 5: sigma(I) 6-7: x and y positions of the reflection on the frame (integer, unbinned) 8-9: x and y refined positions of the peak when the distribution of the intensity is considered. 10: alpha angle 11: maximum intensity value intensity 12: frame number where the peak is found *Remark: when the box "show intensity" in the indexation display options tab is checked it corresponds to the integrated intensity.*
Jobname_petsdata\ Jobname.perspix	Peak search	x and y coordinates of badly behaving pixels that will be ignored in all frames. This list is not frame dependent and is generated for each dataset automatically during the peak search.
Jobname_petsdata\ Jobname.resolution	Peak search	6 columns For each frame: 1: frame path\name 2-6: 5 highest resolution peaks from the highest to the lowest. The resolutions are expressed as the length of diffraction vector in $Å^{-1}$. *Note: this file gives rough information on the resolution in each frame.*
Jobname_petsdata\ Jobname.app	Peak search	the average reflection profile used for the fitting procedure
Jobname_petsdata\ Jobname.xyz	Peak analysis step 2	The xyz file is already a clustered file of the cor file. 11 columns: 1-3: 3d coordinates (corrected with the omega rotation axis) in the Cartesian referential (microscope settings labelled xyz in the GUI) 4: integrated intensity, 5: sigma(I) = 0 because there is no sigma for a clustered file 6-7: x and y positions of the reflection on the frame (integer, unbinned) 8-9: x and y refined positions of the peak when the distribution of the intensity is considered. 10: peak number in the cor file. There are always a group of peaks in the cor file that are merged together in the xyz file. The column is just the position corresponding to the first peak of the group in the cor. 11: number of peaks in the cluster

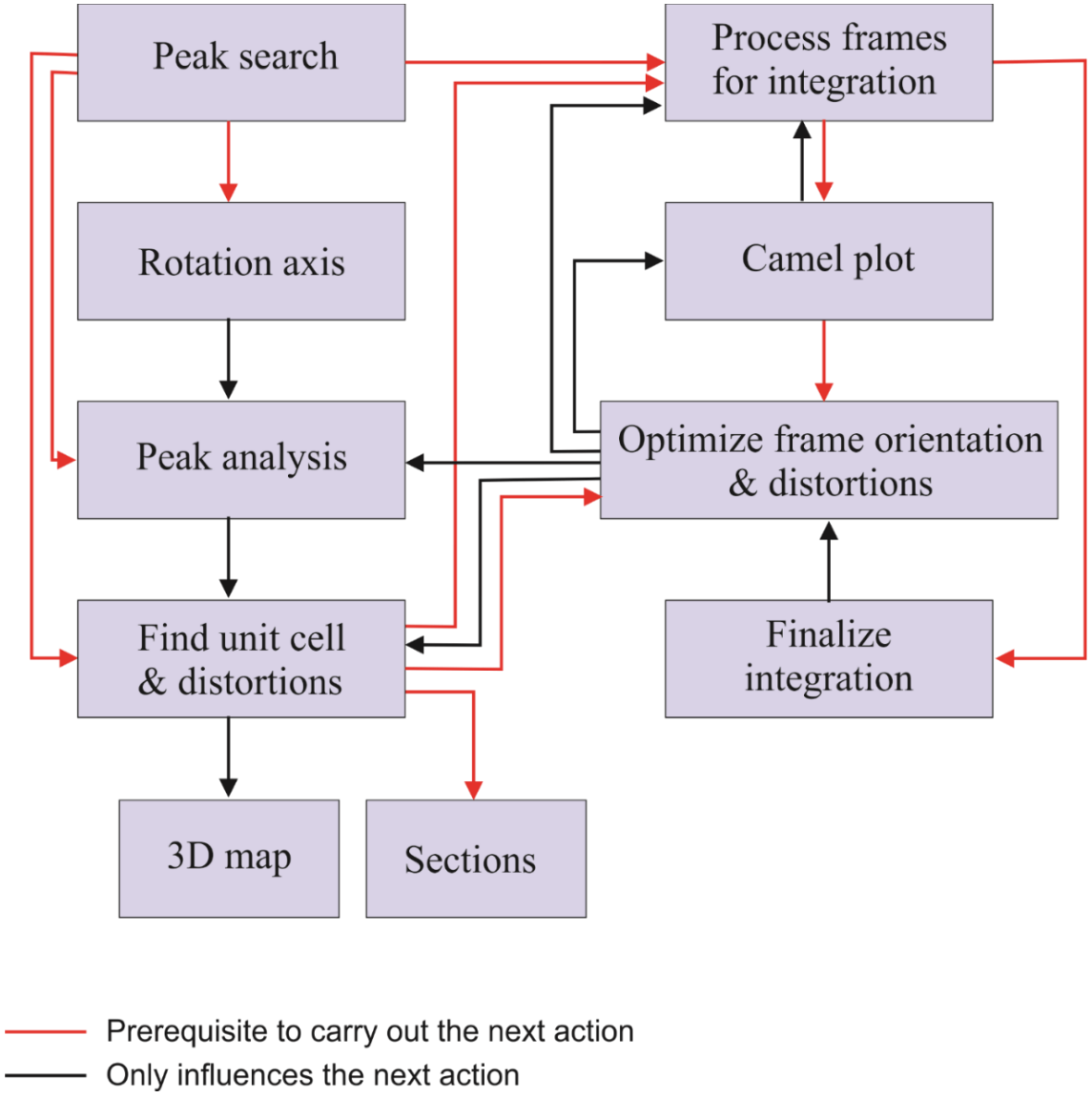
Jobname_petsdata\ Jobname.diff	Peak analysis step 2	5 columns 3d coordinates (xyz), not real intensity, geometric mean of the intensities of the the two peaks that form the difference vector. (the larger this number, the more intense on average are the two peaks), the last number is the same as the previous. The file follows the hkl intensity sigma format, but no sigma is assigned, hence, the intensity is repeated.
Jobname_petsdata\ Jobname.clust	Peak analysis step 3	xyz, I, sigma, number of peaks contributing to the cluster.
Jobname_petsdata\ Jobname.sphtest	Find unit cell auto	The output file of the procedure finds unit cell automatically with all observed directions, possible cells found by the algorithm, and the selected cell.
Jobname_petsdata\ Jobname.symmlog	Find unit cell auto	This file is the output file from procedure Check centering. In this procedure search for the highest symmetry (centered) cell is provided. Input unit cell must be primitive (not centered) cell. Jobname.symmlog contains at the beginning the reduced cell corresponding to input unit cell. After this cell, information is given about cells found during the search for higher-symmetry centered cells. The cell information contains cell parameters, cell volume, UB matrix and Bravais class, and transformation matrix from the primitive to the centered cell. At the end of Jobname.symmlog, there is a final observed cell with the highest symmetry. If cubic symmetry is observed also hexagonal and tetragonal cells are considered. If hexagonal primitive cell is observed also orthorhombic cells are considered.
Jobname_petsdata\ Jobname.distcormat	Ref with distortions	Distortion correlation matrix showing correlations between the parameters refined during the cell and distortion refinement. Name of param, value of param, (standard uncertainty). Number of the param from 1 to… 1: Stars = large correlation (>0.8).
Jobname_petsdata\ Jobname.framestat	Process frame for integration	It contains the numbers shown in the plot average I/sigma, i.e. the average I/sigma in lower and higher resolution shells.
Jobname_petsdata\ Jobname.failedint	Process frame for integration	Contains a list of reflections that are not integrated properly: reflections close to an edge or beamstop. They will be filtered out in the finalized integration step.
Jobname_petsdata\ Jobname.dyntmp	Process frame for integration	15 columns h k l, Intensity, sigma, integer position of the peak x and y on the image predicted, refined position (if shift integration mask is used), frame number, number of saturated pixels, last four numbers = resolution (length of the dif vector), excitation error, RSg (can be negative), distance of the reflection from the 0 plane (z

		coordinate of the reflection – not really useful in 3DED data, introduced for special purposes).
Jobname_petsdata\ Jobname.cml	Process frame for integration/ Optimize reflection profile.	Camel file: resolution shell, excitation error, *RSg*, calculated R curve, experimental RC.
Jobname_petsdata\ Jobname.rprofall	Process frame for integration/ Optimize reflection profile.	Reflection profile for all reflections H k l Resolution, excitation, *RSg*, *Iobs*, sigma, *Icalc*, frame number. Azimuth of the reflection = angle from the horizontal axis in radians. This file was made to be plotted in gnuplot, each block is one reflection, this file is a list of all reflections.
Jobname_petsdata\ Jobname.rprofstr	Process frame for integration/	Reflection profile strong (measure 4 times, and if at least one of the reflections that form the profile as an I more than 20 sigma). Same as the previous one but with strong reflections only.
Jobname_petsdata\ Jobname.ptsoptlist	Optimize frame geometry	Copy of the console after the geometry optimization (will be soon removed and placed in the log file)
Jobname_petsdata\ Jobname.cif_pets	Finalize int	The basic output file with the CIF format, for structure solution and kinematical refinement. Contains integrated reflection intensities and standard deviations with a header giving the basic unit cell and experimental information
Jobname_dyn.cif_pets	Finalize int	The basic output file with the CIF format, for dynamical refinement. Contains frame-based reflection intensities and standard deviations with a header giving the basic unit cell and experimental information and the information on the (overlapping virtual) frames.
Jobname.hkl	Finalize int	H k l list with I and sigma(I)
Jobname.xyzcheck	Write check files	for diagnostic and development purposes
Jobname.corcheck	Write check files	for diagnostic and development purposes
Jobname_petsdata\processed*.tiff	Peak search	Processed frames in TIFF format
Jobname_petsdata\simulation*.tiff	Optimize frame geometry	Simulated frames in TIFF format
Jobname_petsdata\sections	Reciprocal-space sections	Folder containing the sections (tiff)

Jobname_petsdata\sections\png	Reciprocal-space sections/save sections	Folder containing the sections (png) initially created with the sections. Those sections can be updated using different cutoff, color themes etc. and can be more conveniently used to display the sections in documents/presentations than the 16bit tiffs. New sections can be produced using the new settings by selecting “save sections” in the toolbar of the “Section images tab”.
Jobname_petsdata\Jobname.xplor /ccp4/hdf5	Generate 3D map	3D map of intensities in the reciprocal-space. Can be viewed by external programs like VESTA or UCSF Chimera
Jobname_petsdata\ Jobname.gos	Serial ED	Short listing of the global orientation search /SerialED run. For each frame it lists the orientation angles of a couple of the best matching templates and the associated figures of merit. Only symmetry independent templates are listed.
Jobname_petsdata\ Jobname.gosfull	Serial ED	Like Jobname.gos, but with the complete listing of all best matching templates, including the symmetry related. Only for checking and development purposes.
Jobname_petsdata\ Jobname.templ	Serial ED	The definition of the templates. The header contains the settings used to generate the templates (orientation matrix, maximum resolution, angular step between templates). For each template the angular setting of the template is given, followed by the list of coordinates of all peaks with their intensity

F DEPENDENCY SCHEME

This scheme shows how each action relates to other possible actions in PETS2. Performing an action can either be necessary before another action is performed (marked by a red arrow), or it may change some parameters/settings that may influence the result of the other action (black arrow).



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