

Int3D: a data reduction software for single crystal neutron diffraction

Nebil A. Katcho, Laura Cañadillas-Delgado, Oscar Fabelo, Juan Rodríguez Carvajal

Why Int3D?

Status of the software in the single crystal diffractometers of the ILL (D9,D10,D19)

- Outdated software (dates from the 80s, Fortran77, inefficient...)
- Software split in multiple pieces
- Difficult to deal with complex problems (twins, incommensurability)
- Very limited and low quality data visualization
- Demands high expertise (high workload for local contacts)

The Int3D software

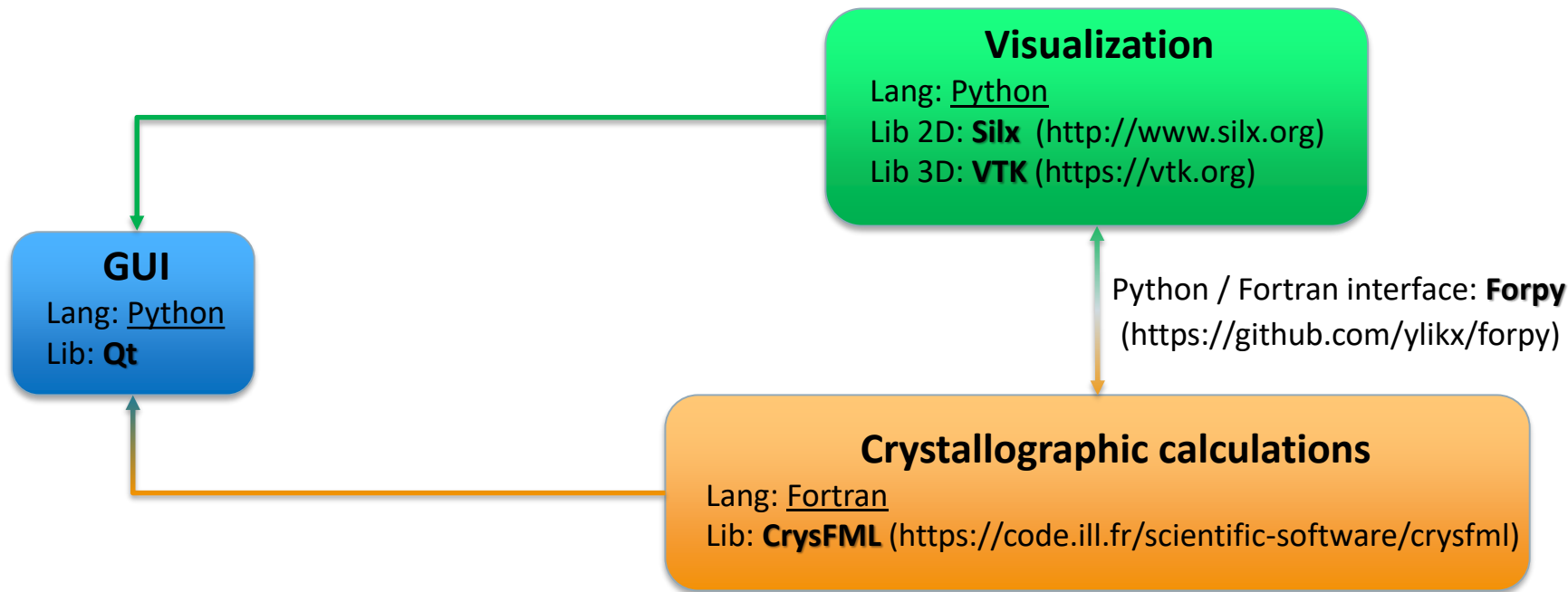
- Integrates in a single application all the required tasks to perform the data reduction:

- Peaks search
- Determination of the orientation matrix
- Integration of the reflections intensity
- Parameters refinement (sample, instrument)

- It provides a graphical user interface that allows users:

- Run crystallographic calculations
- Visualize and interact with the data

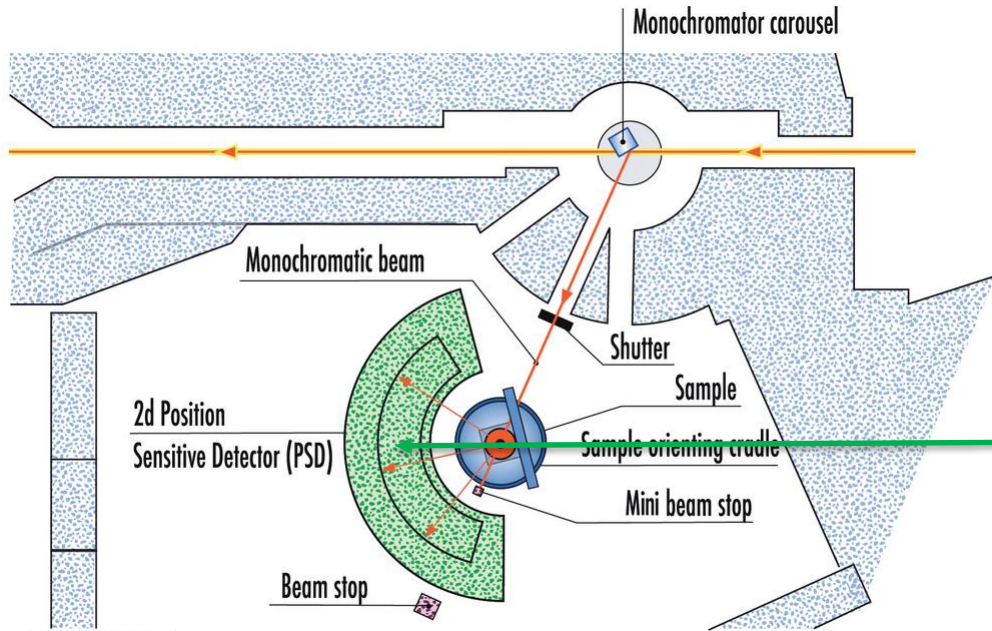
Int3D: languages and libraries



Int3D: current status

Instrument	Status
D9	Ongoing, ready for next ILL cycle, January 2021
D10	Not started yet, but straightforward extension from D9
D19	Fully developed, ready to use

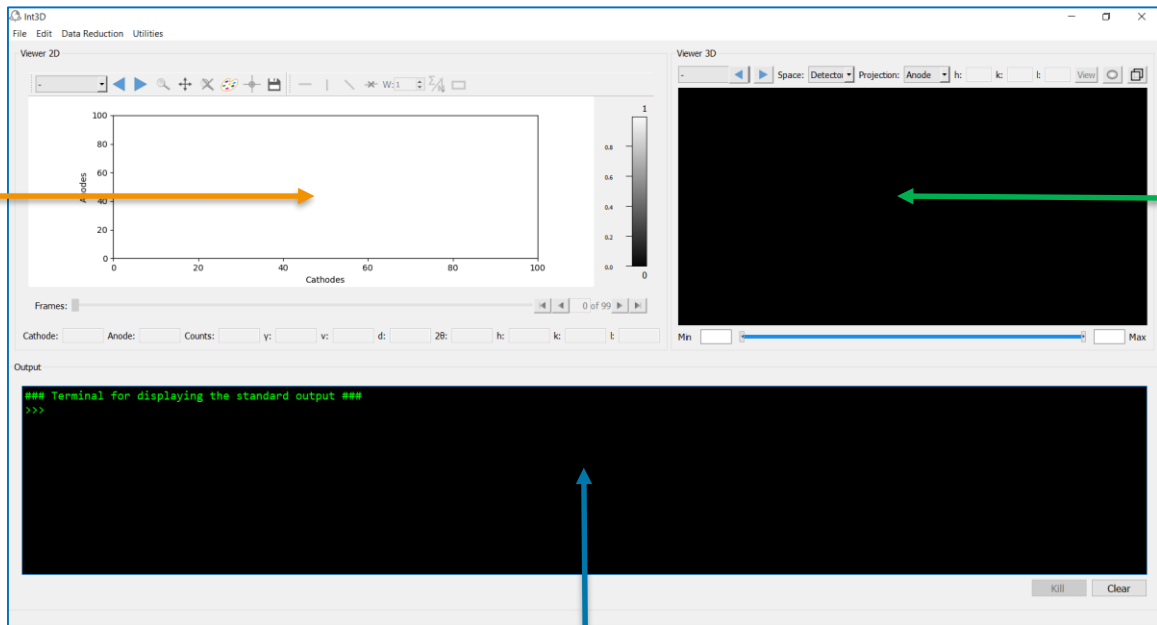
D19 single crystal diffractometer



Very large “banana” position sensitive detector ($120^\circ \times 30^\circ$)

©2016 ILL, A.Filhol & S.Crozel

Int3D: the GUI



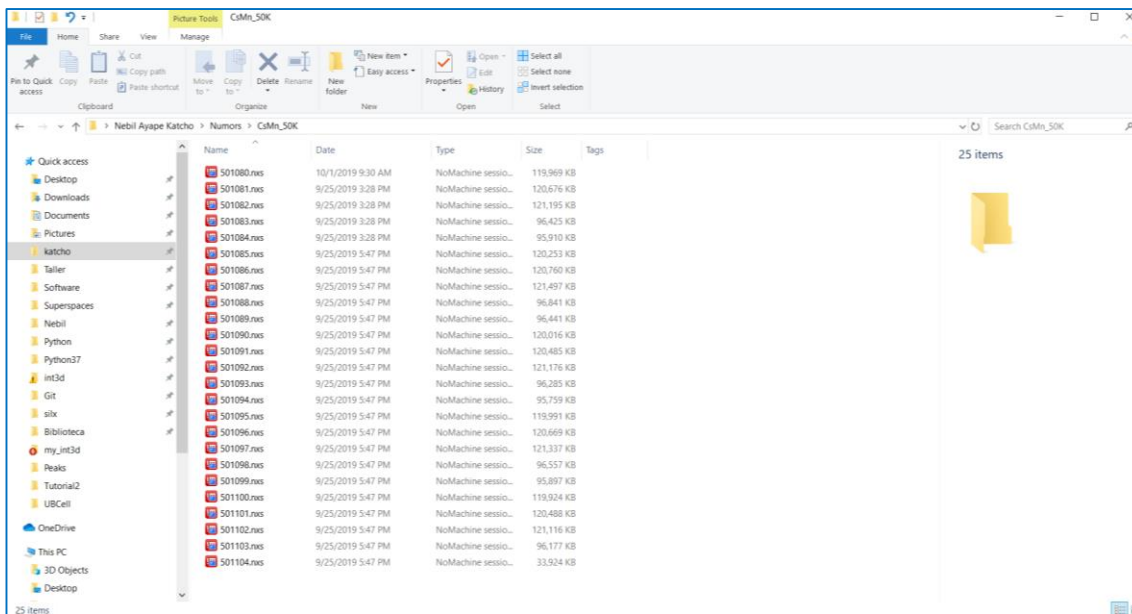
Scan 2D viewer
Frame by frame
visualization

Scan 3D viewer
Detector / reciprocal spaces

Terminal
Output of crystallographic calculations

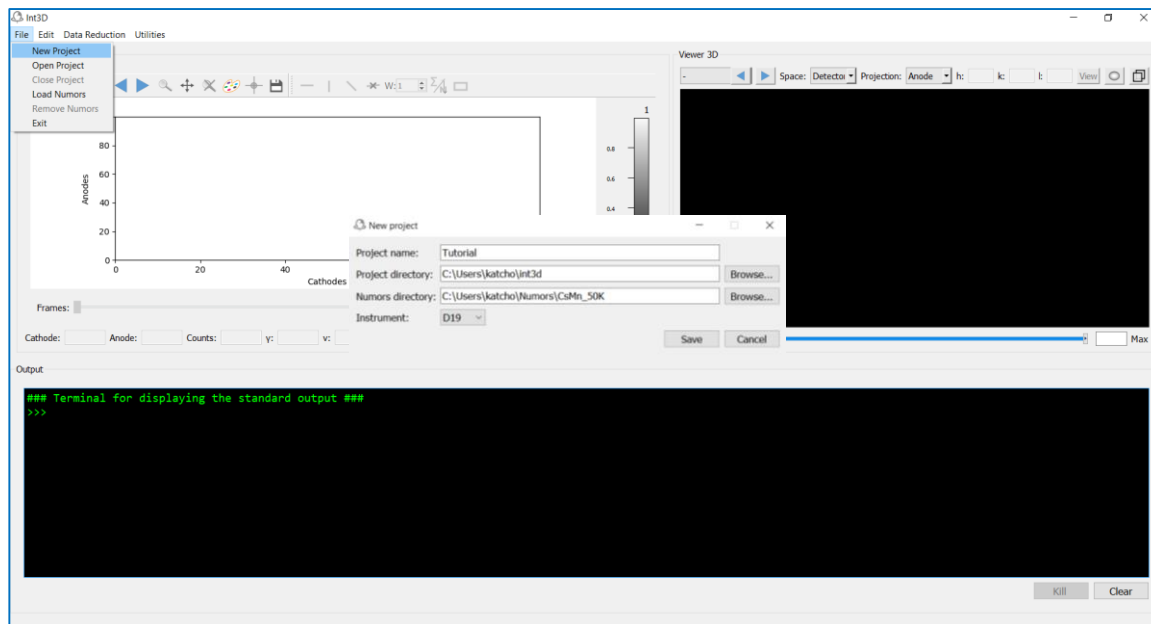
Int3D: the raw data

- The raw data for Int3D consists of a series of nexus files, called numors.
- Every numor corresponds to a given scan, usually an omega scan.



Data reduction step by step

1. Create a project and configure the instrument



Data reduction step by step

1. Create a project and configure the instrument

Viewer 2D

Viewer 3D

File Edit Data Reduction Utilities

Instrument

Name: D19

Geometry: Normal beam Busing-Levy frame: z-up Wavelength (Å): 1.4551

Detector

Type: H-Banana Sample-detector distance (mm): 764.5600 Gamma center (°): 65.0000 Nu center (°): 0.0000

Dimensions (mm)

Horizontal / vertical size: 1600.0000 / 400.0000

Number of cathodes / anodes: 640 / 256

Cathode / anode gap: 2.5000 / 1.5625

Angle limits (°)

Gamma+ minimum / maximum: 0.0000 / 130.0000

Gamma- minimum / maximum: -10.0000 / 0.0000

Nu minimum / maximum: -15.0000 / 15.0000

Offsets (mm)

x: 0.0000 y: 0.0000 z: 0.0000

Scan

Angle	Min (°)	Max (°)	Offset (°)	Velocity (° / s)
Gamma	0.0000	130.0000	0.0000	0.0000
Omega	-33.0000	49.0000	0.0000	1.0000
Nu	-15.0000	15.0000	0.0000	0.0000
2 Theta	0.0000	180.0000	0.0000	0.0000
Chi	80.0000	180.0000	0.0000	1.0000
Phi	-179.9000	180.0000	0.0000	1.0000

Instrument file: C:\Users\katcho\int3d\d19.geom

Default values

Save Import Cancel

Output

```
### Terminal for displaying the standard output ###
>>> Project Tutorial created.
>>>
```

Project structure

File Home Share View

File Explorer

Clipboard

Organize

New

Open

Select

Name

Date modified

Type

SI

Quick access

Desktop

Downloads

Documents

Pictures

Taller

Software

Superspaces

HKLGen

Integration

Peaks

Refinement

UBCell

katcho

d19.geom

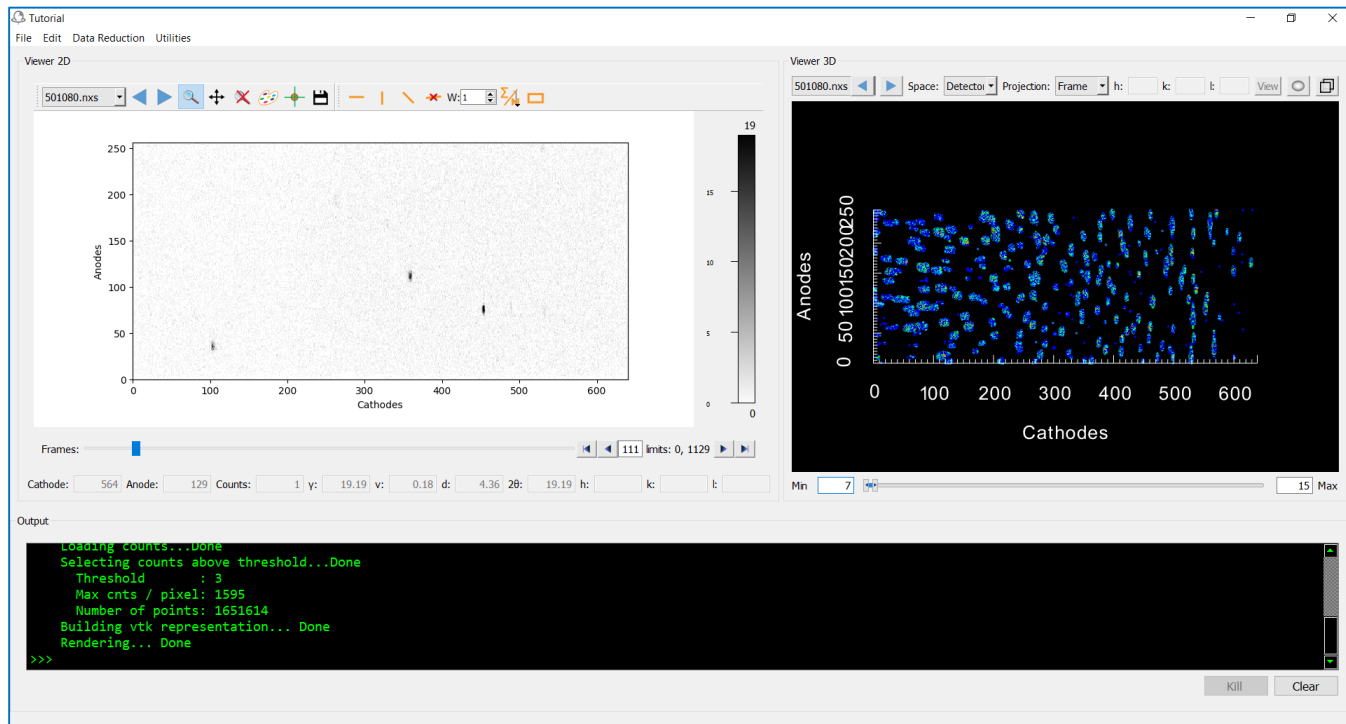
Tutorial.pro

Project file

Instrument file

Data reduction step by step

Appearance of D19 data

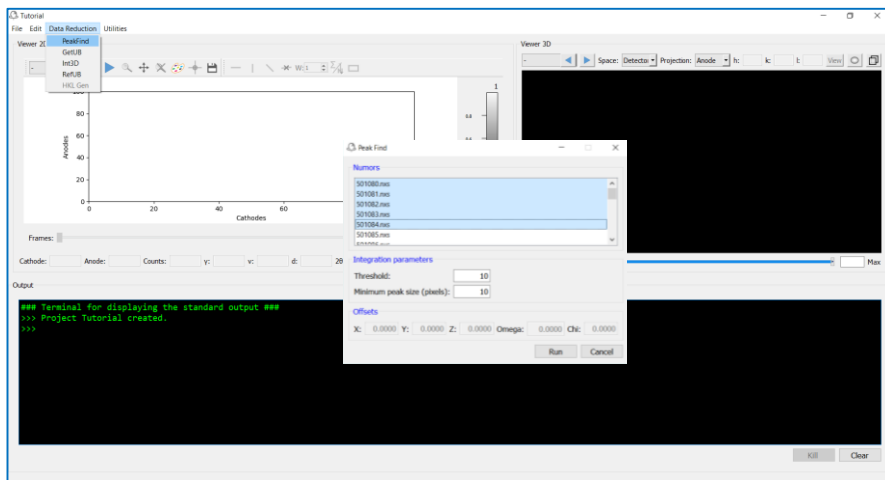


Data reduction step by step

2. Peaks search

From Data Reduction menu, open the Peakfind program

Input



The screenshot shows the Peakfind software interface. The main window displays a plot of Anodes vs Cathodes. A 'Peak Find' dialog box is open, showing a list of files to be processed and integration parameters.

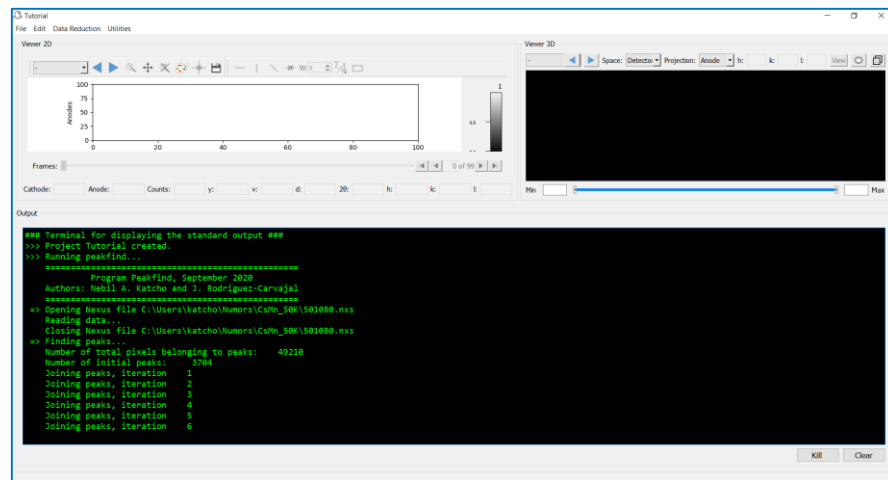
Integration parameters:

- Threshold: 10
- Minimum peak size (pixels): 10

Output:

```
### Terminal for displaying the standard output ###  
>>> Project Tutorial created.  
>>>
```

Output



The screenshot shows the Peakfind software interface. The main window displays a plot of Anodes vs Cathodes. The output window shows the results of the peak finding process, including the number of initial peaks and the number of pixels belonging to peaks.

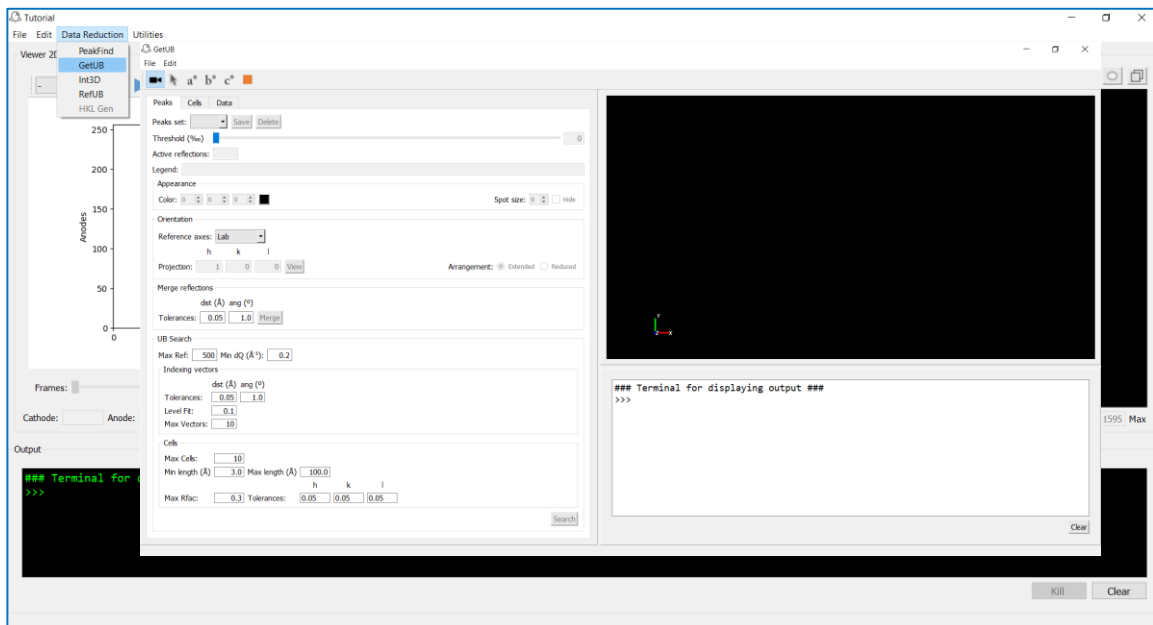
Output:

```
### Terminal for displaying the standard output ###  
>>> Project Tutorial created.  
>>> Running peakfind...  
#####  
Authors: Hehli A. Katcho and J. Rodriguez-Carvajal  
#####  
>> Opening Nexus file C:\Users\katcho\Numors\CDM_50K\501000.nxs  
Reading data...  
Closing Nexus file C:\Users\katcho\Numors\CDM_50K\501000.nxs  
>> Finding peaks...  
Number of total pixels belonging to peaks: 49210  
Number of initial peaks: 3784  
Joining peaks, iteration 1  
Joining peaks, iteration 2  
Joining peaks, iteration 3  
Joining peaks, iteration 4  
Joining peaks, iteration 5  
Joining peaks, iteration 6
```

Data reduction step by step

2. Determine the orientation matrix

From Data Reduction menu, open the GetUB application



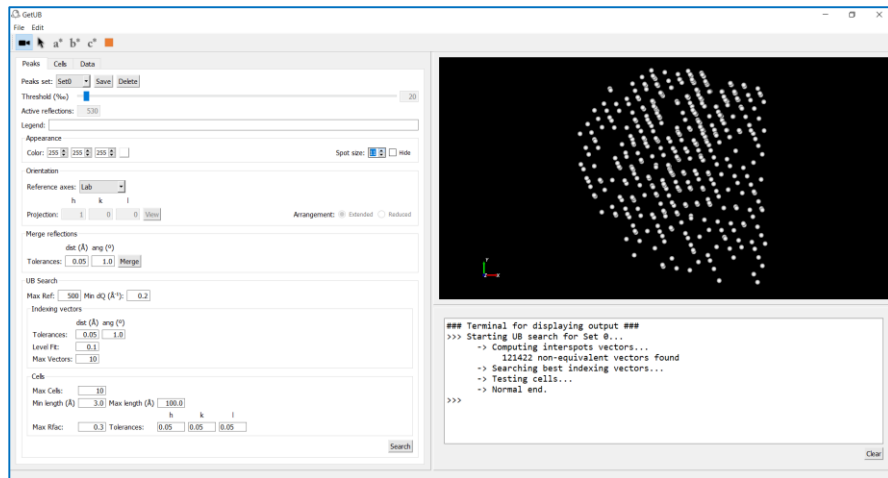
GetUB

- Determination of the orientation matrix
- Visualization of reciprocal space
- Orientation
- Interaction with the data:
 - Classification
 - Filtering
 - Deletion

Data reduction step by step

2. Determine the orientation matrix

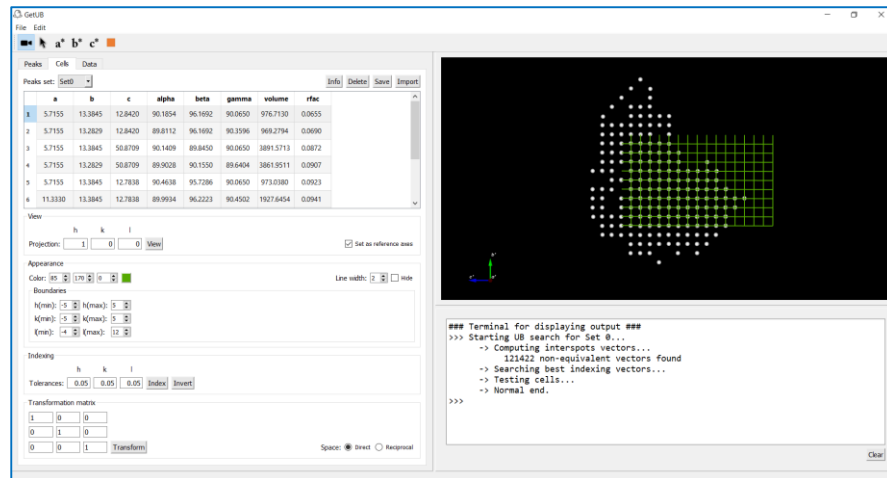
Load peaks found by peakfind and search the UB matrix



Terminal output:

```
### Terminal for displaying output ###
>>> Starting UB search for Set 0...
-> Computing interspots vectors...
    121422 non-equivalent vectors found
-> Searching best indexing vectors...
-> Testing cells...
-> Normal end.
>>>
```

Check proposed cells



	h	k	l	alpha	beta	gamma	volume	rfac
1	5.7155	13.3845	12.8420	90.1854	96.1692	90.0650	976.7130	0.9555
2	5.7155	13.2829	12.8420	89.8112	96.1692	90.3396	969.2794	0.9690
3	5.7155	13.3845	50.8709	90.1409	89.8450	90.0650	38915.713	0.0872
4	5.7155	13.2829	50.8709	89.9029	90.1550	89.6404	3861.9511	0.0907
5	5.7155	13.3845	12.7838	90.4638	95.7286	90.0650	973.0380	0.0923
6	11.3330	13.3845	12.7838	89.9934	96.2223	90.4502	1927.6454	0.0941

Terminal output:

```
### Terminal for displaying output ###
>>> Starting UB search for Set 0...
-> Computing interspots vectors...
    121422 non-equivalent vectors found
-> Searching best indexing vectors...
-> Testing cells...
-> Normal end.
>>>
```

Data reduction step by step

Load the UB matrix

The screenshot shows the 'Tutorial' software interface. The 'Crystal' dialog box is open, displaying the following data:

Cell parameters

a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)
5.7155	13.3845	12.8420	90.1854	96.1692	90.0650

Orientation matrix

Number of domains: 1

Domain: 1

-0.10457012	0.02466105	-0.06200130
0.05582945	-0.05533347	-0.04359465
-0.13006788	-0.04372825	0.01974602

Propagation vectors

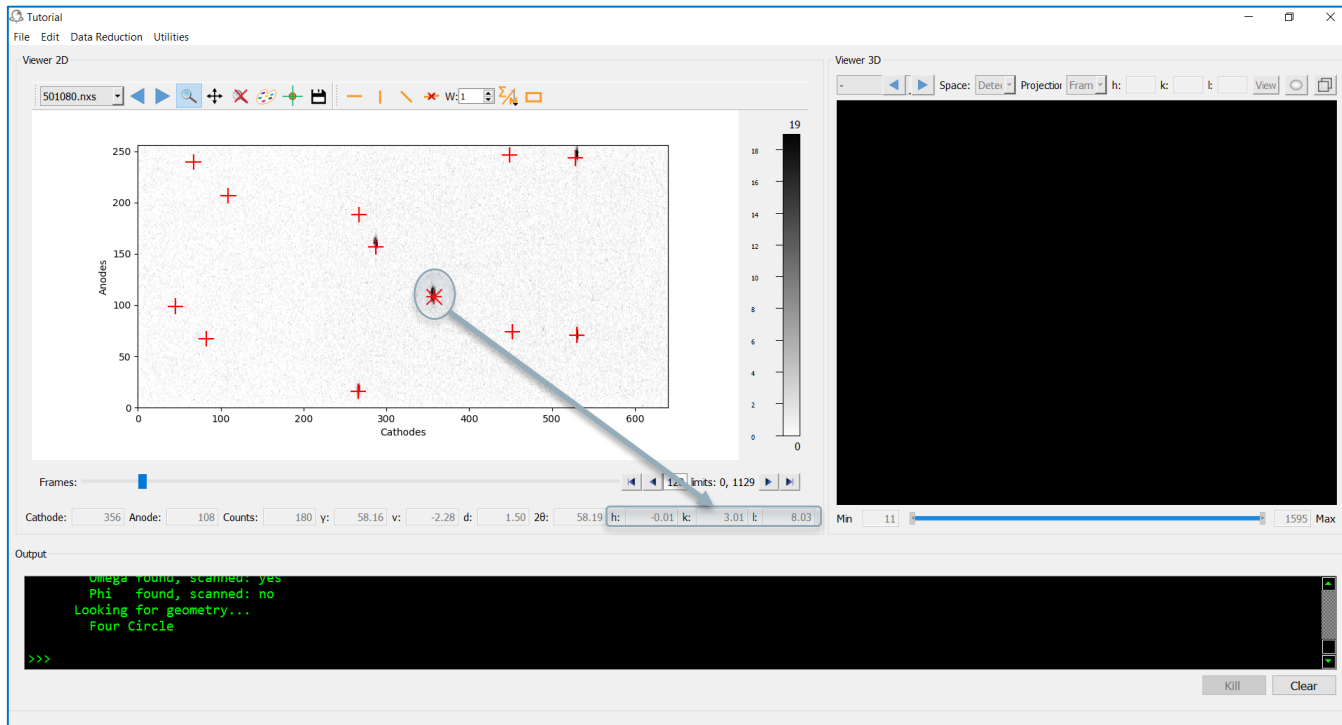
qx	qy	qz	q

Crystal file: C:\Users\katcho\OneDrive\Tutorial\Tutorial.cry

Output terminal: `### Terminal for displaying the standard output ###`
`>>>`

Data reduction step by step

Get the data indexed



Data reduction step by step

3. Integration

From Data Reduction menu, open Int3D

Input

Output

The screenshot shows the Int3D software interface. On the left, there is a plot of Anodes vs Cathodes. The main window displays the following parameters:

Names

501097.ref
501098.ref
501099.ref
501100.ref
501101.ref
501102.ref
501103.ref
501104.ref

Cell and UB-matrix

Space group: **1: P 1**

a (Å)	b (Å)	c (Å)	α (°)	β (°)	γ (°)
5.715520	13.384532	12.842012	90.185439	96.169180	90.064967

UB matrix:

-0.104570	0.024661	-0.062001
0.055829	-0.055333	-0.043595
-0.130068	-0.043728	0.019746

Integration box

nanodes	ncathodes	min anode	max anode	min cathode	max cathode
48	64	1	254	1	639

Integration parameters

lmax	psigxp	psigyp	dval1	dval2	dval3	xmonst	xangst
50	1.0000	5.0000	1000	1000	1000	10000.0	0.07

The screenshot shows a file explorer window displaying the output files generated by the integration process. The files are listed in a table:

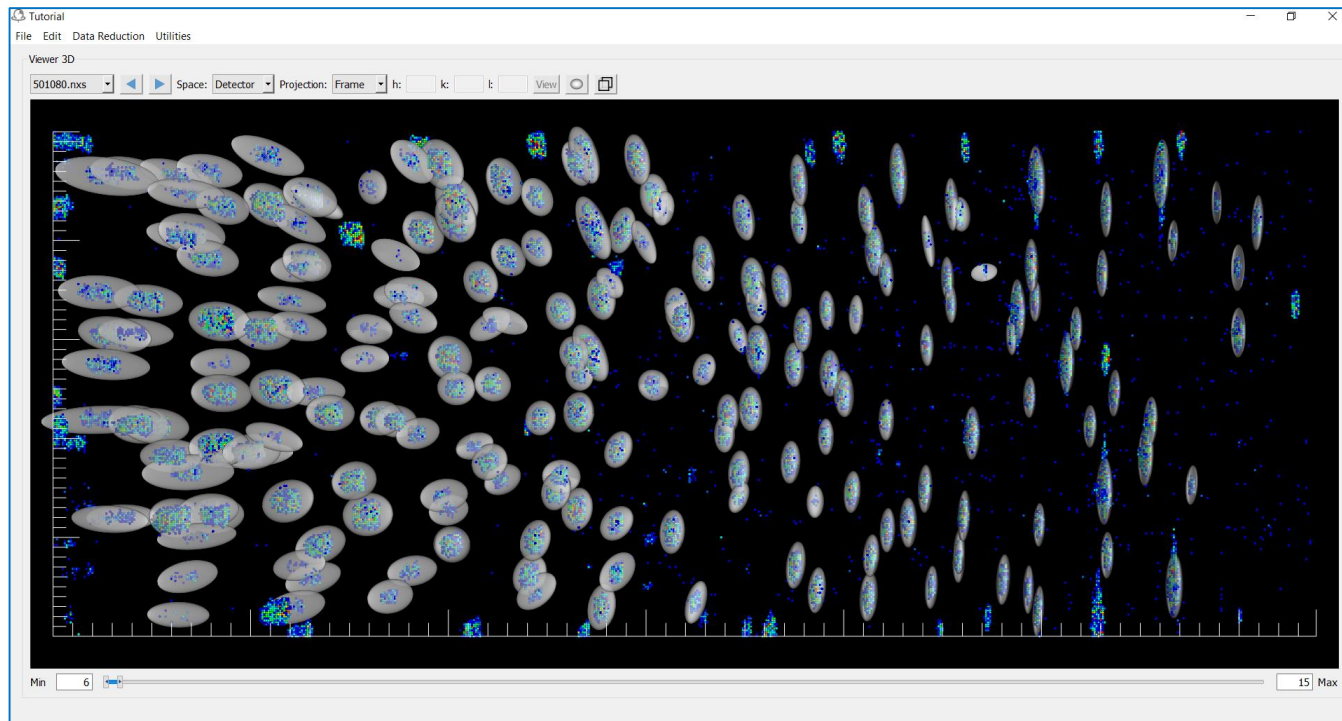
Name	Date modified	Type	Size
501100.lpt	10/16/2020 10:57	LPT File	2,723 KB
501100.ref	10/16/2020 10:57	REF File	26 KB
501100.sel	10/16/2020 10:57	SEL File	37 KB
501101.lpt	10/16/2020 10:57	LPT File	2,727 KB
501101.ref	10/16/2020 10:57	REF File	26 KB
501101.sel	10/16/2020 10:57	SEL File	32 KB
501102.lpt	10/16/2020 10:57	LPT File	2,763 KB
501102.ref	10/16/2020 10:57	REF File	26 KB
501102.sel	10/16/2020 10:57	SEL File	33 KB
501103.lpt	10/16/2020 10:58	LPT File	2,187 KB
501103.ref	10/16/2020 10:58	REF File	21 KB
501103.sel	10/16/2020 10:58	SEL File	24 KB
501104.lpt	10/16/2020 10:58	LPT File	754 KB
501104.ref	10/16/2020 10:58	REF File	8 KB
501104.sel	10/16/2020 10:58	SEL File	8 KB
Tutorial.dara	10/16/2020 10:58	Text File	3,510 KB
Tutorial.cif	10/16/2020 10:58	CRY File	4 KB
Tutorial.hkl	10/16/2020 10:58	HKL File	233 KB
Tutorial.lib	10/16/2020 10:58	Object File Library	348 KB
Tutorial.log	10/16/2020 10:58	Text Document	13 KB
Tutorial.ref	10/16/2020 10:58	RAF File	443 KB
Tutorial.ref.dara	10/16/2020 10:58	RAF File	2,074 KB
Tutorial.ref.hkl	10/16/2020 10:58	HKL File	248 KB
Tutorial_ref.dara	10/16/2020 10:58	RAF File	164 KB
Tutorial_ref.hkl	10/16/2020 10:58	HKL File	11 KB
Tutorial_strong.xyz	10/16/2020 10:58	XYZ File	191 KB
Tutorial_weak.xyz	10/16/2020 10:58	XYZ File	91 KB

Files

- .lpt : integration details
- .ref : peak centroids
- .sel : integration ellipsoids
- .hkl : integrated intensities
- .lib : library for weak refl
- .raf : used for refinement

Data reduction step by step

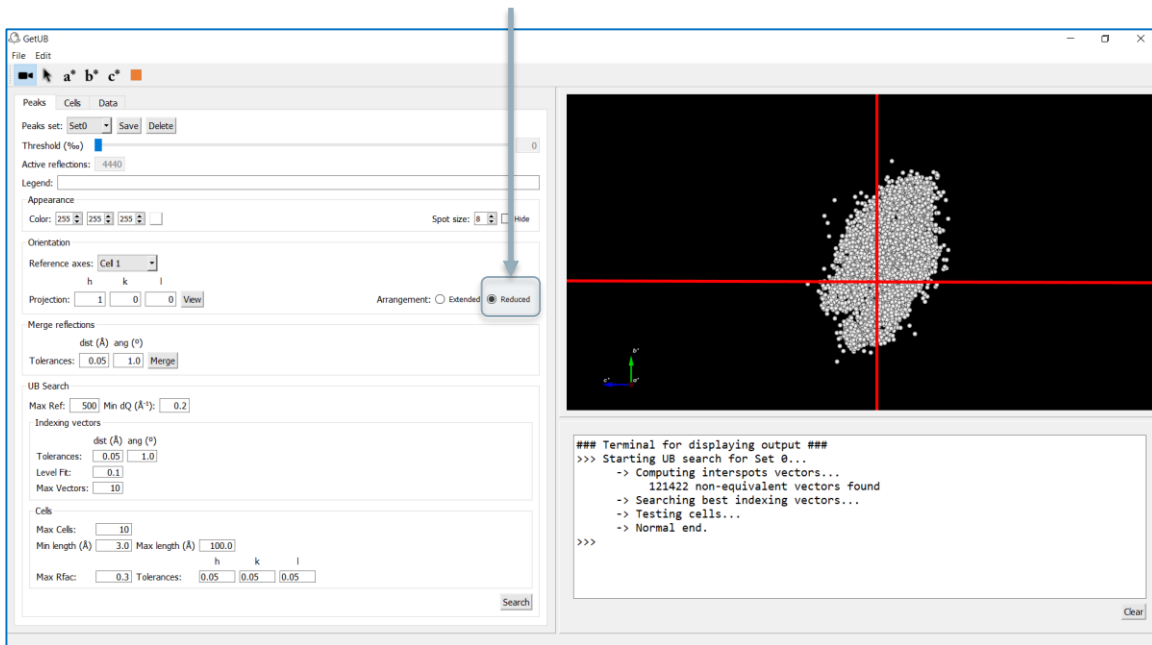
Check integration ellipsoids by visual inspection



Data reduction step by step

Check offsets

Use GetUB to translate all reflections to the origin



The screenshot shows the GetUB software interface. On the left, there are various control panels for 'Peaks', 'Orientation', 'Merge reflections', 'UB Search', and 'Cells'. A blue arrow points from the 'Reduced' radio button in the 'Orientation' section to the 'Reduced' button in the 'Arrangement' section. The main window displays a 2D plot of reflections (grey dots) on a black background, with red axes. The plot shows a distribution of points that is not perfectly centered. Below the plot is a terminal window with the following output:

```
### Terminal for displaying output ###
>>> Starting UB search for Set 0...
-> Computing interspots vectors...
    121422 non-equivalent vectors found
-> Searching best indexing vectors...
-> Testing cells...
-> Normal end.
>>>
```

Not well centred
Anisotropy

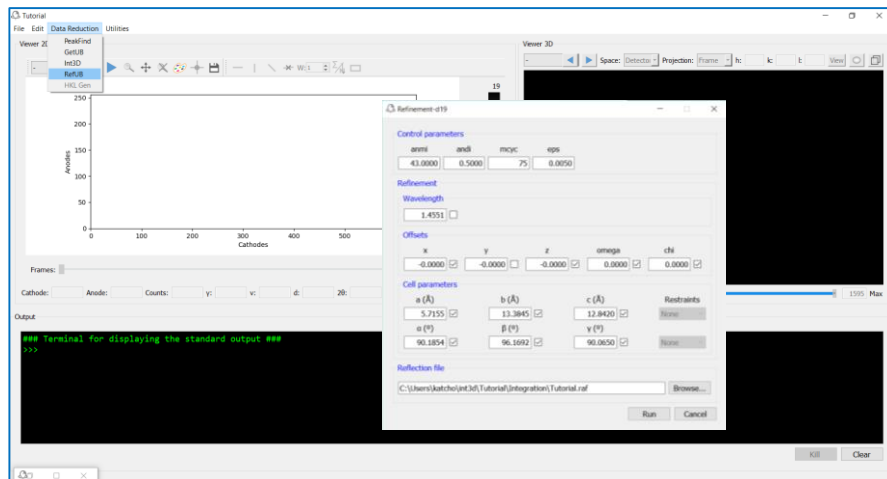
Refine!

Data reduction step by step

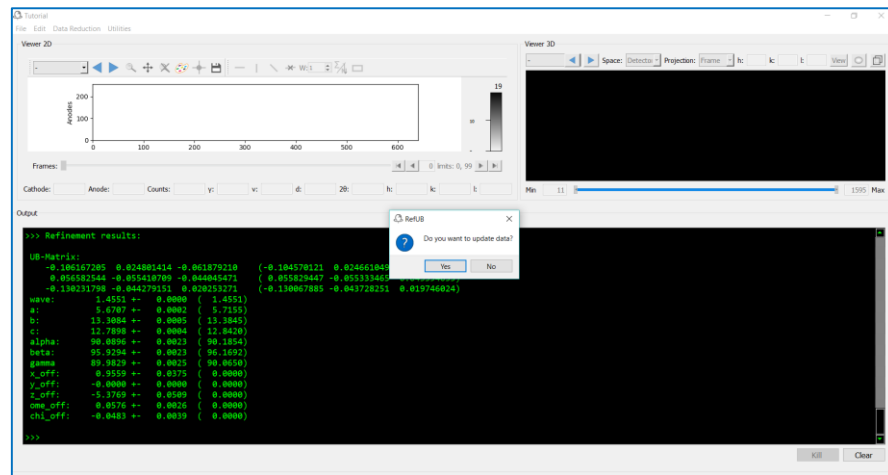
4. Refinement

From Data Reduction menu, open RefUB

Input

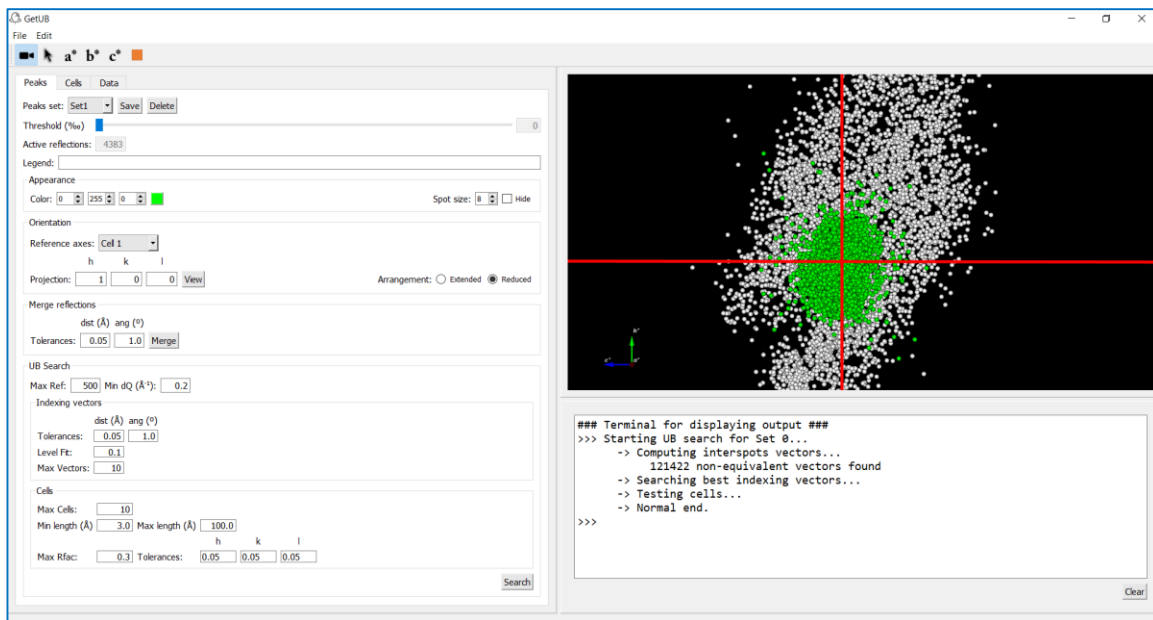


Output



Data reduction step by step

Check offsets



The screenshot shows the GetUB software interface. The main window displays a 2D plot of reflection spots, with a central cluster of spots highlighted in green. The plot is overlaid with a red crosshair. The interface includes a menu bar (File, Edit), a toolbar with icons for a* b* c*, and several panels for configuration and data display.

Peaks Cells Data

Peaks set: Set1 Save Delete

Threshold (%) 0

Active reflections: 4383

Legend:

Appearance

Color: 0 255 0 0 Spot size: 8 Hide

Orientation

Reference axes: Cell 1

h k l

Projection: 1 0 0 View Arrangement: Extended Reduced

Merge reflections

dst (Å) ang (°)

Tolerances: 0.05 1.0 Merge

UB Search

Max Ref: 500 Min dQ (Å⁻¹): 0.2

Indexing vectors

dst (Å) ang (°)

Tolerances: 0.05 1.0

Level Ff: 0.1

Max Vectors: 10

Cells

Max Cells: 10

Min length (Å) 3.0 Max length (Å) 100.0

h k l

Max Rfac: 0.3 Tolerances: 0.05 0.05 0.05

Search Clear

```
### Terminal for displaying output ###
>>> Starting UB search for Set 0...
-> Computing interspots vectors...
    121422 non-equivalent vectors found
-> Searching best indexing vectors...
-> Testing cells...
-> Normal end.
>>>
```

Well centred
Isotropy
Less dispersion

Summary and conclusions

- We have integrated in a single application all the required tasks for performing the data reduction in single crystal neutron diffraction experiments.
- Int3D provides powerful graphical tools and suggest reasonable parameters for the different steps of the data reduction process, which makes it accessible to users without demanding high expertise.
- The application is distributed as a single folder containing all required libraries and executables. No installation of libraries or python is required.
- Video tutorial: www.int3d.fr
- Future improvements: absorption correction, complete visualization of data in reciprocal space, users demands...



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