XRD data visualization, processing and analysis with *d1Dplot* and *d2Dplot* software packages

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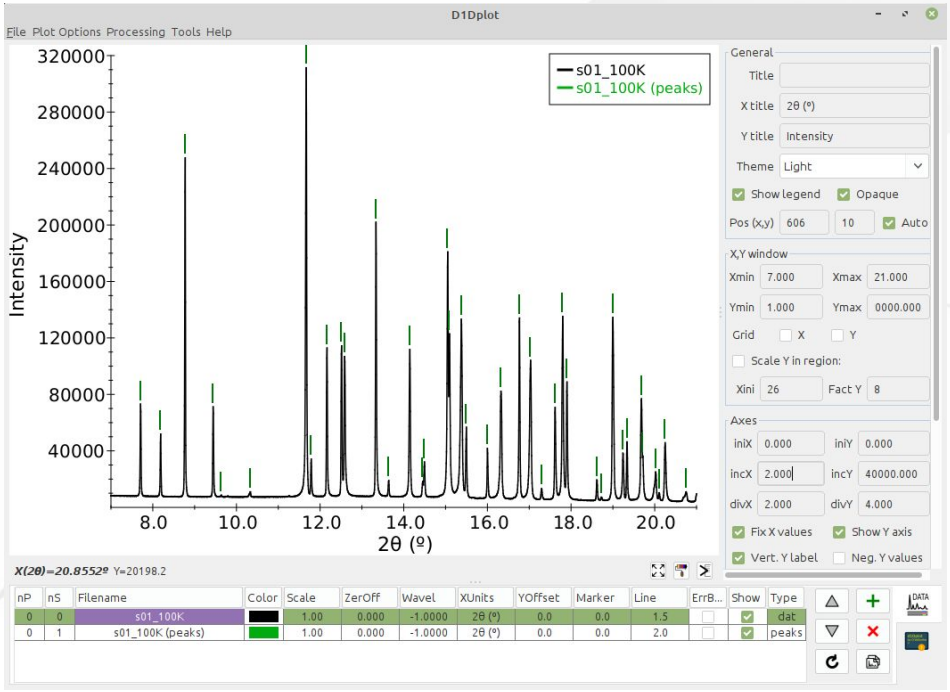
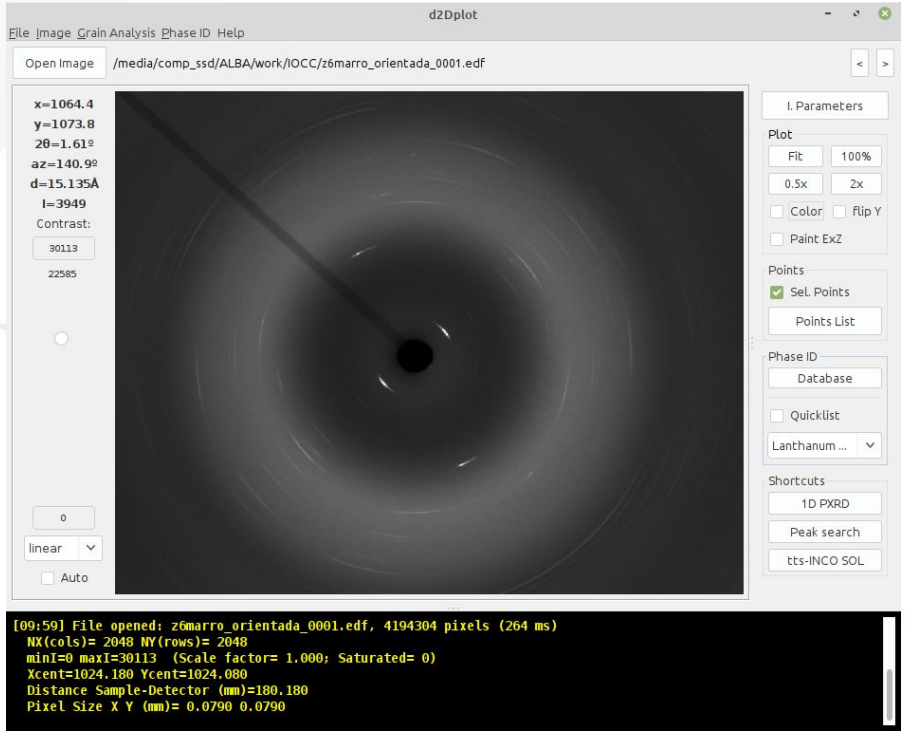
Session H. Software/Tools to Deal with Crystal and Crystallographic Issues & Teaching Crystallography

The 2nd International Online Conference on Crystals
10-20th November 2020

Introduction

- X-ray diffraction tools for 1D and 2D data
- Emphasis to the plotting capabilities, ease-of-use and preparation of figures.
- General processing capabilities + specific data analysis features such as tts-*μ*XRD methodology.





d2Dplot

[O.Vallcorba, J.Rius. *J. Appl. Crystallogr.* **2019**, 52, 478–484]

- **Visual Inspection** and **basic processing** of **2D X-ray diffraction data**
  - Mouse navigation, contrast, color, on-screen info, point selection,...
  - Sum/subtract frames, instrumental calibration, radial/azimuthal integration,...
  - Supported formats: EDF, IMG, GFRM (Bruker), SPR (text file), CBF (Pilatus), TIFF. Optimized for ALBA Synchrotron MSPD beamline

    [F.Fauth, I.Peral, C.Popescu, M.Knapp. *Powder Diffr.* **2013**, 28, S360-S370]

- **Grain analysis** (peaks) for **tts-*μ*XRD**
  - Through-the substrate microdiffraction methodology for the structural study of *μ*volumes of crystals embedded in polished thin sections of compact materials

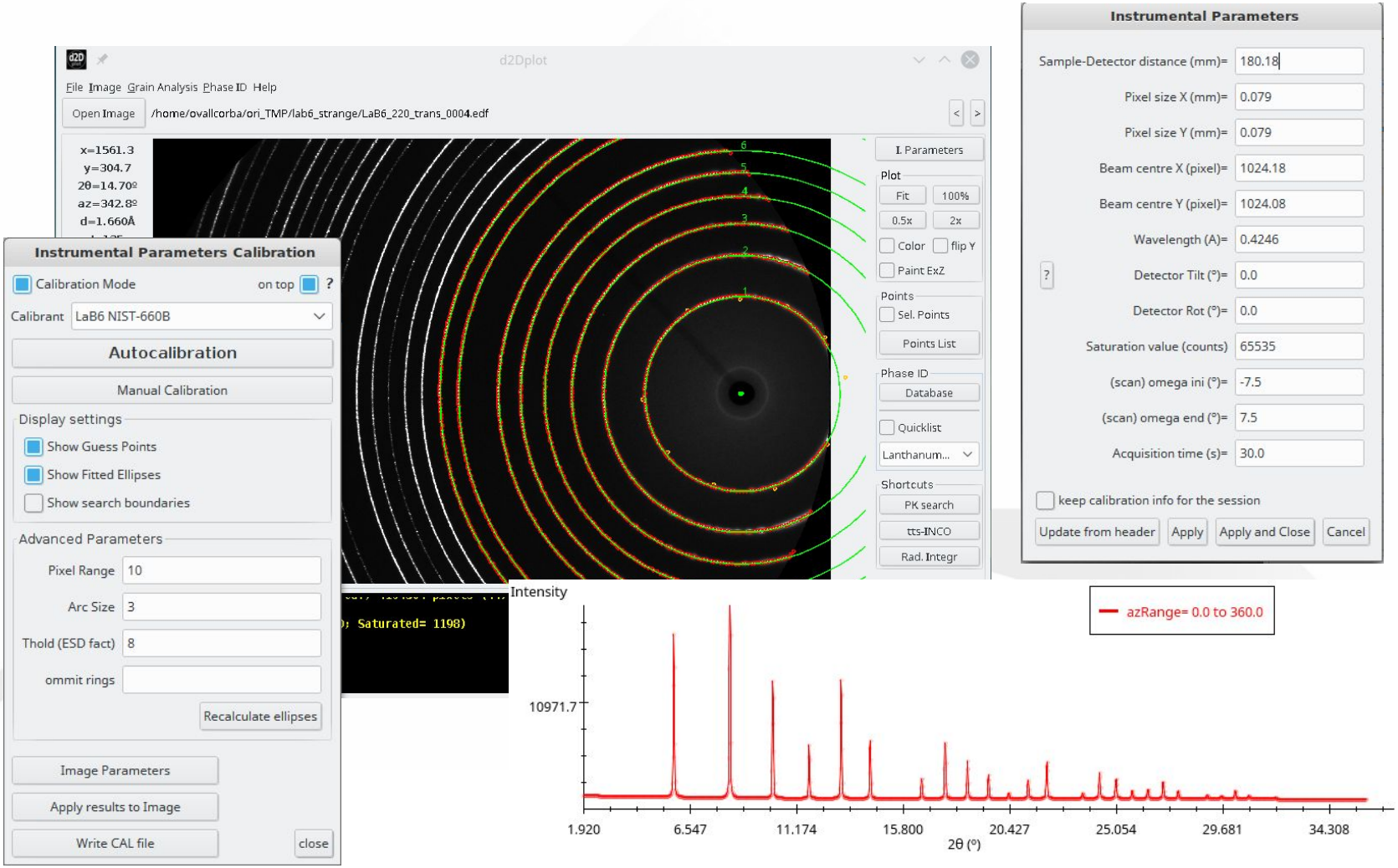
    [J.Rius, O.Vallcorba, C.Frontera, I.Peral, A.Cresi & C.Miravitlles. *IUCrJ* **2015**, 2, 452-463]
  - Peak search and integration. Check orientation results.

- **Compound Database** for easy phase identification on the images
  - Plot expected reflection positions, generate reflections, search-match, ...

d2Dplot: General data processing

Instrumental calibration (from LaB_6, Si, etc… diffraction data)

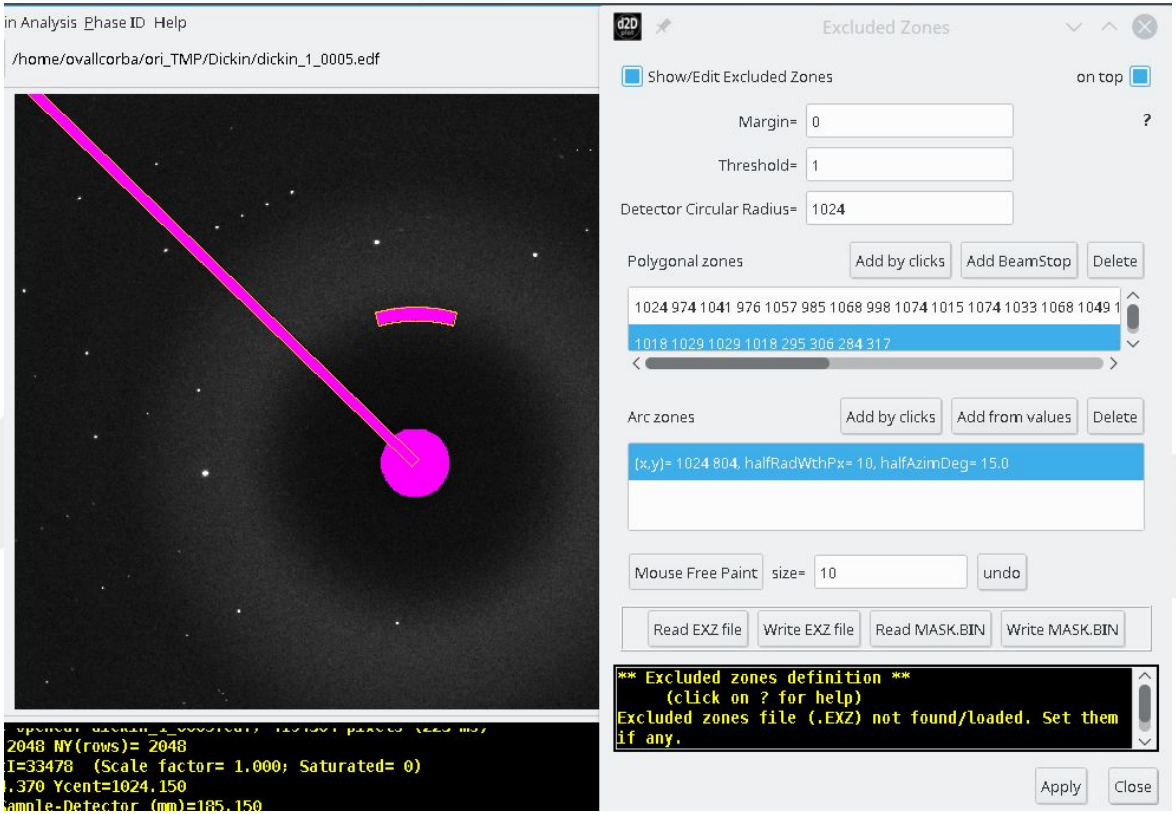
- Sample-to-detector distance, beam center and orthogonality of the detector.



d2Dplot: General data processing

Excluded Zones

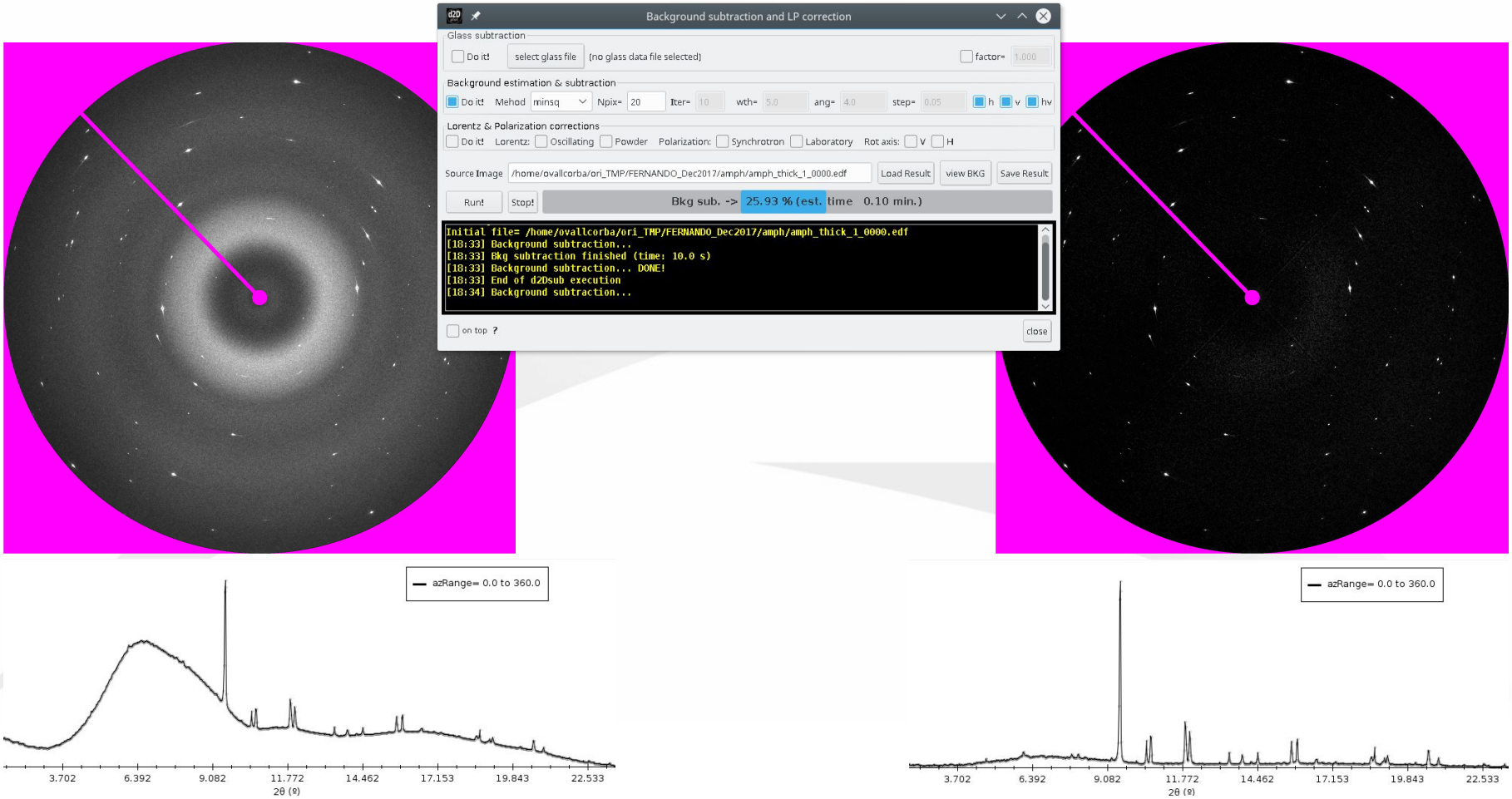
- Pixels to be omitted in further calculations
- Margin, Intensity threshold, Detector Radius, Geometrical shapes, BS, Arcs, Paint



d2Dplot: General data processing

Background subtraction

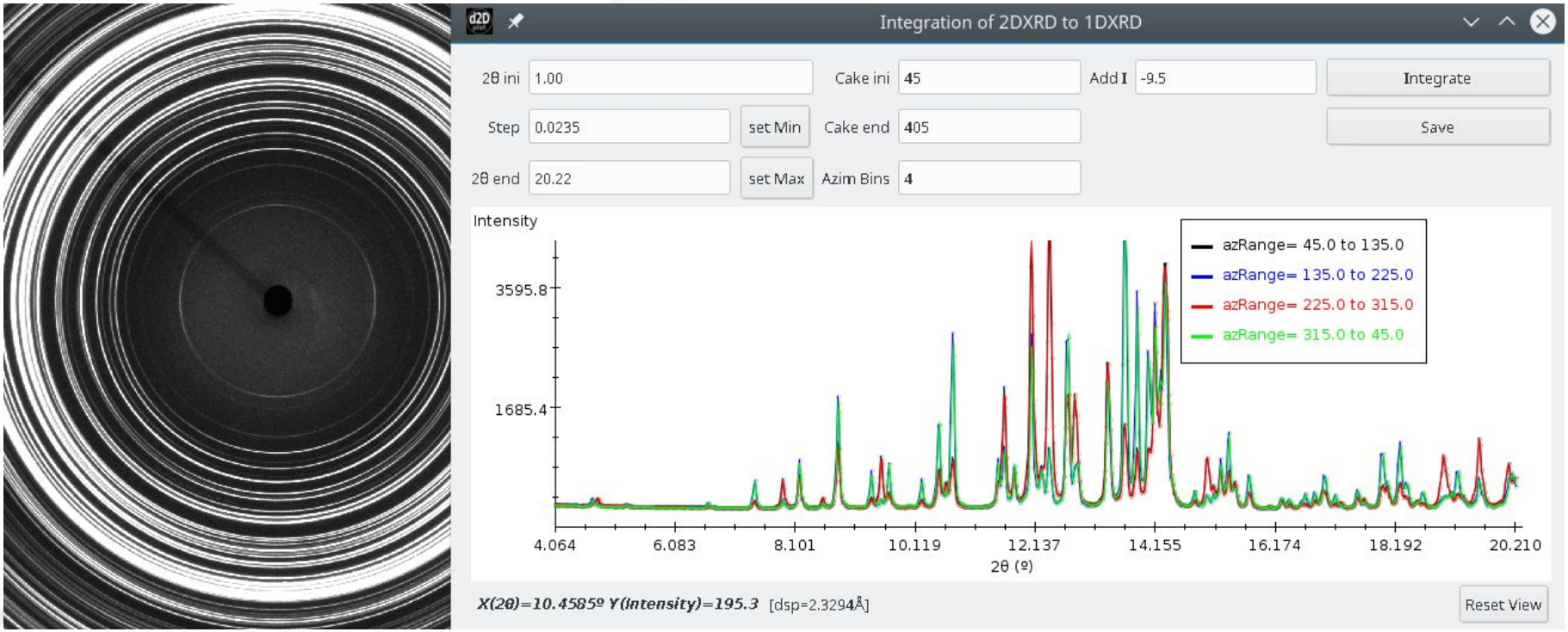
- Remove the contribution of a holder. May be directly subtracted or estimated.



d2Dplot: General data processing

Conversion to 1D-XRD

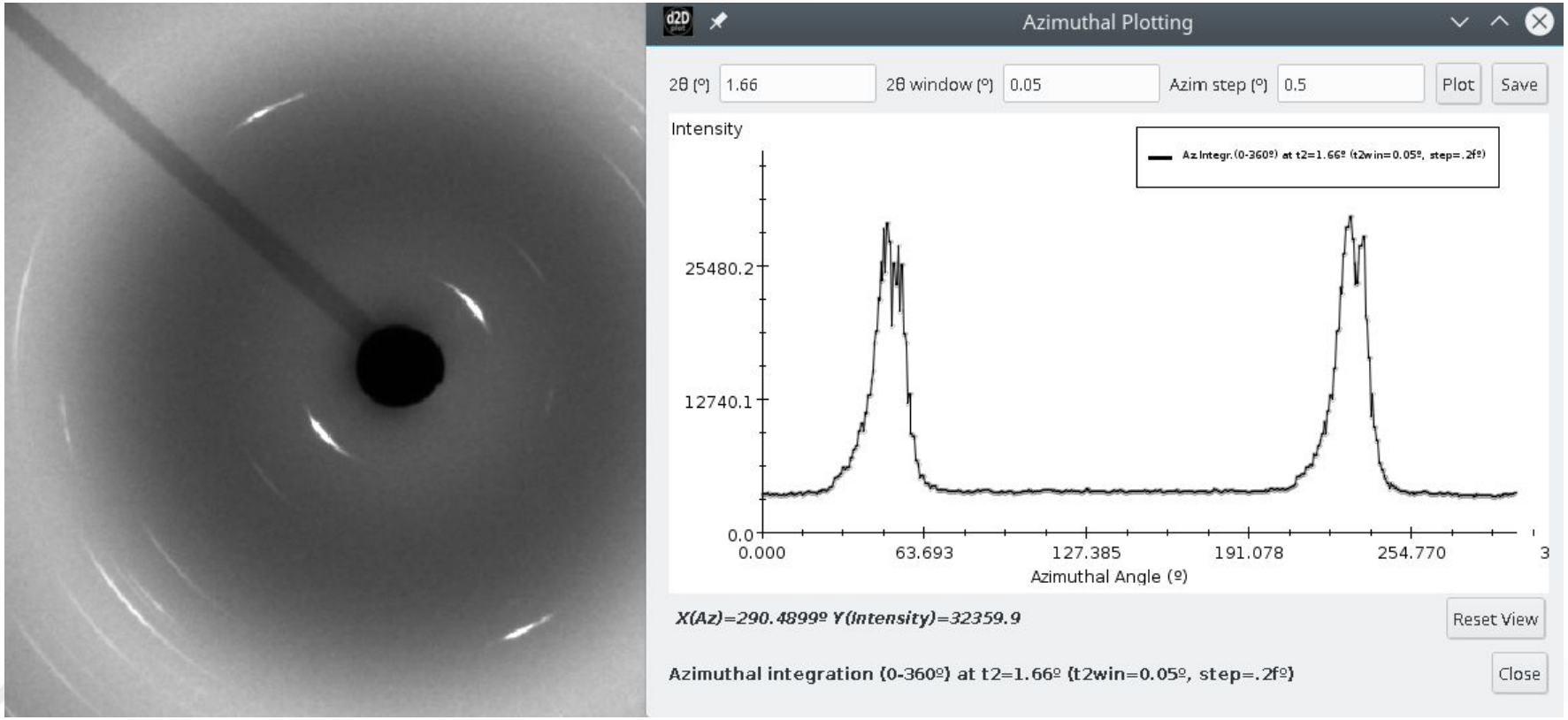
- Generate 1D diffraction pattern by populating an histogram of 2θ intervals
- Azimuthal bins (“cakes”)



d2Dplot: General data processing

Azimuthal (circular) plot

- Integration along the ellipse specified by a 2θ value and tolerance



d2Dplot: tts-*μ*XRD

Journal of Synchrotron Radiation
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Capabilities of through-the-substrate micro-diffraction: application of Patterson-function direct methods to synchrotron data from polished thin sections

Jordi Rius,[a]* Ana Labrador,[b] Anna Crespi,[a] Carlos Frontera,[a] Oriol Vallcorba[a] and Joan Carles Melgarejo[c]

J. Synchrotron Rad. **2011**, 18, 891-898

IUCrJ
ISSN 2052-2525
MATERIALS | COMPUTATION

Application of synchrotron through-the-substrate microdiffraction to crystals in polished thin sections

Jordi Rius,[a]** Oriol Vallcorba,[b] Carlos Frontera,[a] Inmaculada Peral,[b] Anna Crespi[a] and Carles Miravitlles[a]

IUCrJ **2015**, 2, 452-463

DE GRUYTER Z. Kristallogr. 2017; aop

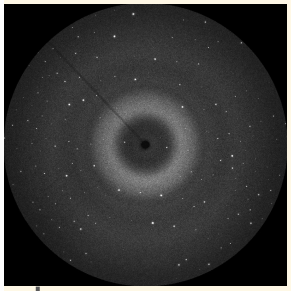
Jordi Rius*, Oriol Vallcorba, Anna Crespi and Fernando Colombo

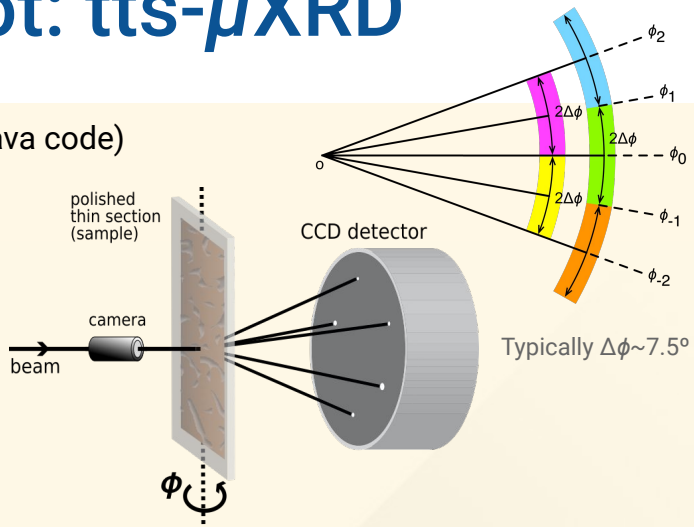
Increasing data completeness in synchrotron tts-microdiffraction experiments for δ-recycling phasing of low-symmetry compounds

Z. Kristallogr. **2017**, 232, 827-834

d2Dplot: tts-*μ*XRD

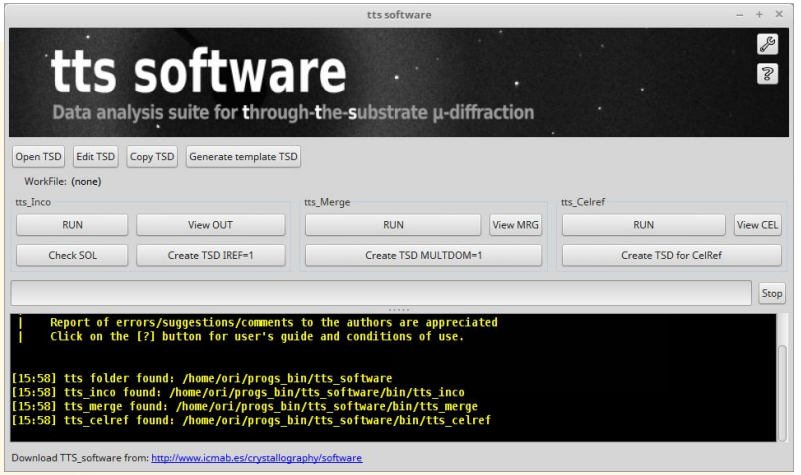
d2Dplot (Java code)





Typically $\Delta\phi$~7.5º

Diffraction images (*tts data collection strategy*)



Help prepare input files for *tts_software*, run and check results.

Create mask.bin file

Check intermediate outputs

Peak integration

List of diffraction peaks (PCS)

tts_inco (ioff=0)

Orientation (hkl) of central frame

tts_inco (ioff=1)

Orientation (hkl) of all frames of the microvolume

Merged HKL

tts_merge

tts_celref

Cell refinement (if necessary)

For all the measured microvolumes

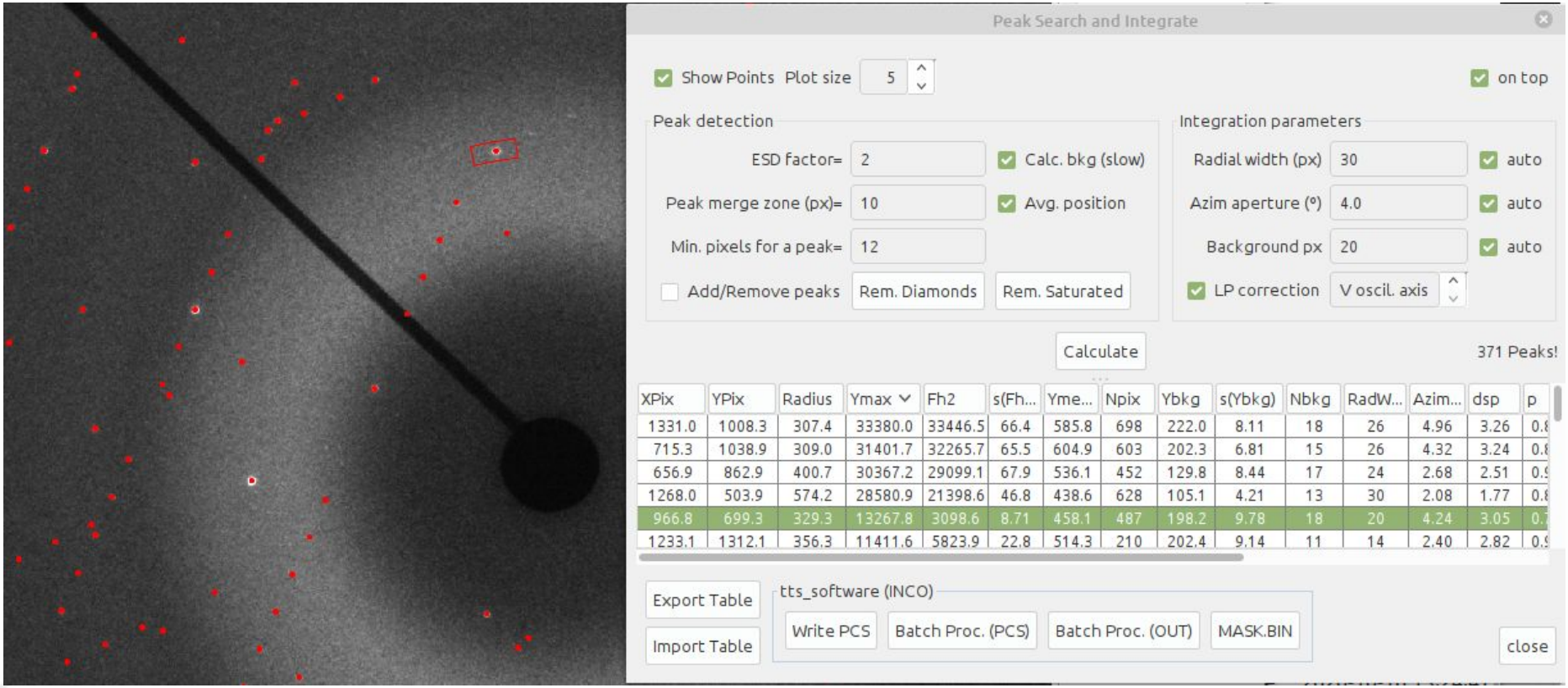
TTS software

(Fortran codes)

d2Dplot: tts-*μ*XRD

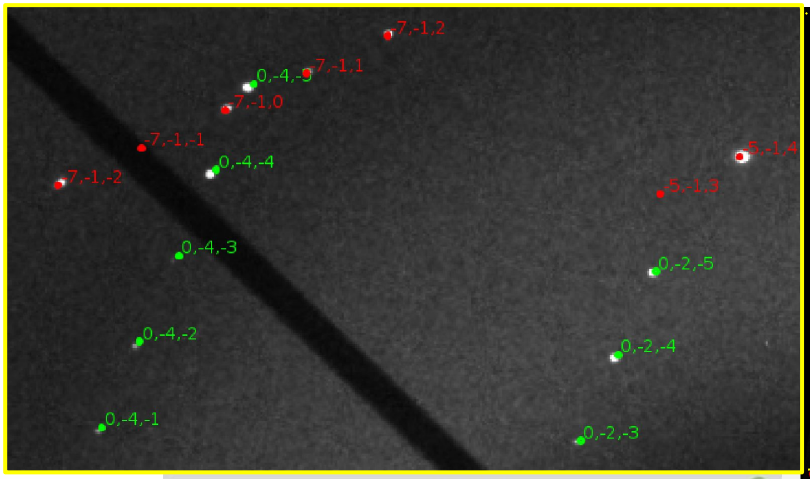
Peak search and integration

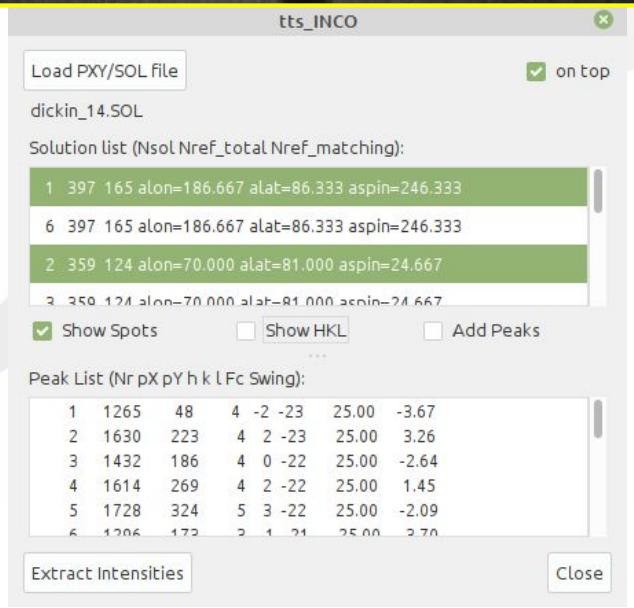
- Find diffraction peaks with options. Export for tts-INCO.

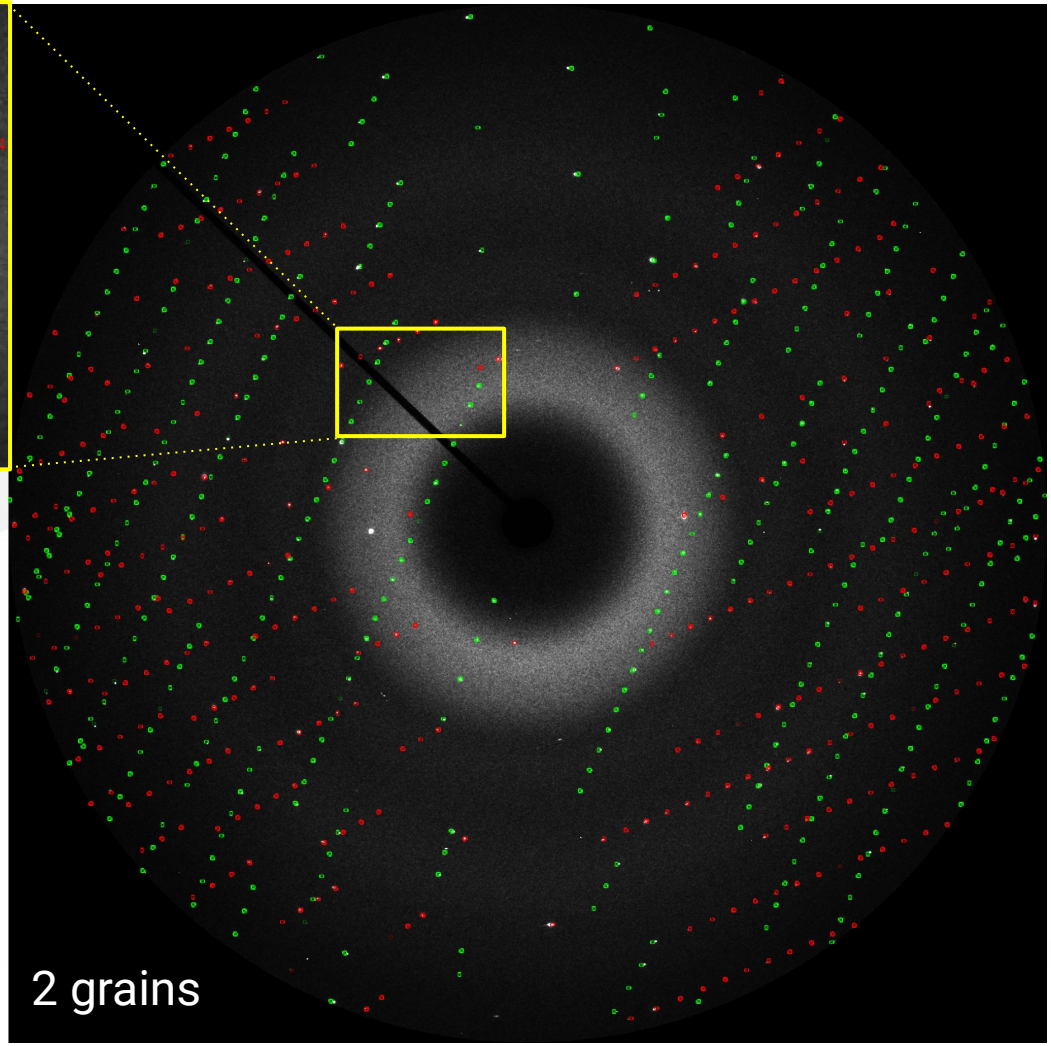


d2Dplot: tts-*μ*XRD

Select the correct grain orientation(s)





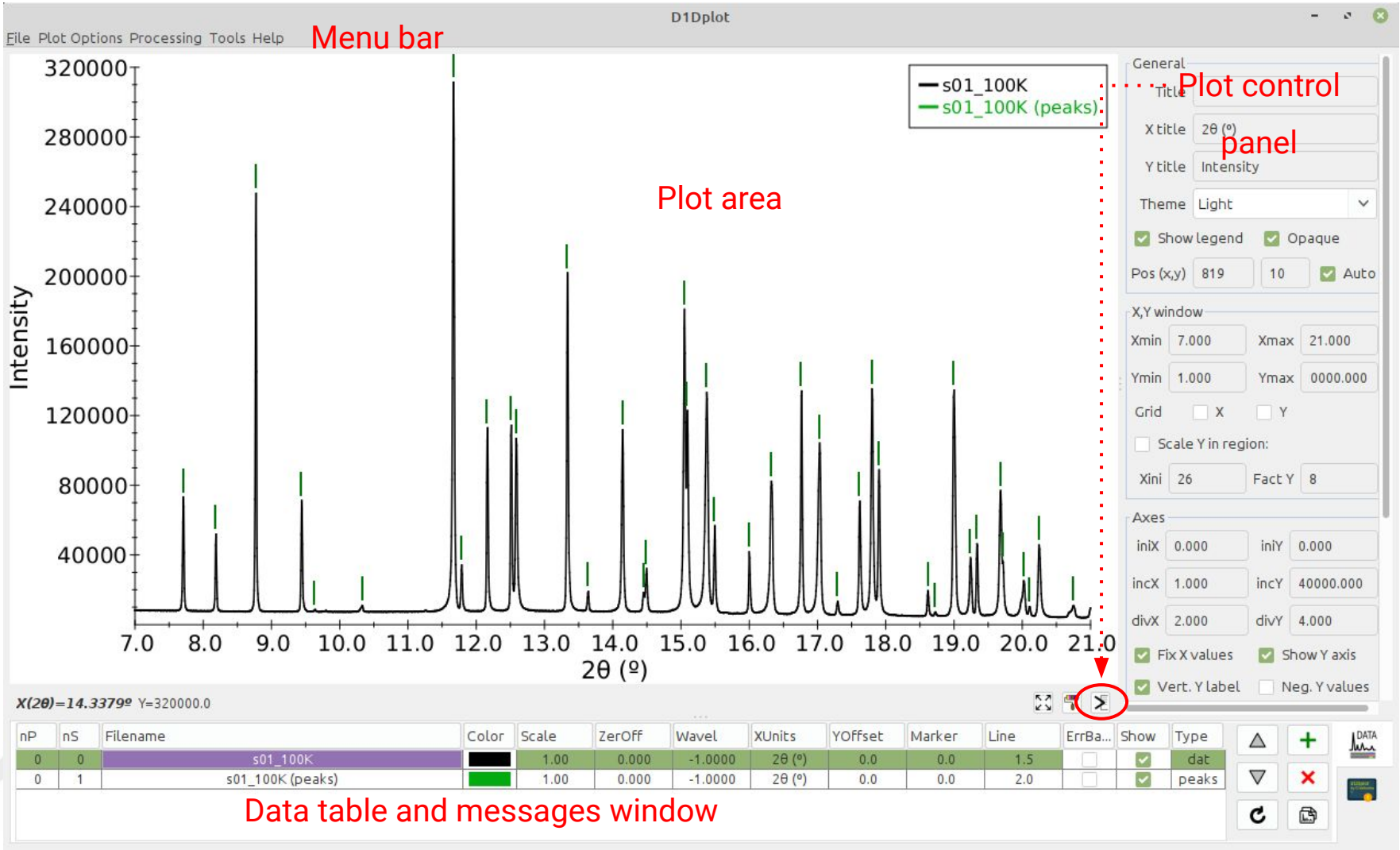


d1Dplot

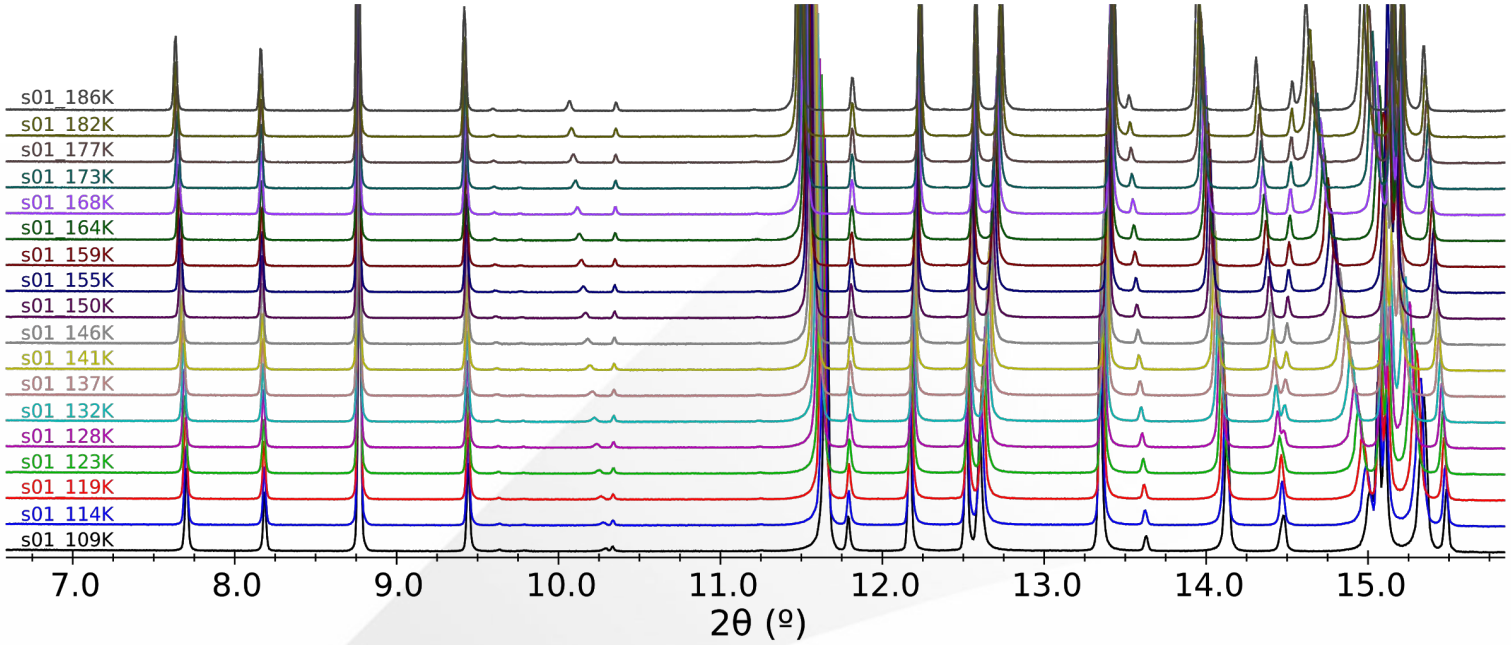
- **Visual Inspection** and **basic processing** of **1D X-ray diffraction data**
  - User-friendly interface with easy mouse navigation and on-screen info
  - Multi-pattern stacking, 2D plot
  - Total control of the aspect and functionality (axes, zones, zoom, mouse…)
  - Basic operations as rebinning, change X-units, background estimation, peak finding, sum/subtract data, fit peaks,...
  - Supported formats: 2 or 3 columns + headers (DAT, XYE, XY, ASC), GSAS, XRDML, FullProf profile (PRF), PdfGetX3 G(r), + *d1Dplot* own formats for projects, profile fitting and data.
  - Save figures as PNG or SVG vector graphics.

- **Compound Database** for easy phase identification on the images
  - Plot expected reflection positions, generate reflections, search-match, ...

d1Dplot: User interface and main window

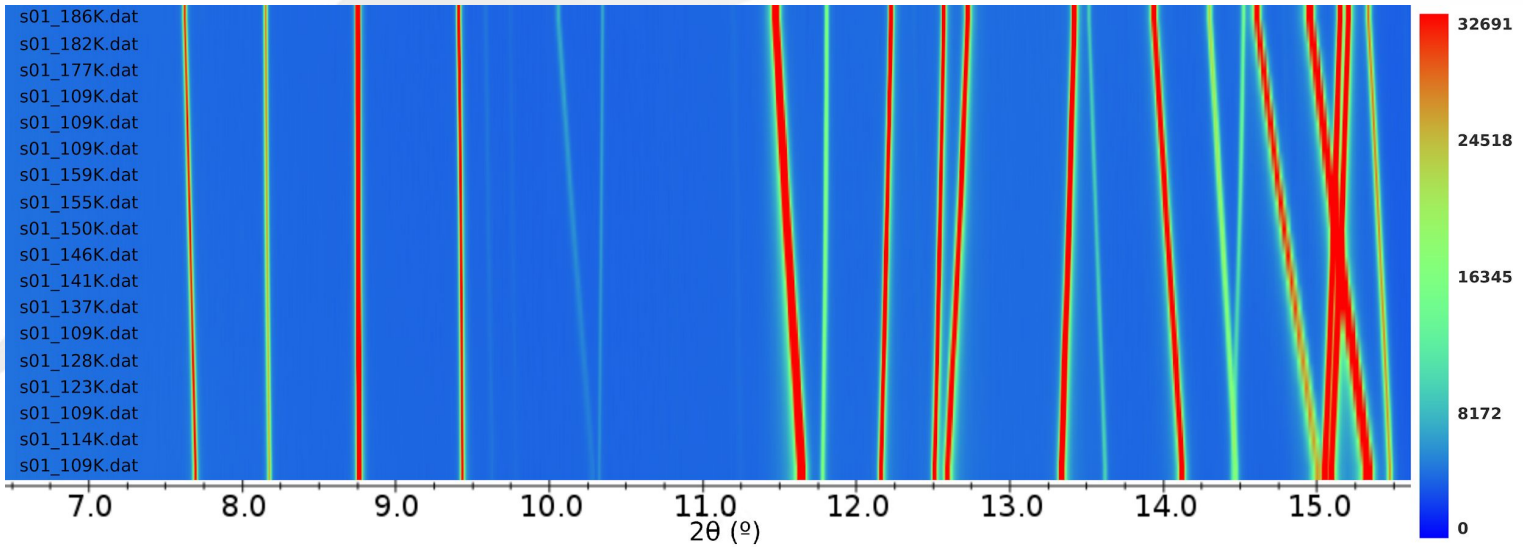


d1Dplot: Plot of multiple data



Sequential

Y-offset



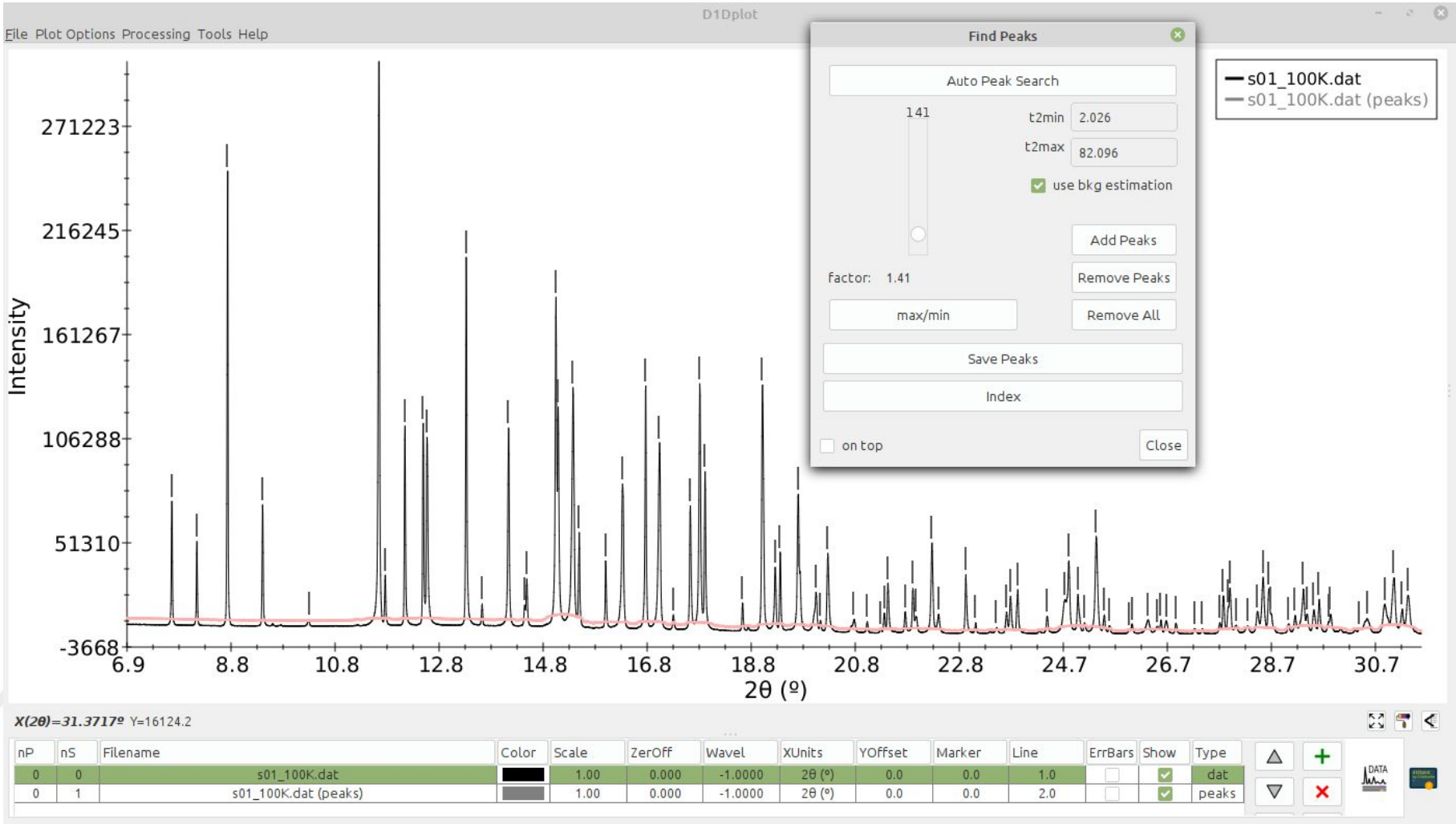
2D plot

(heatmap)

d1Dplot: General data processing

Peak search

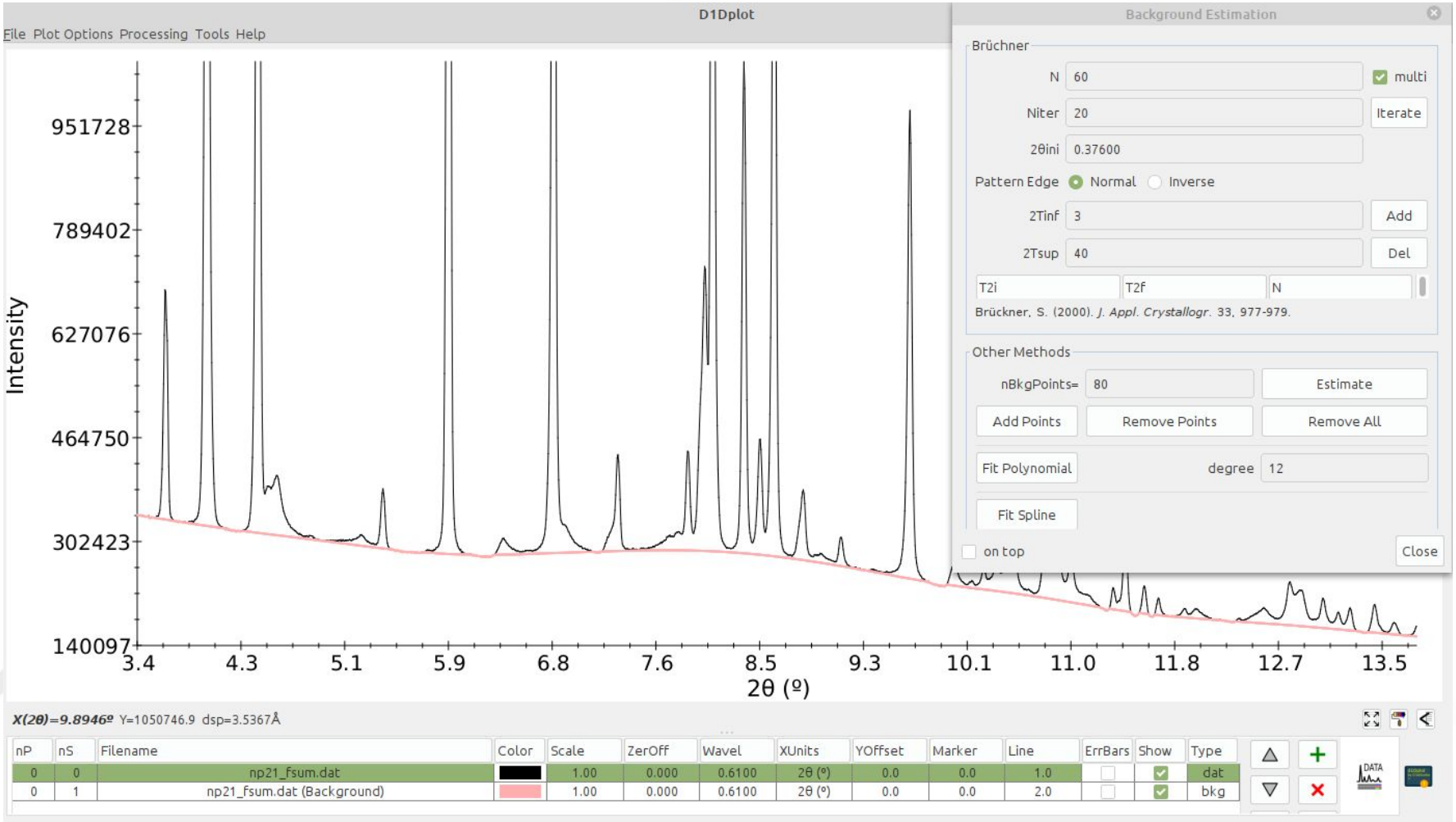
- Find the bragg peaks and save them for further analysis.



d1Dplot: General data processing

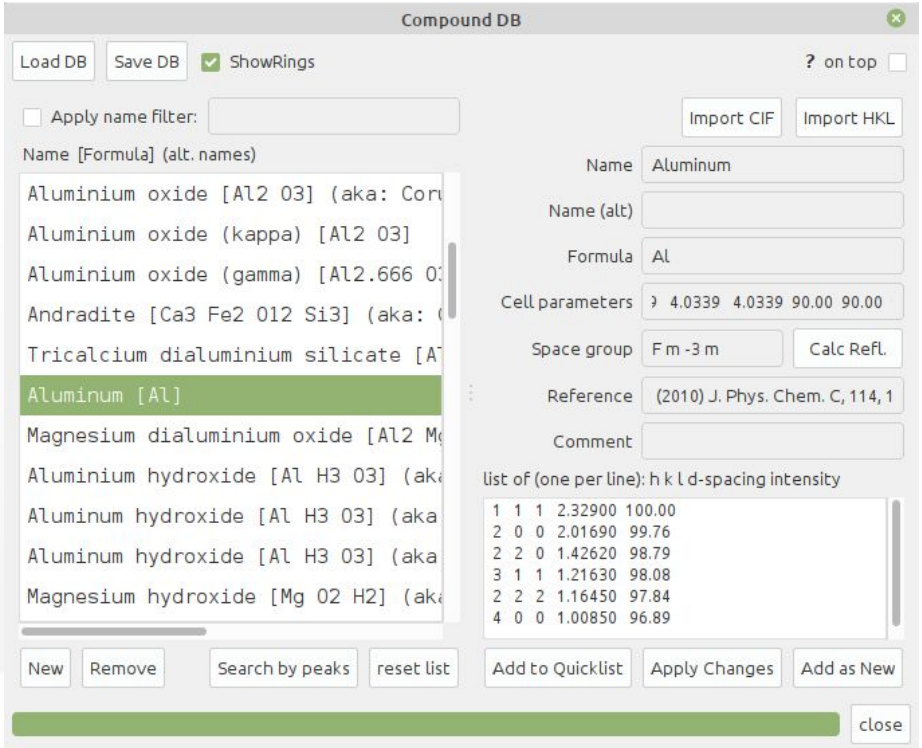
Background estimation

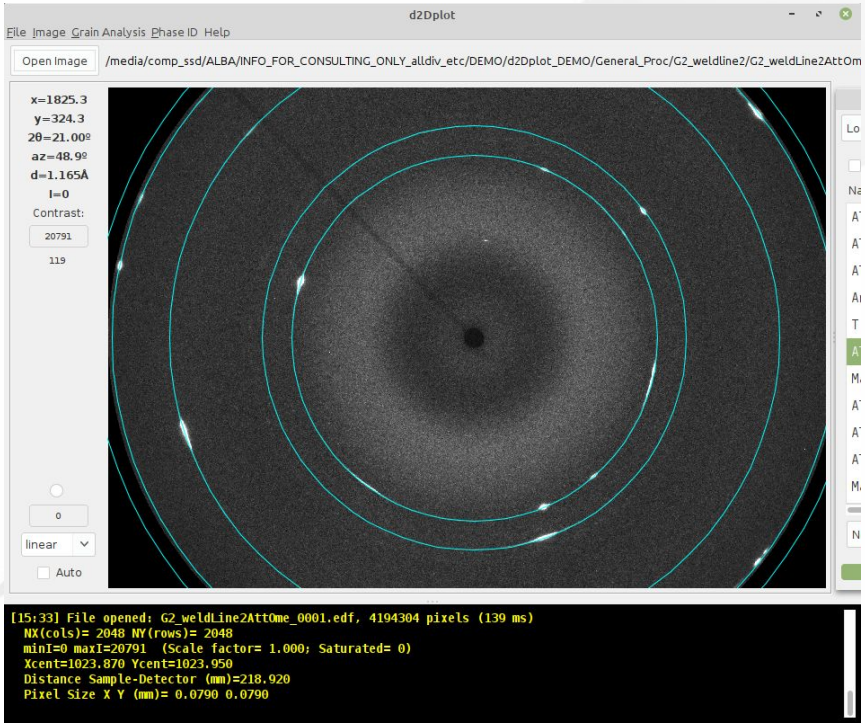
- Smoothing procedure (Brüchner) or interpolation (polynomial or spline)

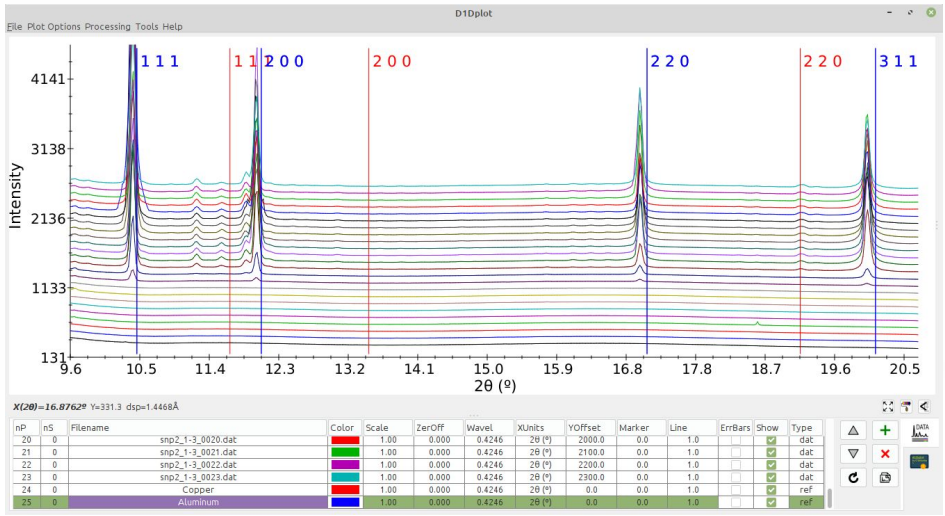


User compound database

- Plot expected reflection positions of selected phase(s)
- Calculate reflections from crystal structure (CIF file) or from cell & space_group
- Search/match option







Command line operation

- Directly open data or perform operations without GUI on multiple files.

```
ori@vava:~$ d1Dplot -help
[16:04] Enter pattern filenames as arguments to open them directly
[16:04] d1Dplot silicon.dat mydata.xye ...
[16:04]
[16:04] TWO AVAILABLE OPTIONS FOR COMMAND LINE ARGUMENTS:
[16:04]  a) Entering pattern filenames as arguments will open them
[16:04]  b) Entering -macro as 1st argument to enable command line
[16:04]
[16:04] In (b) after the -macro argument, the following OPERATIONS
[16:04]
[16:04]   -conv   Individually convert entered patterns according t
[16:04]   -sum Sum the input patterns, additional OPTIONS will be a
[16:04]   -diff FACT [T2I T2F]
[16:04]        In this case, first pattern on the list will act as
[16:04]        The operation is: Patt - Fact*Background
[16:04]        Additional options will be applied on the resulting
[16:04]        If FACT<0 automatic scaling will be performed using
[16:04]        (T2I and T2F can be supplied only when FACT<0)
[16:04]   -rebin T2I STEP T2F
[16:04]        Applies a rebinning on the input patterns according
[16:04]        Additional options may be applied on the resulting f
[16:04]
[16:04] Which can can be combined with the following OPTIONS:
[16:04]
[16:04]   -out NAME    NAME will be added as suffix to the output files when batch processing (before the extension),
[16:04]                For sum and diff options NAME will be the full output filename (without extension)
[16:04]   -xIn XUN     Specify the input x units of the pattern(s) (XUN= 2Theta, d-spacing, 1/dsp2, Q) (def=2Theta)
[16:04]   -xOut XUN    To change the x units of the pattern(s) (XUN= 2Theta, d-spacing, 1/dsp2, Q)
[16:04]   -fmtIn EXT   Specify the input file format of the pattern(s) (EXT= DAT, XYE, GSA, XRDML,...) (def=autodetect)
[16:04]   -fmtOut EXT  Output format of the pattern(s) (EXT= DAT, XYE, GSA, XRDML,...) (def=same as input
[16:04]   -waveIn WL   Wavelength (A) of the input pattern(s) (def= from header if available
[16:04]   -waveOut WL  To change the wavelength of the pattern(s)
```

```
ori@ori-TP /tmp $ ./d2Dplot -macro lab6_180_0003.edf -rint lab6_180.cal
Running on Unix or Linux
Console logging DISABLED
[19:26] MACRO MODE ON
[19:26] Reading img file: lab6_180_0003.edf
[19:26] RINT option found, performing Radial Integration
[19:26] Using integration parameters from CAL file: lab6_180.inp
[19:26]
[19:26] x-beam center: 1023.430
[19:26] y-beam center: 1023.450
[19:26] distance: 181.576
[19:26] wavelength: 0.3187
[19:26] tilt rotation: 35.8
[19:26] angle of tilt: -1.25
[19:26]
[19:26] t2ini: 0.000
[19:26] t2fin: 23.866
[19:26] stepsize: 0.0236
[19:26] start azim: 0.0
[19:26] end azim: 360.0
[19:26] subadu: -9.5
[19:26]
[19:26] Writting output DAT file: lab6_180_0003.dat
ori@ori-TP /tmp $
```

Availability

https://www.cells.es/en/beamlines/bl04-mspd/preparing-your-experiment

Free-of-charge for non-commercial use, user manual, etc...

---

d1Dplot and *d2Dplot* are programmed with the OpenJDK implementation of the Java platform
(License: http://openjdk.java.net/legal/gplv2+ce.html)

The following 3rd party libraries have been used (without modifications):

- Commons Math. https://commons.apache.org/proper/commons-math/
  Apache License: http://www.apache.org/licenses/LICENSE-2.0
- MigLayout. http:// www.miglayout.com
  BSD license: http://directory.fsf.org/wiki/License:BSD_4Clause
- ImageJ 1.50i. https://imagej.nih.gov/ij/index.html
  Public-domain: https://imagej.net/Licensing.
- Apache Batik. https://xmlgraphics.apache.org/batik/
  Apache License: http://www.apache.org/licenses/LICENSE-2.0

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Examples and programs feedback
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Trinitat Pradell (BRCMSE, UPC)
Iris Henríquez (UAB GTS, Chemistry)
Pascal Schmalen & Inma Peral (ULux)
Jose Antonio Ayllón (UAB)
Imanol de Pedro (Univ. Cantabria)
Ana Cuesta & M. Angeles Gómez (UMA)

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Thank you!

& do not hesitate to contact me for any doubt or question!

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