General review of the software to integrate synchrotron and laboratory high-pressure single-crystal data from area-sensitive detectors

Andrzej Grzechnik
Institute of Crystallography, RWTH Aachen University, Germany

Ross Angel
Dipartimento di Geoscienze, Universita di Padova

Commercial

Free-ware
Macromolecular crystallography

Supported data formats

- Bruker and Crysalis software can be used for the data from some other detectors & instruments, e.g., synchrotrons.
- X-Area is restricted to the STOE diffractometers.
- XDS reads essentially any data format for any detector in a laboratory and synchrotron facility. It is also able to understand a geometry of any diffractometer.

Integration: area detectors

$$F_{hkl} = \sum_{1}^{N} f_n^{\,2\pi i(hx_n + ky_n + lz_n)}$$

What influences measured single-crystal x-ray intensities in general?

- Lorentz polarisation
- Extinction
- Absorption
- Double diffraction
- Instability of the source of the primary beam
- Instability of the crystal (decomposition and/or positional instability)

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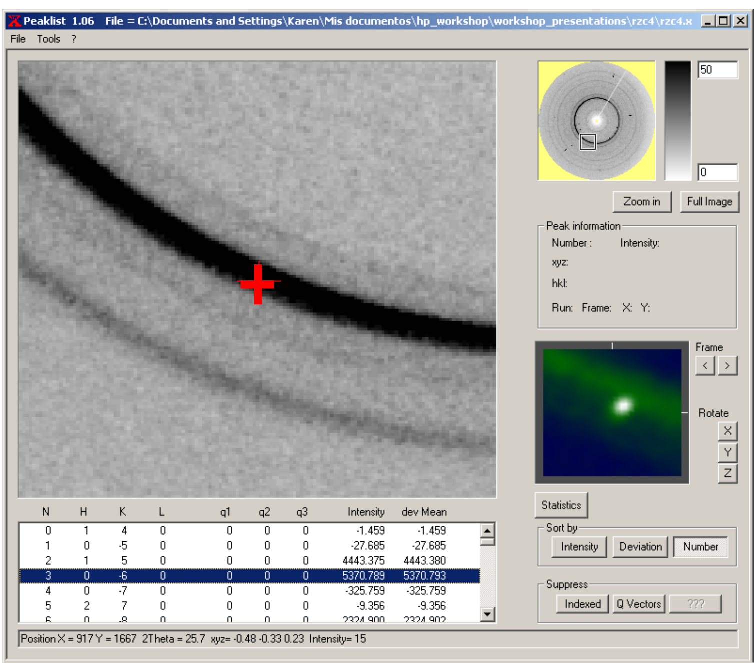
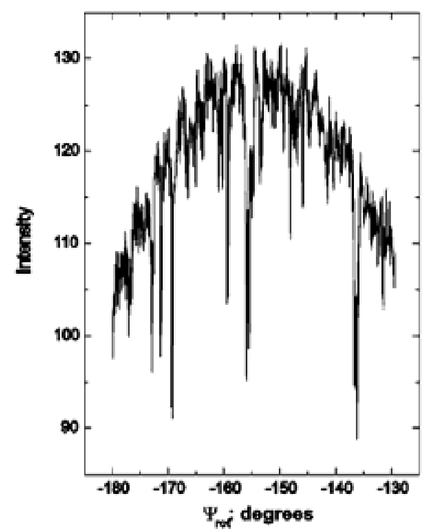
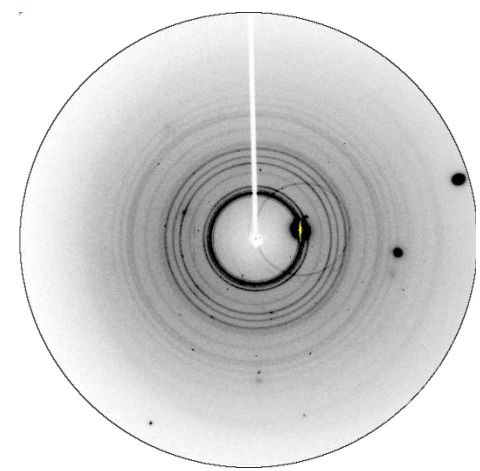
What else influences measured single-crystal x-ray intensities in high-pressure measurements?

- Varying intensity of the primary beam due to strong diamond reflections ("diamond dips")
- Absorption by the diamonds and gasket
- Overlap with reflections due to diamonds, ruby, quartz, etc., and gasket rings
- Shading of the detector







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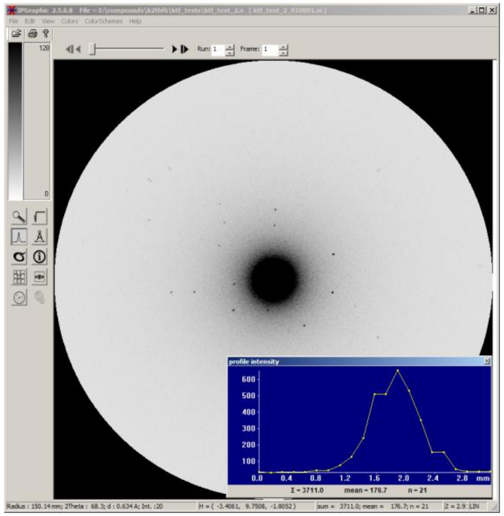
What influences the profile of the Bragg reflections in general?



Resolution

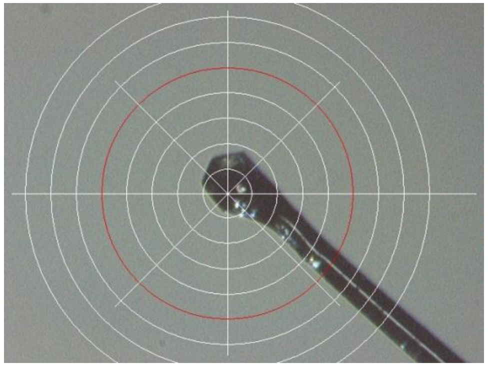
Crystal size, shape & orientation

Mosaicity

Beam focus dimensions and divergence

Wavelength dispersion

Optics



Detector point spread and spatial distortions

Background (dependent on whether the CCD, image plate, or PILATUS are used – global vs. local; also diffuse scattering, etc.)

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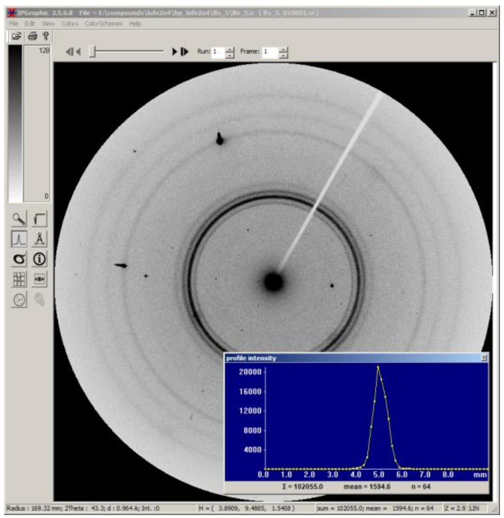
What else influences the profile of the Bragg reflections at high pressures?



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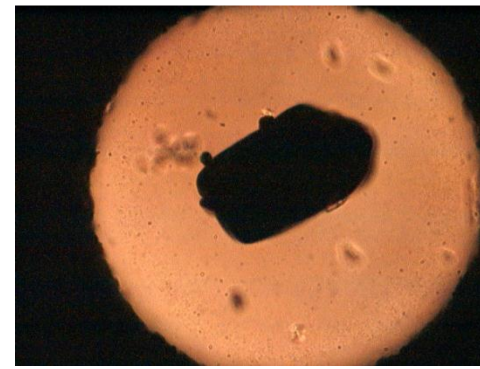

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Background due to the overlap with the reflections from diamonds, ruby, quartz, etc.

Background due to the overlap with the gasket rings

Lattice distortions (non-hydrostatic conditions)

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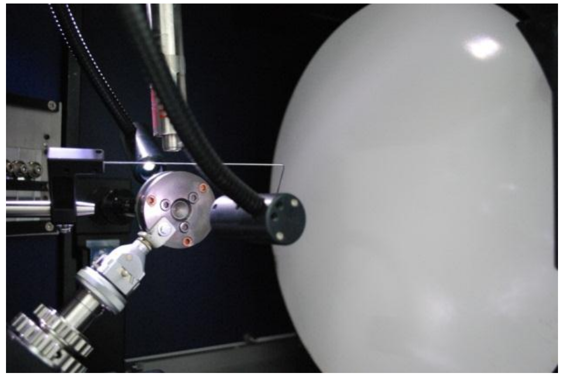
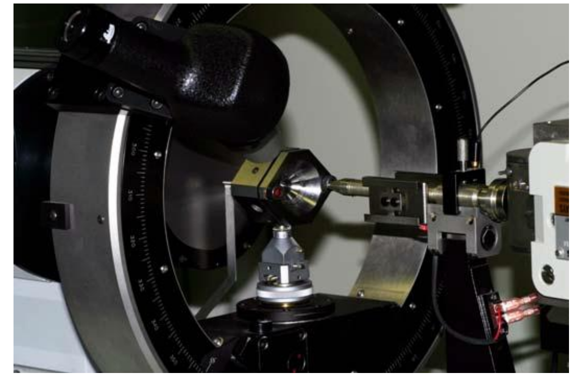

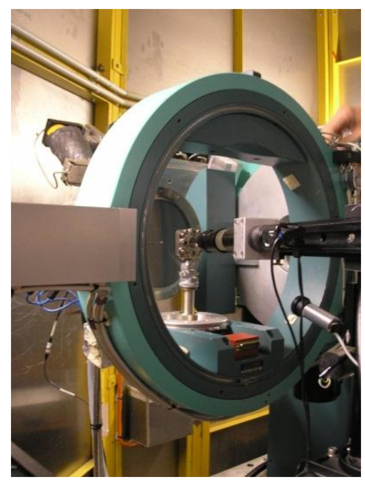
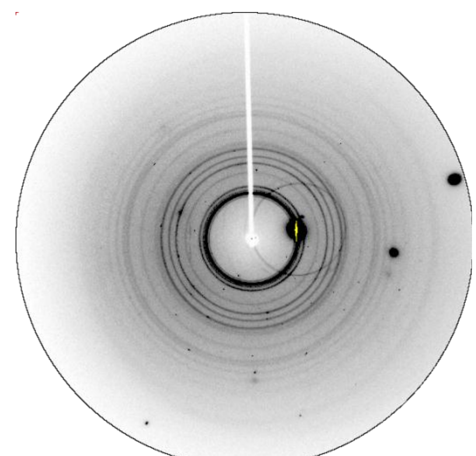

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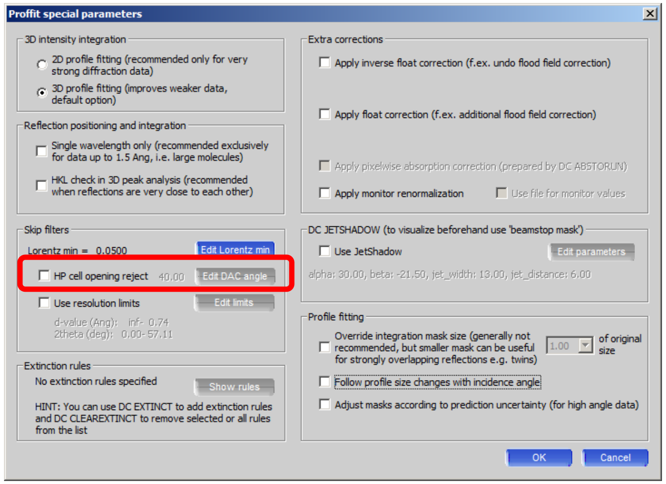

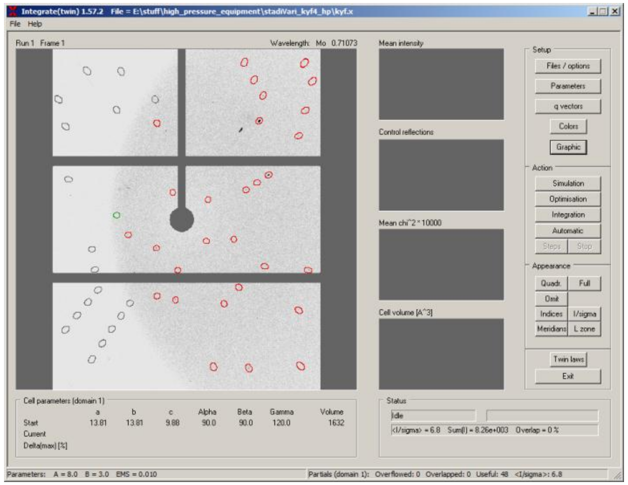

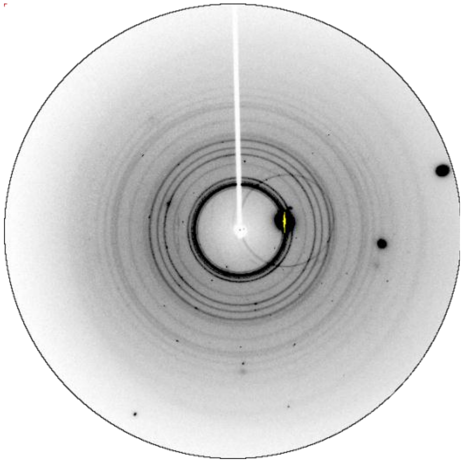

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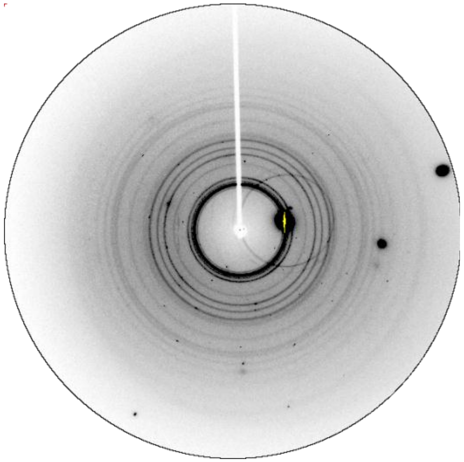

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Robust profile fitting beyond the assumption of an average profile and integration of multiple phases.

0	0	0	0	0	1	0	0	0	0	0	0	0	1	1	0	0	0	0	0	-1	-1	0	0	0	0	0	
0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	0	0	
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0	0	0	0	0	0	0	0	0	0	0	0	1	2	0	0	0	0	0	0	1	5	12	5	1	0	0	
0	0	0	0	0	0	0	0	0	0	0	0	2	4	2	0	0	0	0	0	1	10	26	10	1	0	0	block 1 2 3
0	0	0	0	0	0	0	0	0	0	0	0	1	2	1	0	0	1	0	0	1	5	12	5	1	0	0	
0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	1	2	1	0	0	0	
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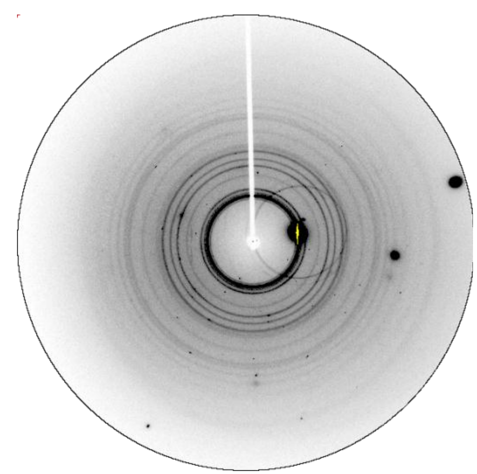
e.g., "*Kabsch*" profiles in XDS



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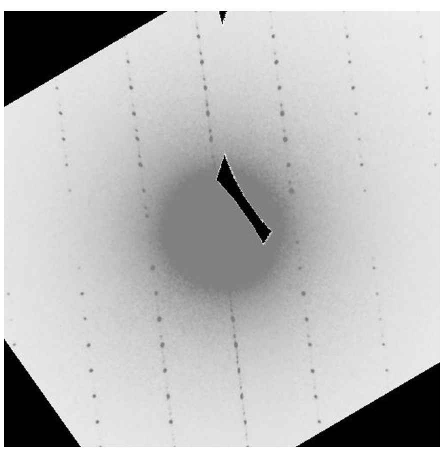

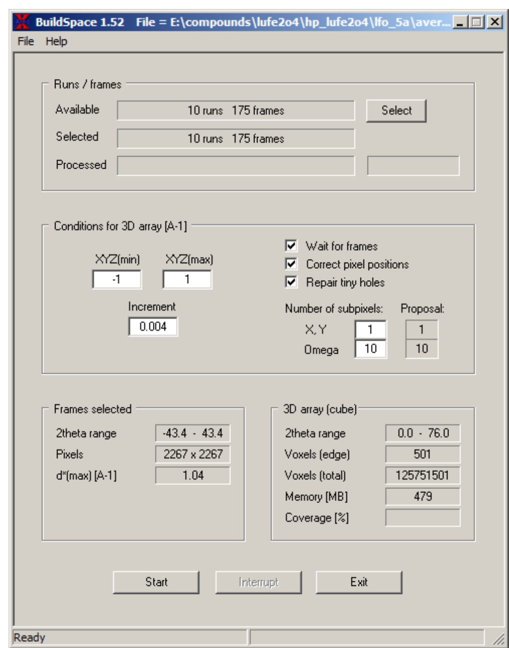

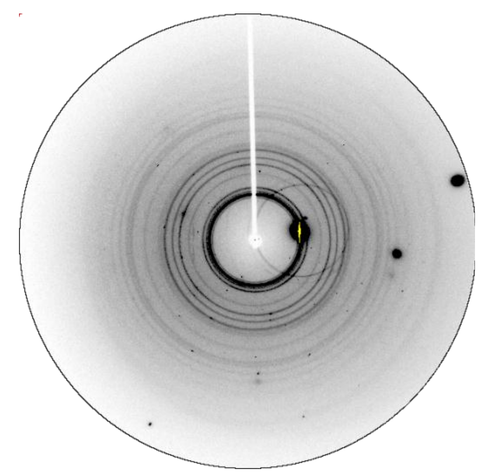

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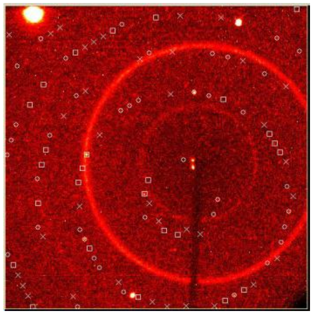

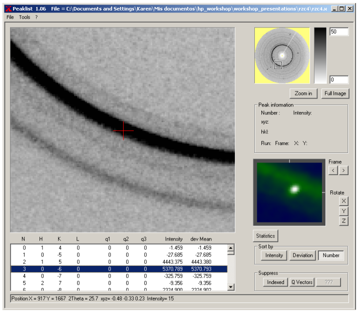

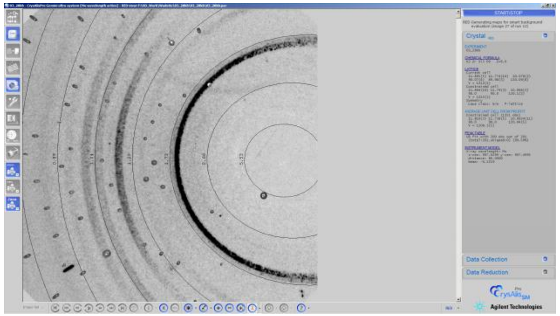

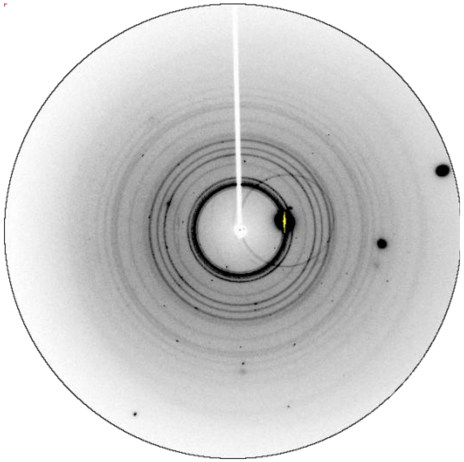

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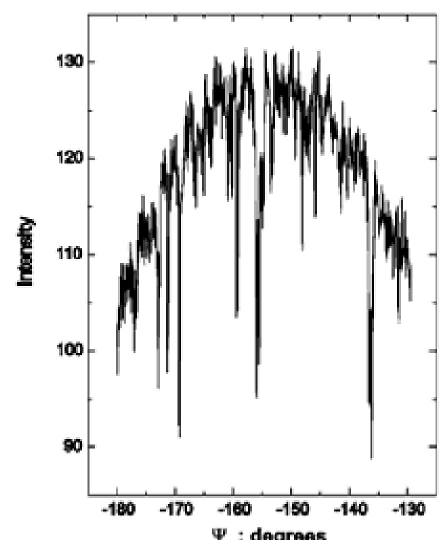

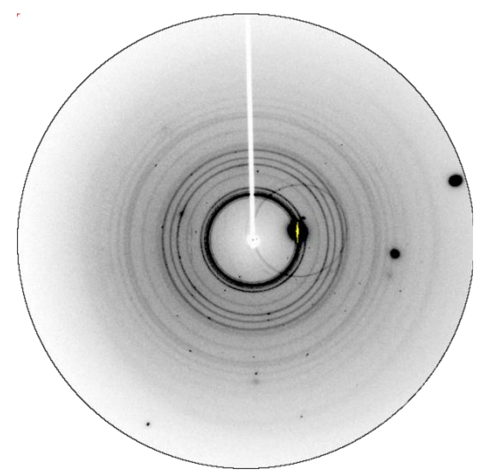

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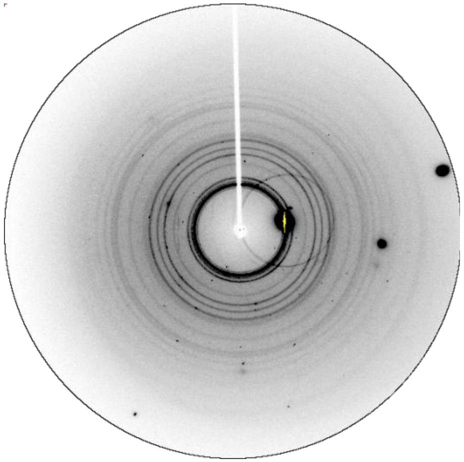

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Does the "ideal" software exist?!

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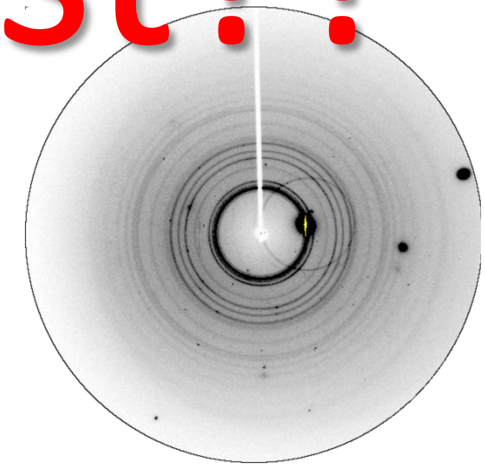

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No!

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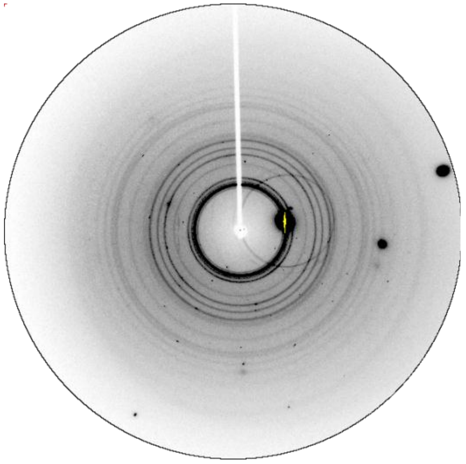


Reality check

Area X | CrysAlis Pro | BRUKER | XDS

Commercial

Free-ware

Macromolecular crystallography

Reality check

Indexing

Commercial software is very robust and handles multiple lattices very easily. Indexing with XDS can be more complicated and time consuming and involves several separate steps for each phase.

XDS

Reality check

Multiple integration of more than one lattice

Area X CrysAlis Pro BRUKER

Commercial software is very robust. XDS handles only one phase at a time.

XDS

Reality check

Shading (avoidable on 4-circle diffractometers), masking, and background

No problem with shading in the commercial software as they use cones defined by the opening angles of the DAC. It is also possible to mask diamond reflections. XDS has no possibility to exclude shadowed areas of a detector but gasket rings could be excluded using the "*ice ring*" option.

XDS

Reality check

More on the masking: possible developments

Masquerade for LT data

J.A. Coome, A.E. Goeta, J.A.K. Howard, M.R. Probert, *J. Appl. Cryst*. **45**, 292 (2012)

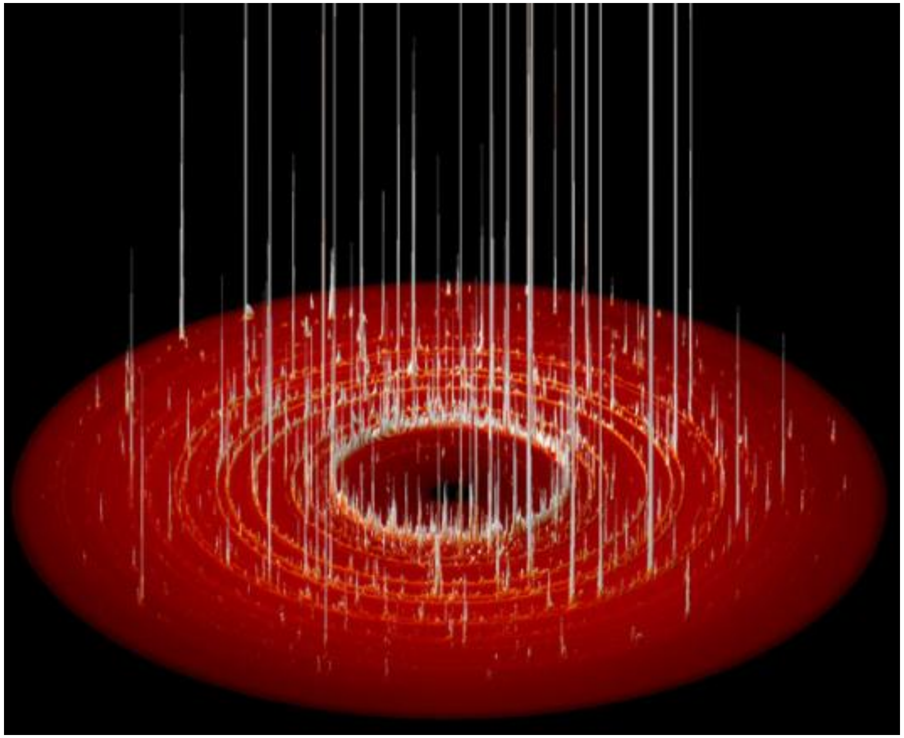
Masks corresponding to the scattering from beryllium in a cryostat
(a similar approach could be used for HP data with gasket rings)

Reality check

More on the background: possible developments

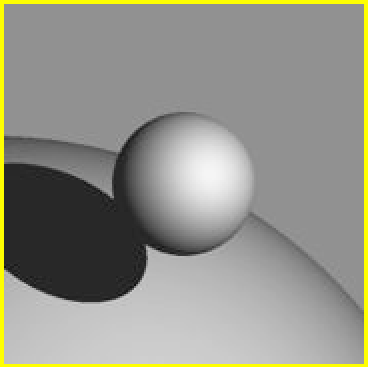
Powder3D: Automatic detection of Debye–Scherrer ellipses on the basis of the pattern recognition techniques and signal filtering using fractile statistics

B. Hinrichsen, R.E. Dinnebier, P. Rajiv, M. Hanfland, A. Grzechnik, M. Jansen
J. Phys.: Condens. Matter **18**, S1021 (2006)



Reality check

Profile fitting



Recorded spots/reflections on area-sensitive detectors are distorted due to their trajectory through the Ewald sphere. Kabsch devised a transformation from the rotation geometry to a precession-like geometry, in which all the reflections have the same profile.

XDS

W. Kabsch, *J. Appl. Cryst*. **21**, 916 (1988)
W. Kabsch, *Acta Cryst*. D**66**, 125-132 (2010)
W. Kabsch, *Acta Cryst*. D**66**, 133-144 (2010)

XDS uses the *"Kabsch"* procedure to obtain uniform 3D profiles for all reflections by transformation of the recorded spots to an undistorted reciprocal space. It also performs local profile fitting for different areas of the detector. Commercial software uses "3D" reflection boxes/masks/spots ("2D" on the detector in pixels and "1D" from the rotation angle of the crystal) to obtain an average profile or some variations of the *"Kabsch"* procedure.

Area X

CrysAlis Pro

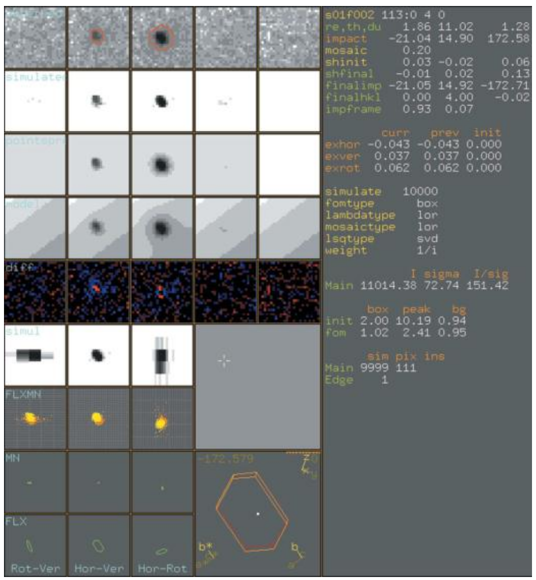
BRUKER

Reality check

A.M.M. Schreurs, X. Xian, and L.M.J. Kroon-Batenburg
J. Appl. Cryst. **43**, 70 (2010)

Profile fitting in non-commercial ***EVAL-15***

“*Fundamental approach*” to 3D profiling of single-crystal reflections to obtain individual models for each reflection based on the physical parameters of the data collection:



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Beam focus dimensions and divergence

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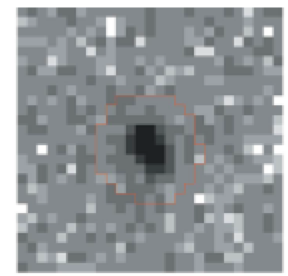
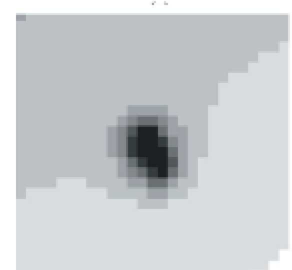
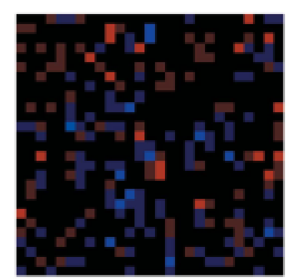
Detector point spread and spatial distortions

Background


Obs.


Calc.


Diff.

Reality check

Reciprocal space reconstruction

XArea CrysAlis Pro BRUKER

No problem in the commercial software. XDS does not make it. Non-commercial programs ***Xcavate*** (M. Estermann, ETH Zürich) and ***Map2Layer/Map2zone*** (C. Paulmann, DESY & U. of Hamburg) are able to make the reconstructions from synchrotron and laboratory data.

XDS

Reality check

Inspection of the reflections on the frames

Area X

CrysAlis Pro

BRUKER

No problem in the commercial software. In XDS, reflections have to be traced using their coordinates on the frame and the frame number.

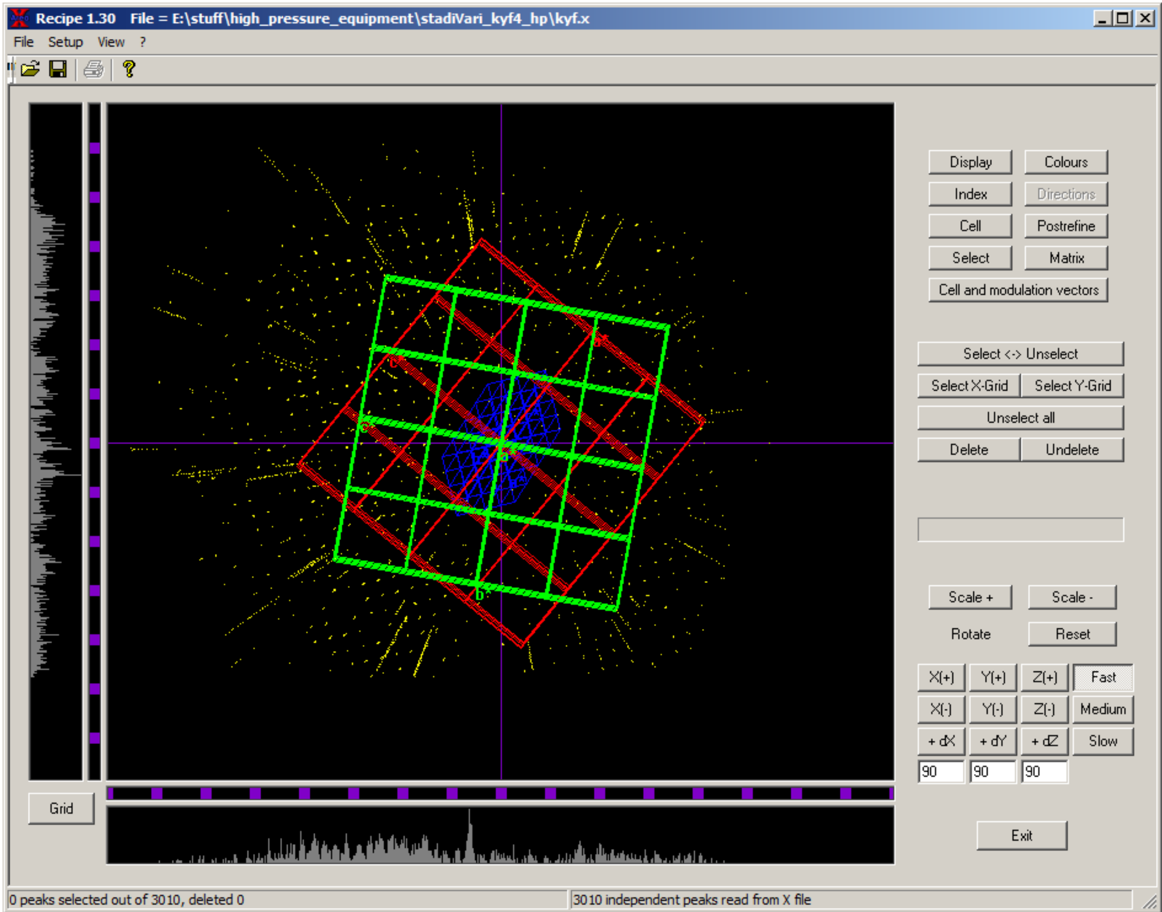
XDS

Reality check

"Diamond dips"

No software can do it.

A possible solution for "diamond dips"?



Knowing the diamond matrices, one can determine which diamond reflections are in diffraction conditions → proper scaling of the frames

Reality check results

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When used with care and understanding, each software produces excellent data. It is just necessary to know your tools very well and some little tricks.

Reality check results

When used with care and understanding, each software produces excellent data. It is just necessary to know your tools very well and some little tricks.

PS. Everybody uses what they like the best and what suits their scientific interests.

Software fayre

Bruker (Mike)

Crysalis (Clivia)

X-Area: image plates & PILATUS (Andrzej & Karen)

XDS (Andrzej & Karen)

WinIntegrstp (Ross)