

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



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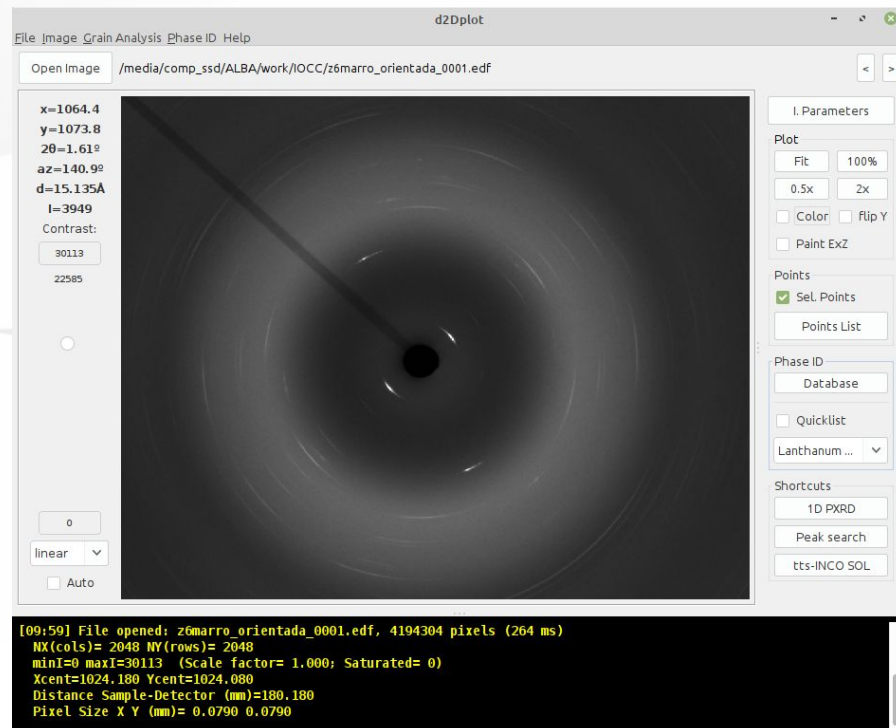
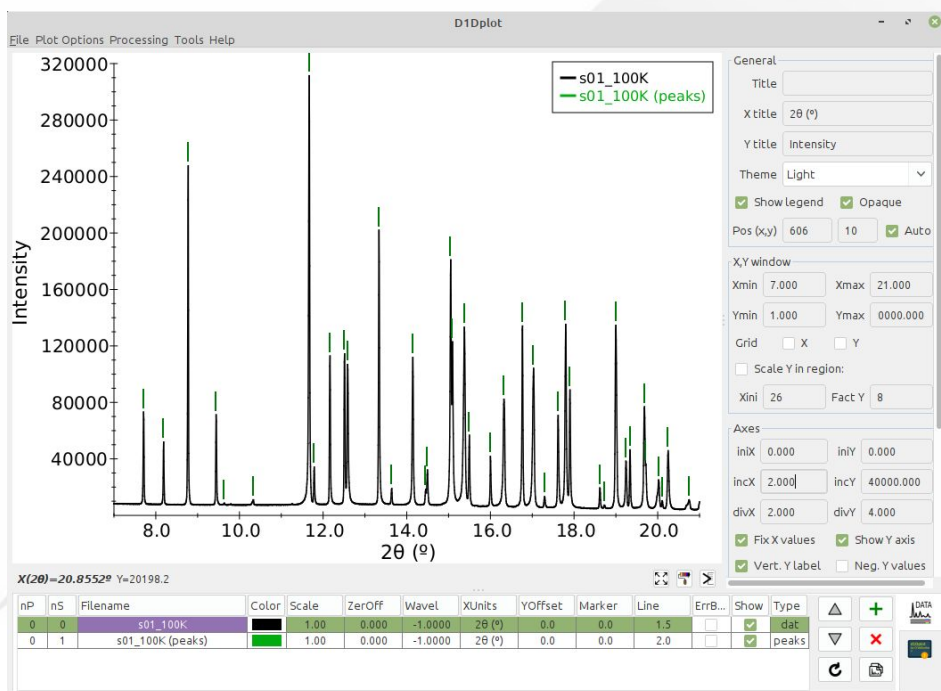
Session H. Software/Tools to Deal with Crystal and Crystallographic Issues & Teaching Crystallography

The 2nd International Online Conference on Crystals

10-20th November 2020



- 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 $tts-\mu$ XRD methodology.





[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 μ XRD**

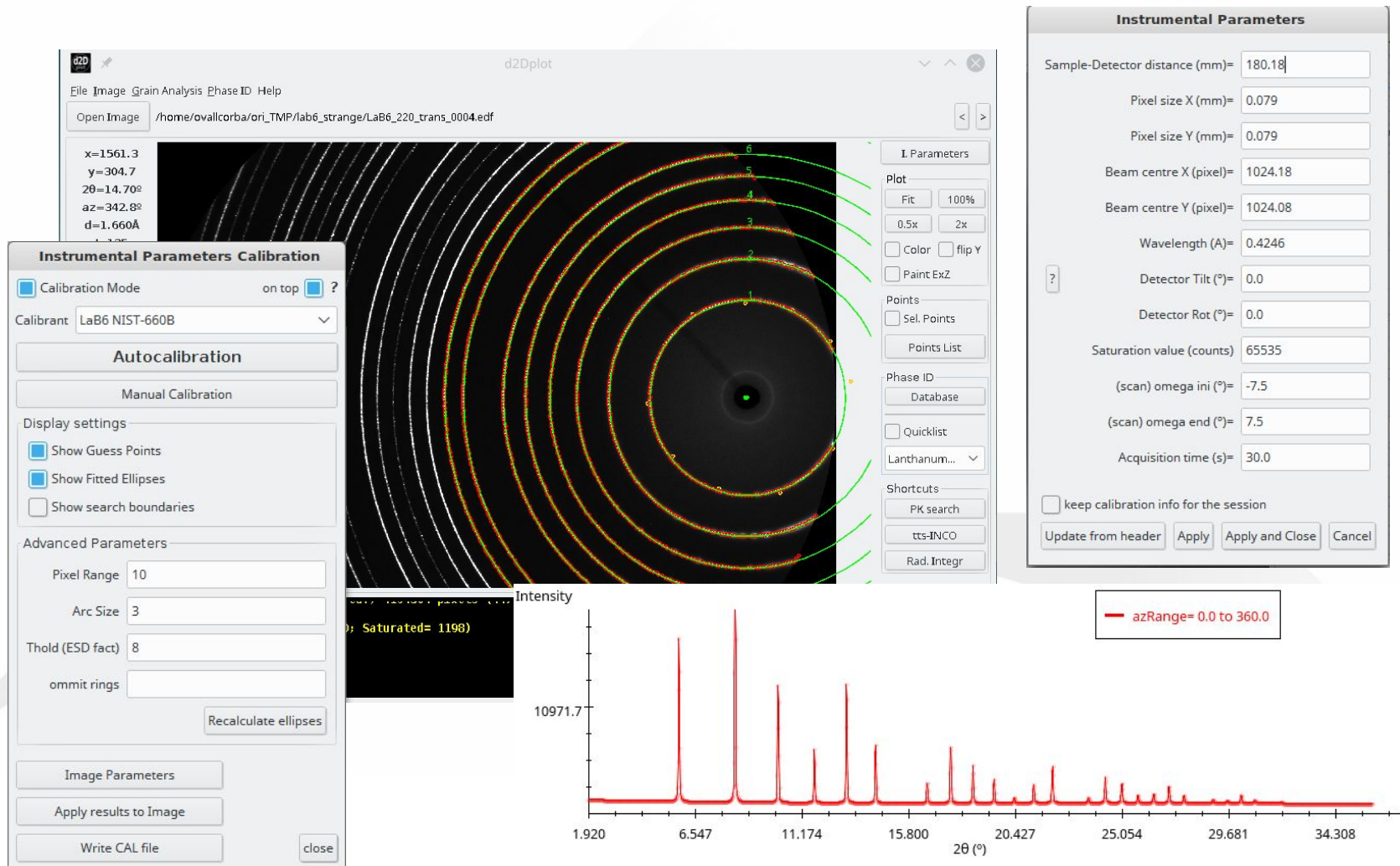
- ◊ Through-the substrate microdiffraction methodology for the structural study of μ volumes of crystals embedded in polished thin sections of compact materials
[J.Rius, O.Vallcorba, C.Frontera, I.Peral, A.Cresi & C.Miravittles. *IUCrJ* **2015**, 2, 452-463]
- ◊ Peak search and integration. Check orientation results.

- **Compound Database for easy phase identification on the images**

- ◊ Plot expected reflection positions, generate reflections, search-match, ...

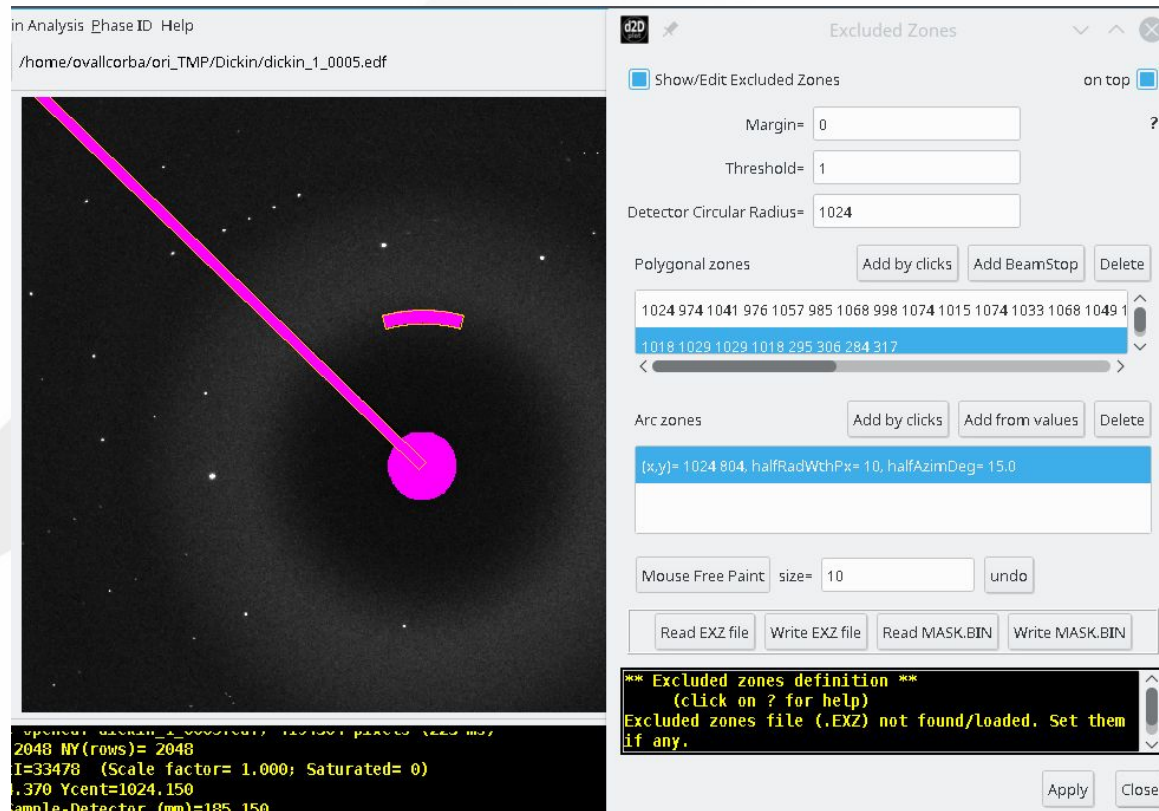
Instrumental calibration (from LaB_6 , Si, etc... diffraction data)

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



Excluded Zones

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



in Analysis Phase ID Help
/home/ovallcorba/ori_TMP/Dickin/dickin_1_0005.edf

d2D Excluded Zones

Show/Edit Excluded Zones on top

Margin= 0 ?

Threshold= 1

Detector Circular Radius= 1024

Polygonal zones

1024 974 1041 976 1057 985 1068 998 1074 1015 1074 1033 1068 1049 1018 1029 1029 1018 295 306 284 317

Arc zones

(x,y)= 1024 804, halfRadWchPx= 10, halfAzimDeg= 15.0

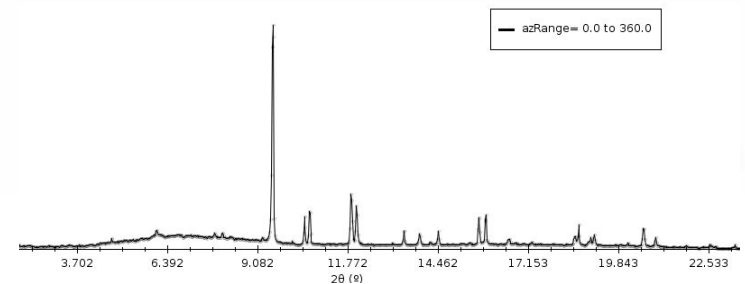
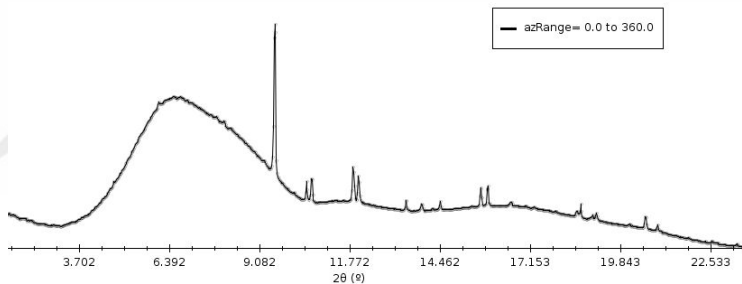
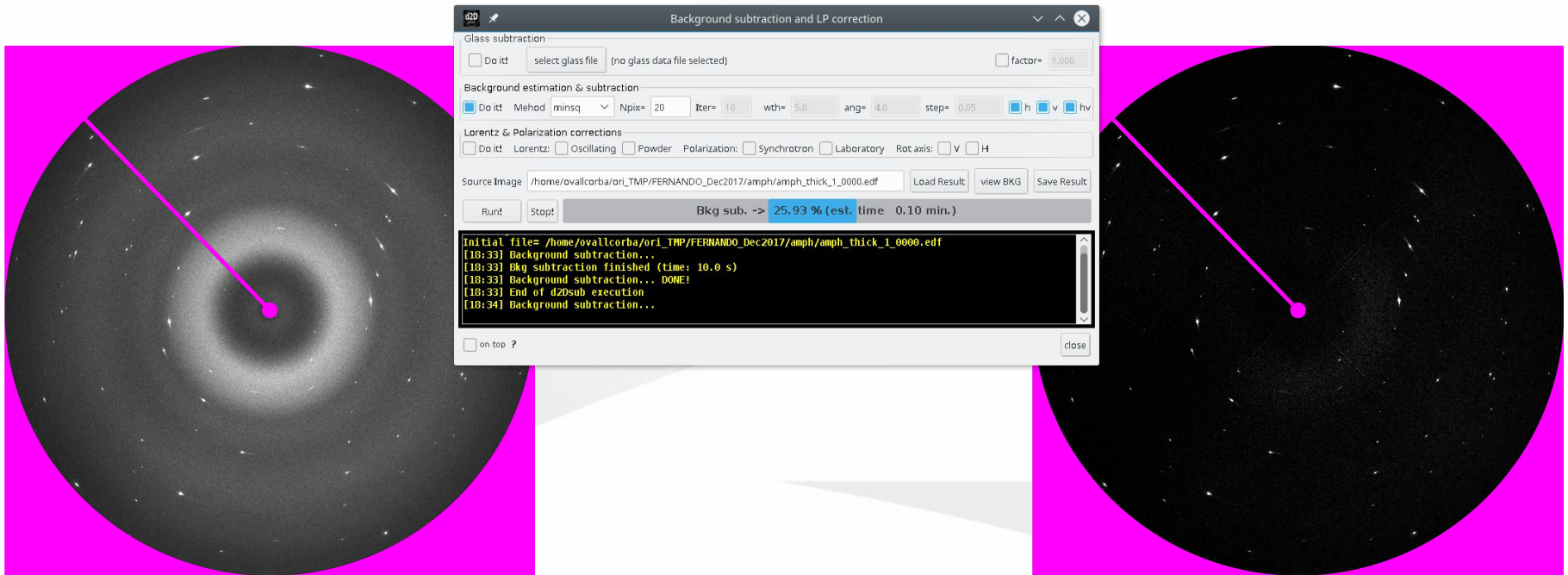
Mouse Free Paint size= 10

*** Excluded zones definition ***
(click on ? for help)
Excluded zones file (.EXZ) not found/loaded. Set them if any.

2048 NY(rows)= 2048
I=33478 (Scale factor= 1.000; Saturated= 0)
1,370 Ycent=1024,150
sample-Detector (mm)=185,150

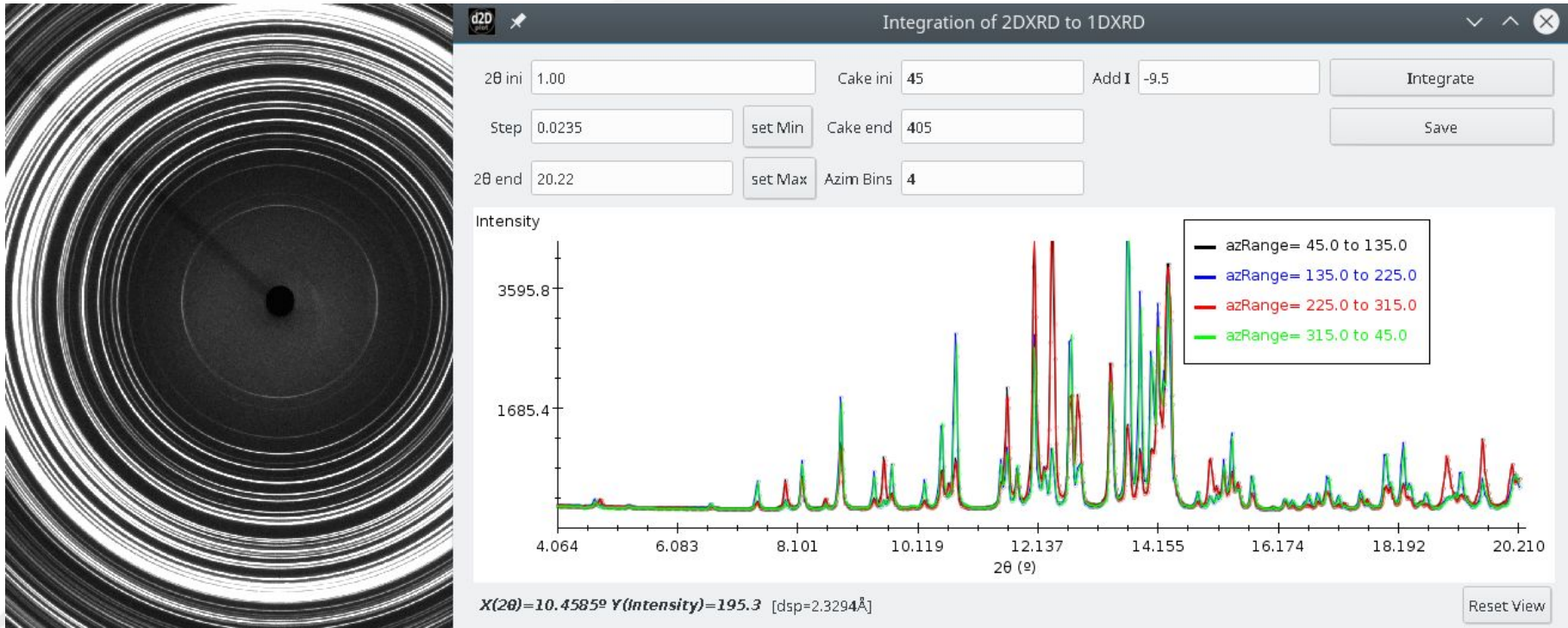
Background subtraction

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



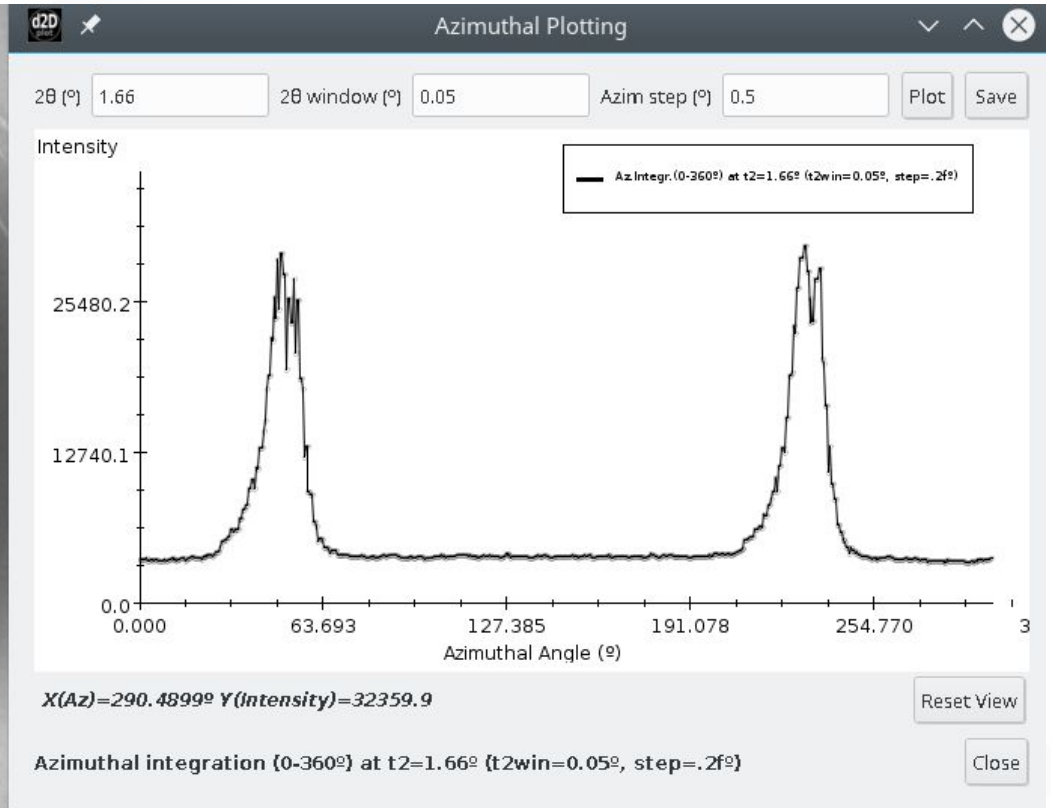
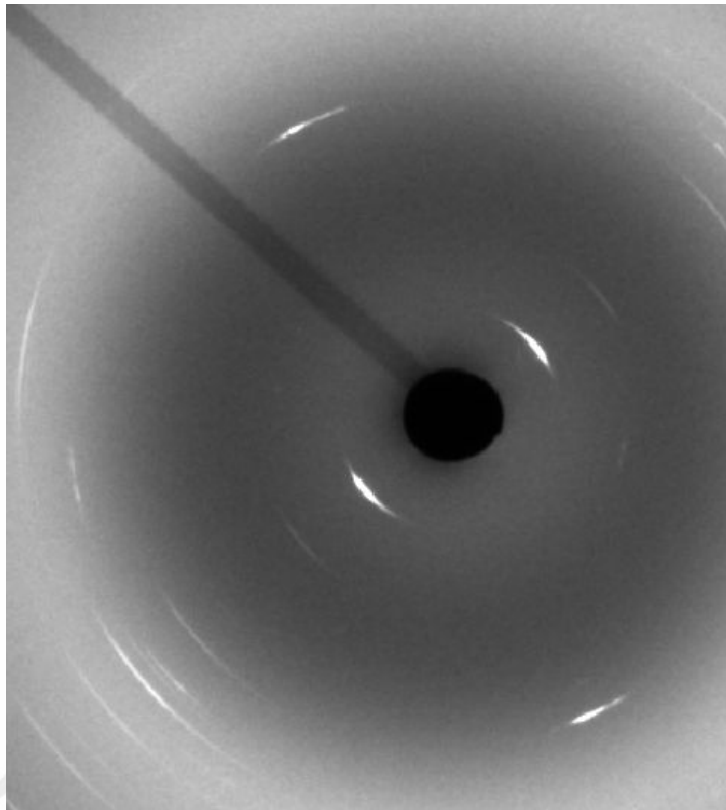
Conversion to 1D-XRD

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



Azimuthal (circular) plot

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





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Consejo Superior de Investigaciones Científicas



Collaborators: Dr. Oriol Vallcorba (ALBA Synchrotron light Source)
Dr. Carlos Frontera (ICMAB-CSIC)

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Disclaimer: This software is distributed WITHOUT ANY WARRANTY. The authors (or their Institutions) have no liabilities in respect of errors in the software, in the documentation and in any consequence of erroneous results or damages arising out of the use or inability to use this software. Use it at your own risk. TTS_software is programmed with Fortran [LF95 (win), GNU Fortran (linux)]. This frontend is programmed with Java.

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

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

Jordi Rius,^{a*} Ana Labrador,^b Anna Crespi,^a Carlos Frontera,^a Oriol Vallcorba^a and Joan Carles Melgarejo^c



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 Miravittles^a

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

DE GRUYTER

Z. Kristallogr. 2017; aop

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

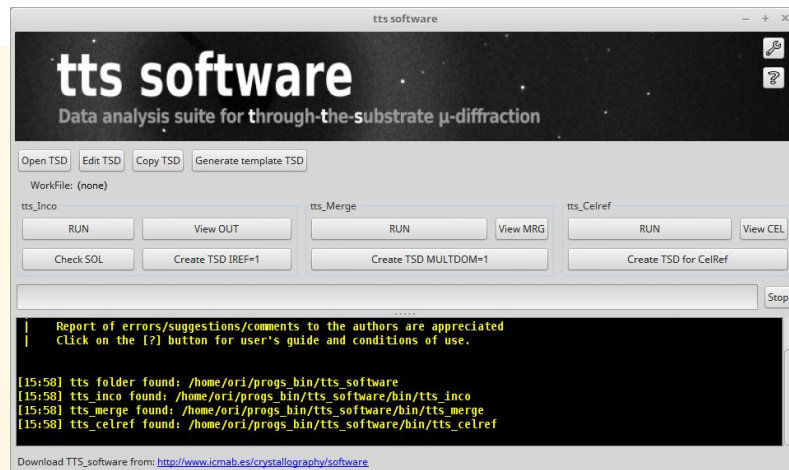
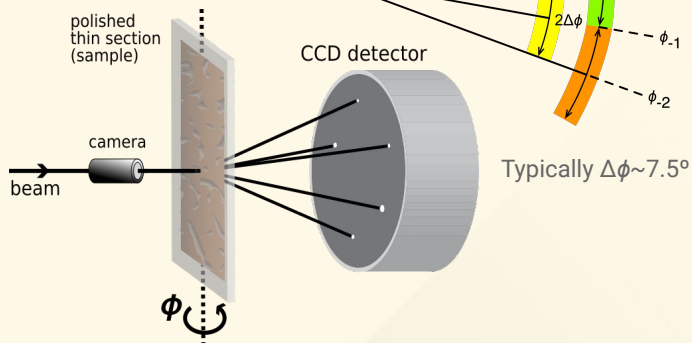
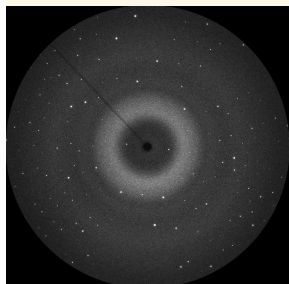
Increasing data completeness in synchrotron tts-microdiffraction experiments for δ -recycling phasing of low-symmetry compounds

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

d2Dplot: tts- μ XRD

d2Dplot

(Java code)



Diffraction images
(*tts data collection strategy*)

Check intermediate outputs

Help prepare input files for *tts_software*, run and check results.
Create mask.bin file

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)

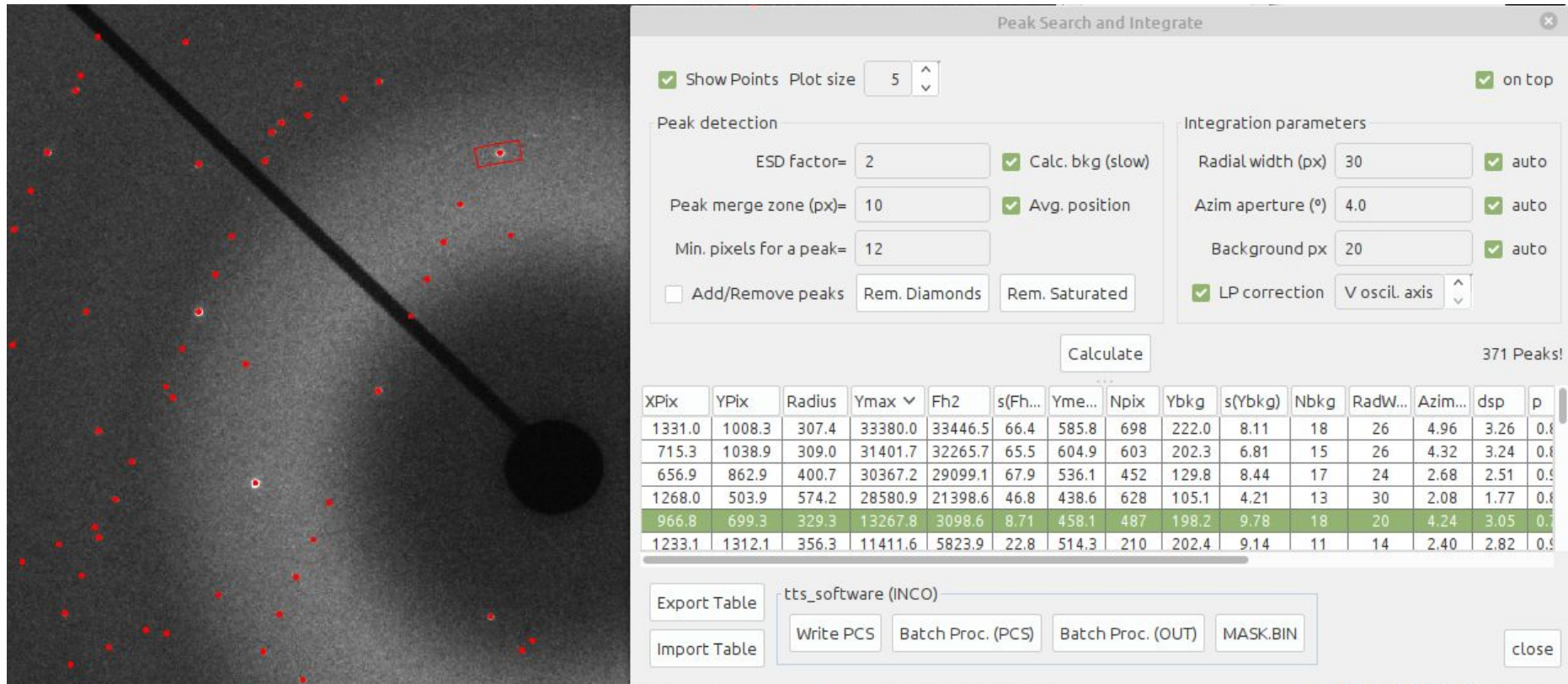
TTS software

(Fortran codes)

For all the measured microvolumes

Peak search and integration

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



Peak Search and Integrate

Show Points Plot size: 5 on top

Peak detection

ESD factor= 2 Calc. bkg (slow)

Peak merge zone (px)= 10 Avg. position

Min. pixels for a peak= 12

Add/Remove peaks Rem. Diamonds Rem. Saturated

Integration parameters

Radial width (px) 30 auto

Azim aperture (°) 4.0 auto

Background px 20 auto

LP correction V oscil. axis

Calculate 371 Peaks!

XPix	YPix	Radius	Ymax	Fh2	s(Fh...	Yme...	Npix	Ybkg	s(Ybkg)	Nbkg	RadW...	Azim...	dsp	p
1331.0	1008.3	307.4	33380.0	33446.5	66.4	585.8	698	222.0	8.11	18	26	4.96	3.26	0.8
715.3	1038.9	309.0	31401.7	32265.7	65.5	604.9	603	202.3	6.81	15	26	4.32	3.24	0.8
656.9	862.9	400.7	30367.2	29099.1	67.9	536.1	452	129.8	8.44	17	24	2.68	2.51	0.9
1268.0	503.9	574.2	28580.9	21398.6	46.8	438.6	628	105.1	4.21	13	30	2.08	1.77	0.8
966.8	699.3	329.3	13267.8	3098.6	8.71	458.1	487	198.2	9.78	18	20	4.24	3.05	0.7
1233.1	1312.1	356.3	11411.6	5823.9	22.8	514.3	210	202.4	9.14	11	14	2.40	2.82	0.9

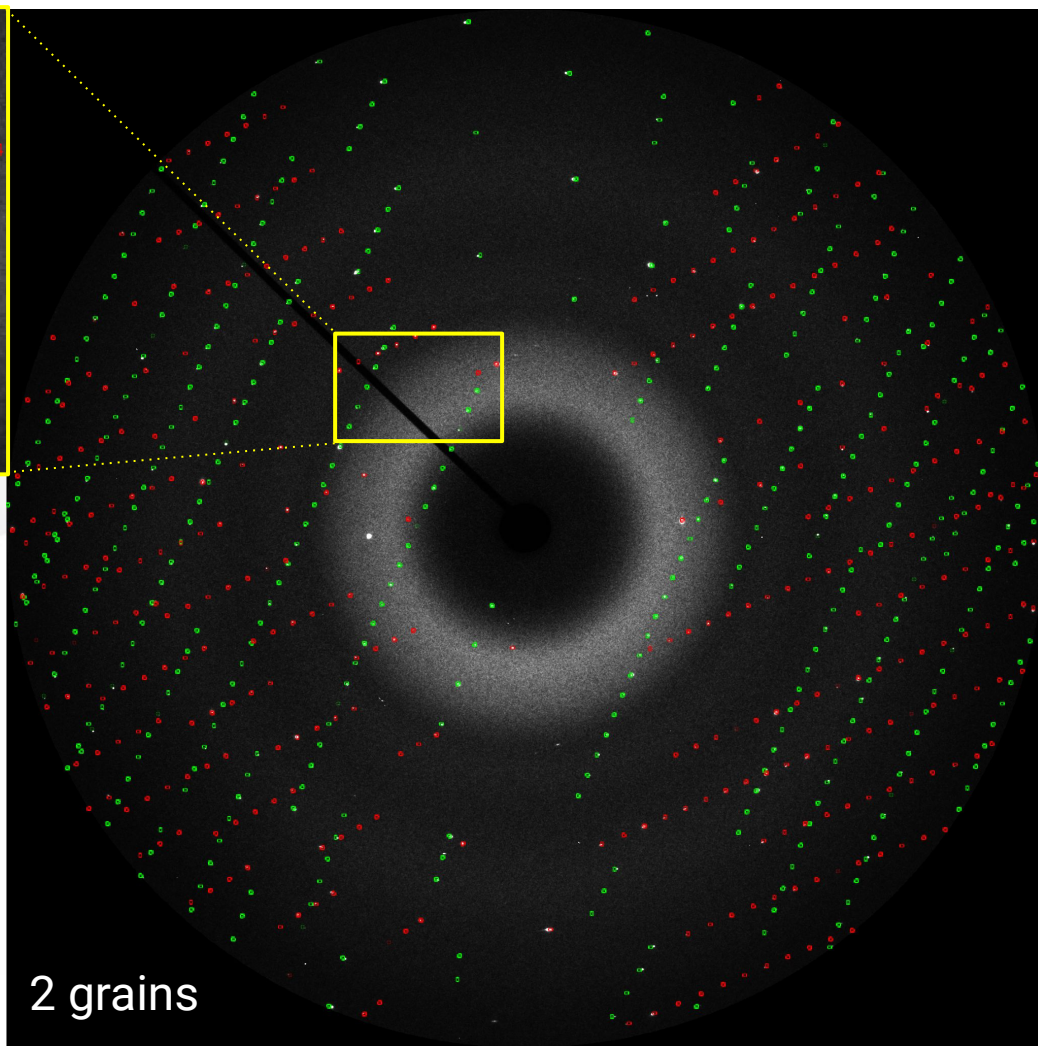
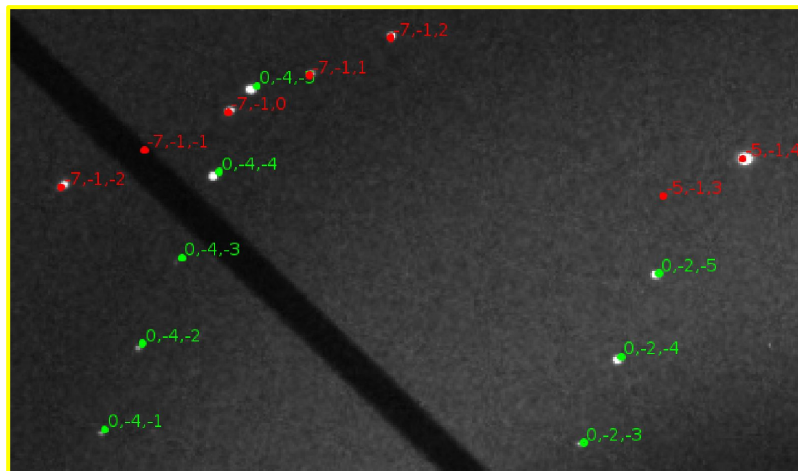
Export Table Import Table

tts_software (INCO)

Write PCS Batch Proc. (PCS) Batch Proc. (OUT) MASK.BIN

close

Select the correct grain orientation(s)



tts_INCO

Load PXY/SOL file on top

dickin_14.SOL

Solution list (Nsol Nref_total Nref_matching):

1	397	165	alon=186.667	alat=86.333	aspin=246.333
6	397	165	alon=186.667	alat=86.333	aspin=246.333
2	359	124	alon=70.000	alat=81.000	aspin=24.667
3	359	124	alon=70.000	alat=81.000	aspin=24.667

Show Spots Show HKL Add Peaks

Peak List (Nr pX pY h k l Fc Swing):

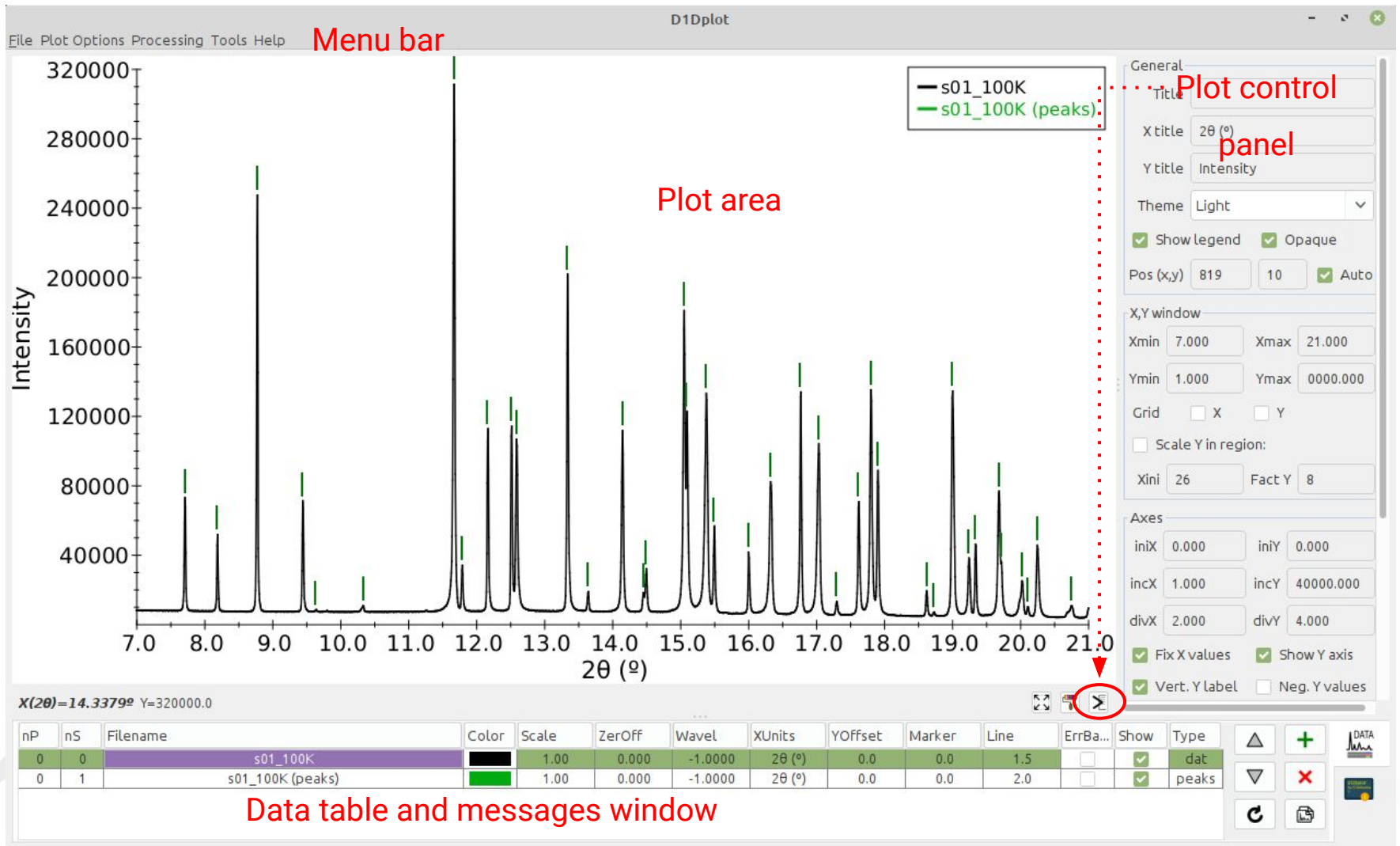
Nr	pX	pY	h	k	l	Fc	Swing
1	1265	48	4	-2	-23	25.00	-3.67
2	1630	223	4	2	-23	25.00	3.26
3	1432	186	4	0	-22	25.00	-2.64
4	1614	269	4	2	-22	25.00	1.45
5	1728	324	5	3	-22	25.00	-2.09
6	1296	172	2	1	-21	25.00	2.70

Extract Intensities



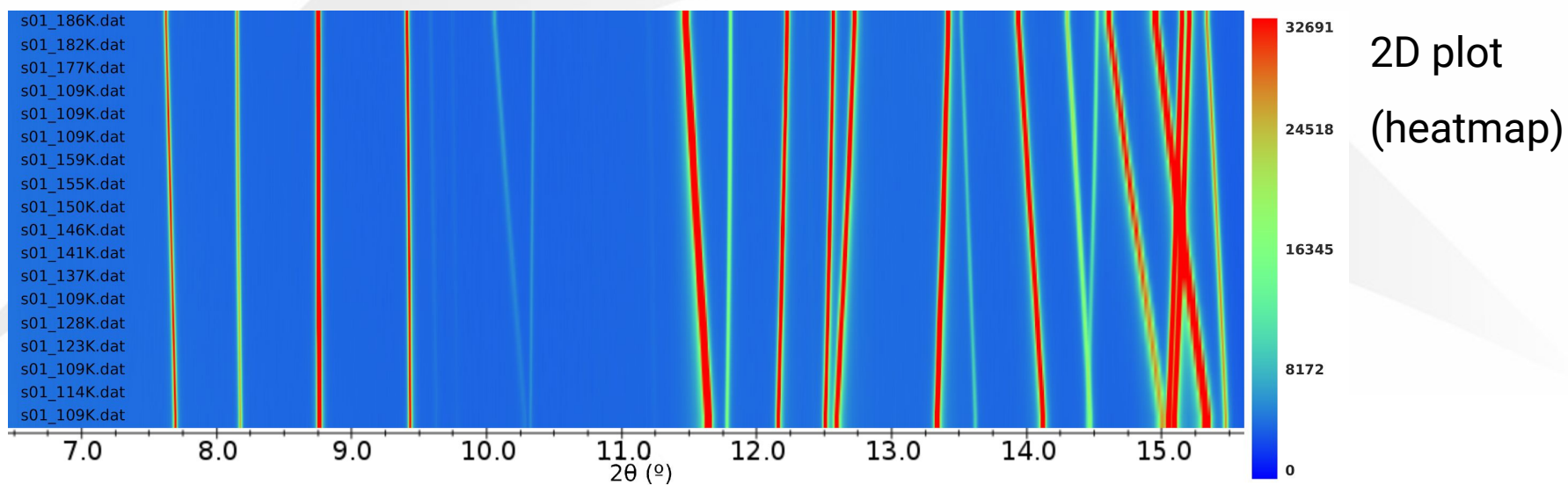
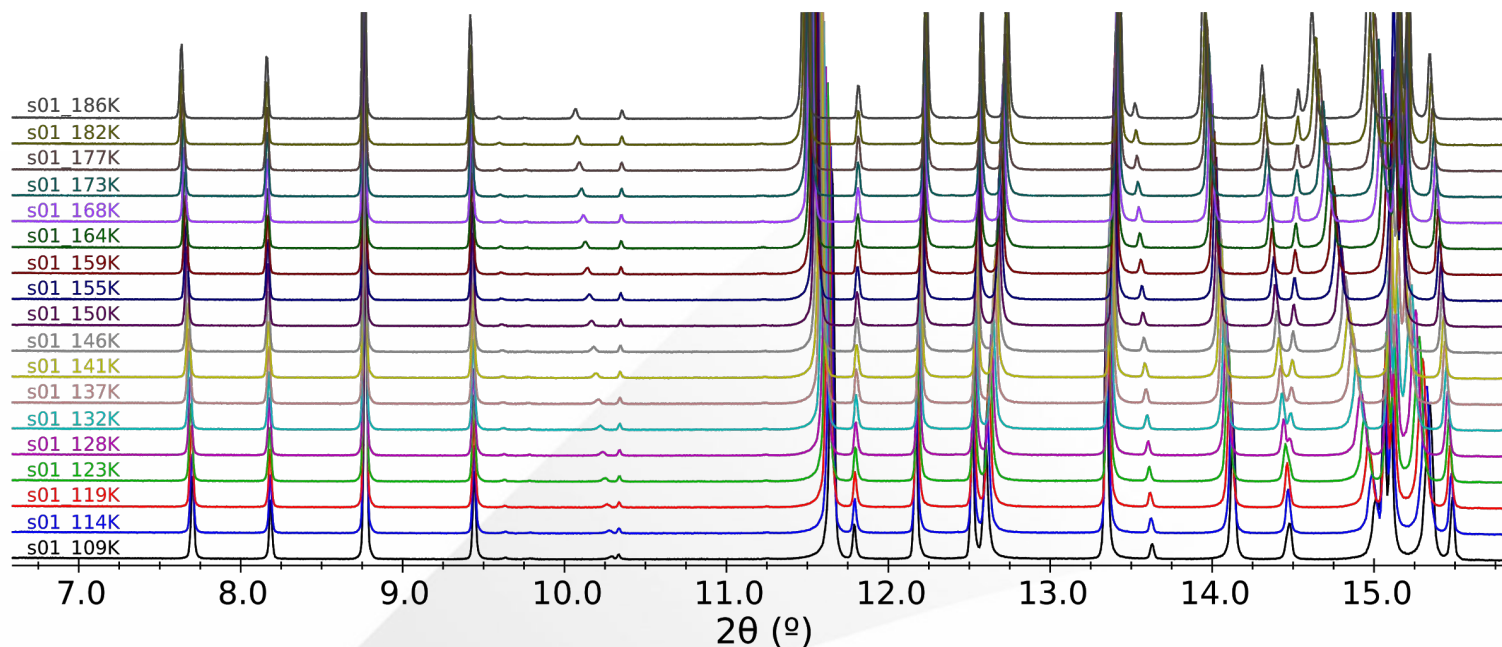
- **Visual Inspection and basic processing of 1D X-ray diffraction data**
 - ◊ User-friendly interface with easy mouse navigation and on-screen info
 - ◊ Multi-pattern stacking, 2D plot
 - ◊ Total control of the aspect and functionality (axes, zones, zoom, mouse...)
 - ◊ Basic operations as rebinning, 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



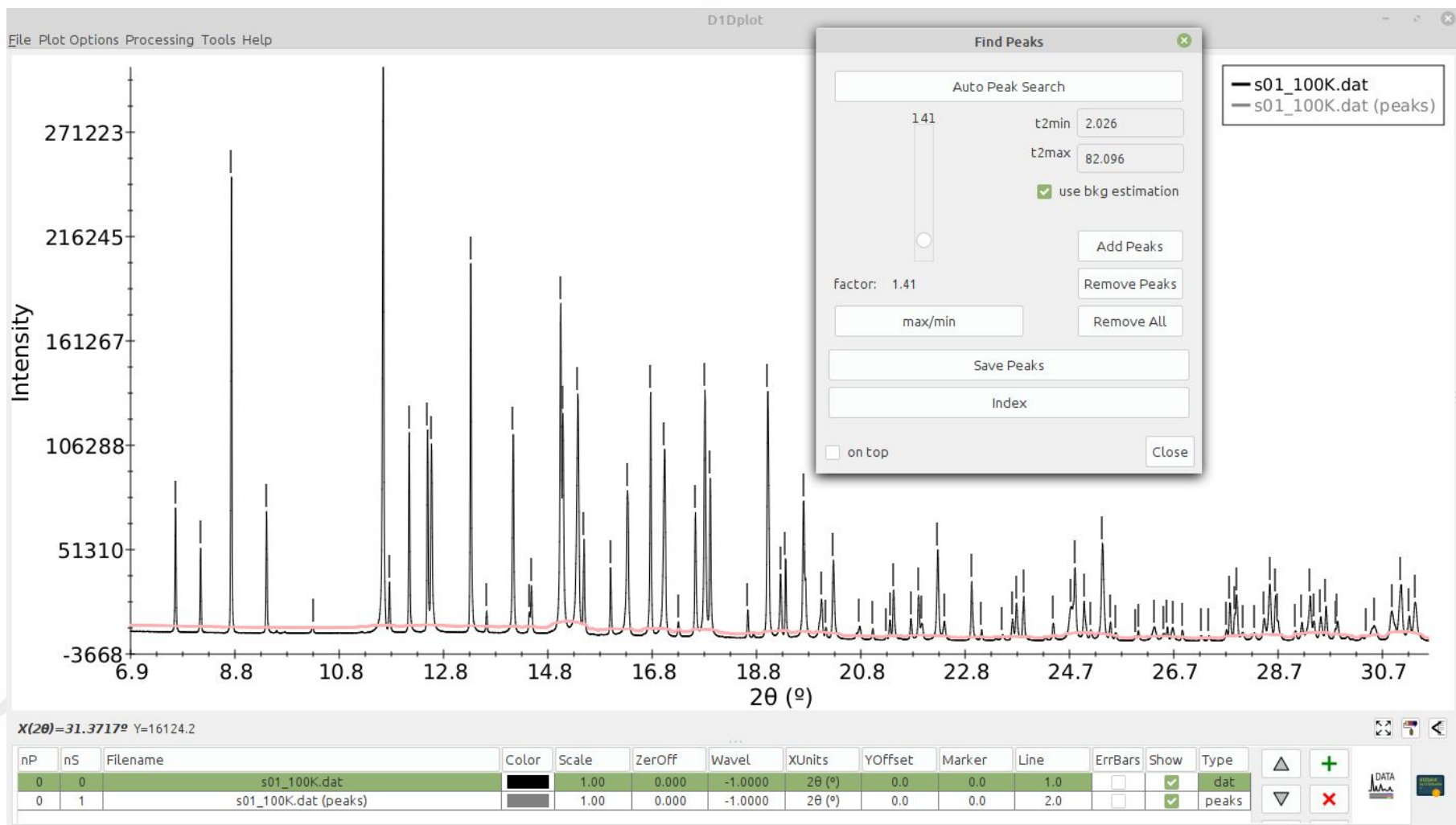
Data table and messages window

d1Dplot: Plot of multiple data



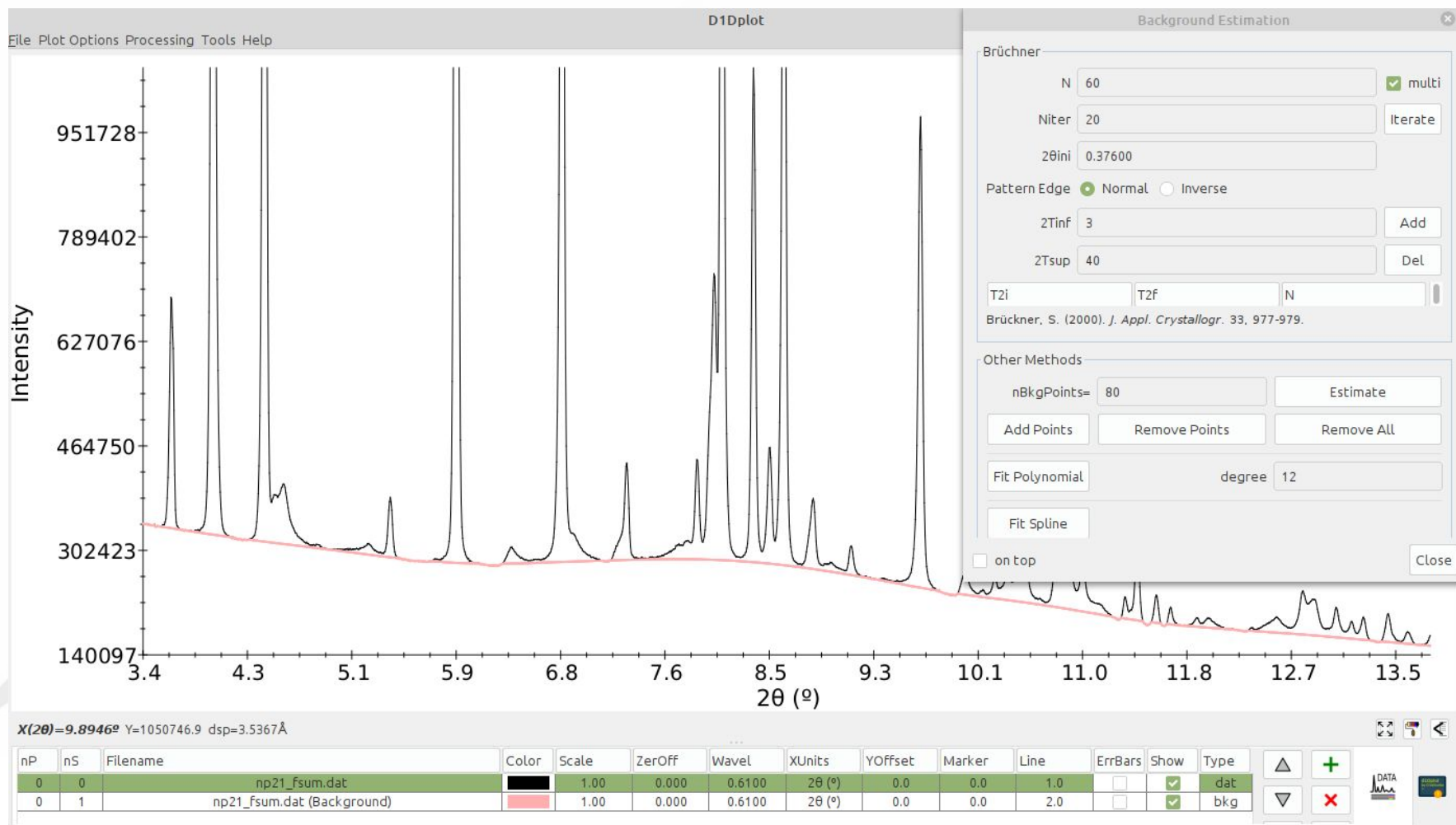
Peak search

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



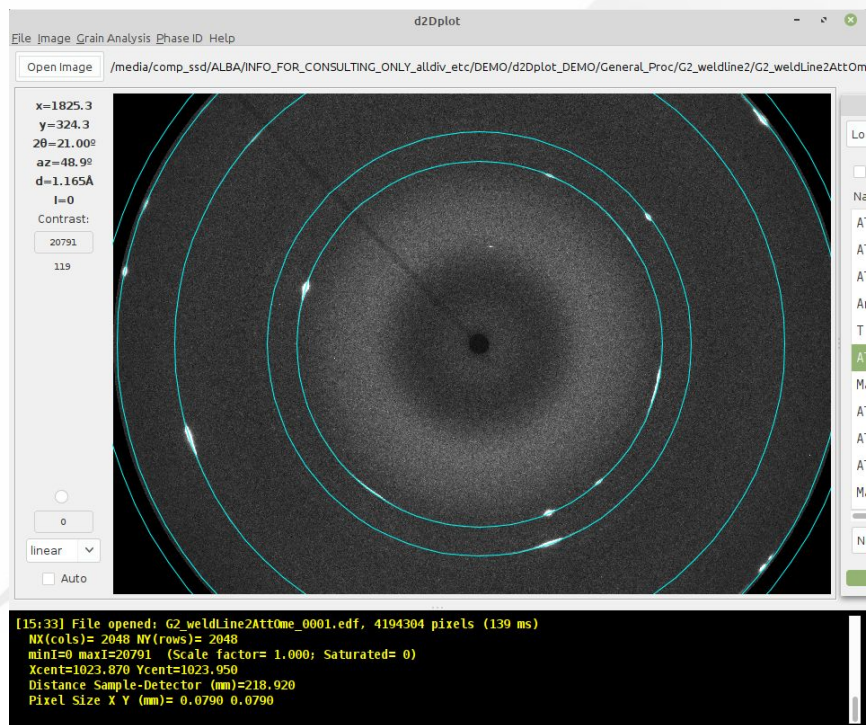
Background estimation

- 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



Compound DB

Load DB Save DB ShowRings ? on top

Apply name filter:

Import CIF Import HKL

Name [Formula] (alt. names)

- Aluminium oxide [Al₂O₃] (aka: Corundum)
- Aluminium oxide (kappa) [Al₂O₃]
- Aluminium oxide (gamma) [Al₂.666 O₃]
- Andradite [Ca₃ Fe₂ O₁₂ Si₃] (aka: Andradite)
- Tricalcium dialuminium silicate [Al₂Si₂O₇]
- Aluminum [Al]**
- Magnesium dialuminium oxide [Al₂MgO₄]
- Aluminium hydroxide [Al H₃ O₃] (aka: Gibbsite)
- Aluminum hydroxide [Al H₃ O₃] (aka: Gibbsite)
- Aluminum hydroxide [Al H₃ O₃] (aka: Gibbsite)
- Magnesium hydroxide [Mg O₂ H₂] (aka: Brucite)

Name Aluminum

Name (alt)

Formula Al

Cell parameters 4.0339 4.0339 90.00 90.00

Space group Fm -3 m Calc Refl.

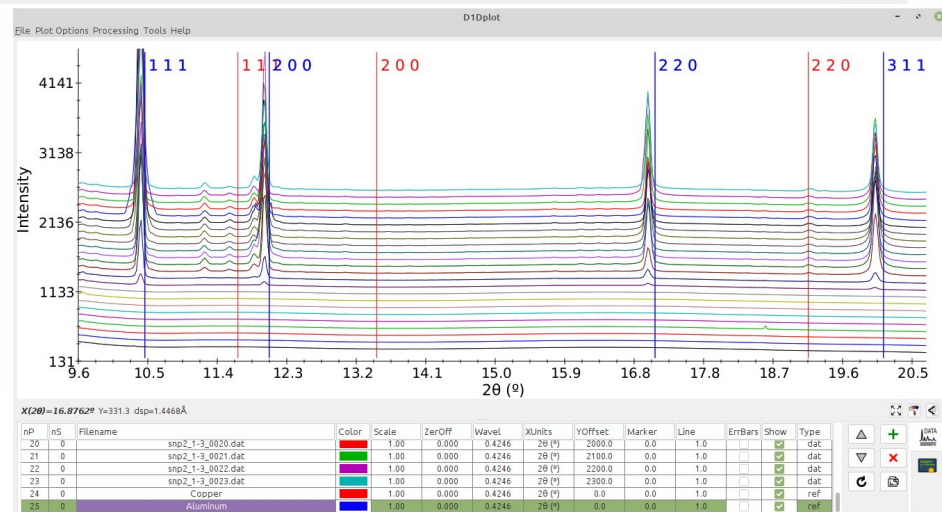
Reference (2010) J. Phys. Chem. C, 114, 1

Comment

list of (one per line): h k l d-spacing intensity

1	1	1	2.32900	100.00
2	0	0	2.01690	99.76
2	2	0	1.42620	98.79
3	1	1	1.21630	98.08
2	2	2	1.16450	97.84
4	0	0	1.00850	96.89

New Remove Search by peaks reset list Add to Quicklist Apply Changes Add as New close



- 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 to
[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] Writing output DAT file: lab6_180_0003.dat
ori@ori-TP /tmp $
```



<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](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>



ALBA-CELLS MSPD Beamline

François Fauth (BL responsible)
Aleksandr Missiul (BL postdoc)
Catalin Popescu (BL scientist)

ICMAB-CSIC

Jordi Rius
Anna Crespi
Carlos Frontera

€€€

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

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10-20th November 2020