# **Basics of X-Area for image plates**

Commercial software to process single-crystal and powder x-ray data

from STOE image plates and PILATUS detectors

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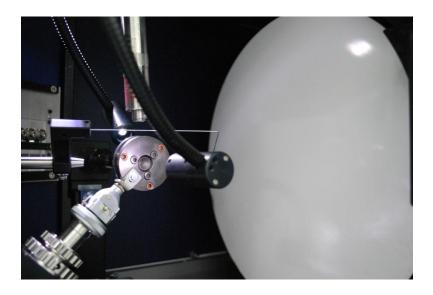
<sup>1</sup> Institute of Crystallography, RWTH Aachen, Germany

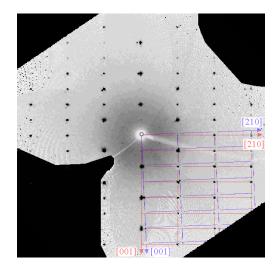
<sup>2</sup> Jülich Centre for Neutron Science, Jülich, Germany

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# Crystallographic problems that X-Area handles without any problems

- twinning
- modulated structures
- composites
- polytypism
- high-pressure data

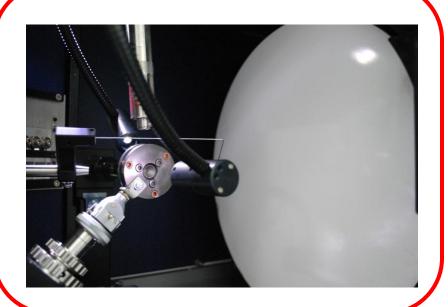


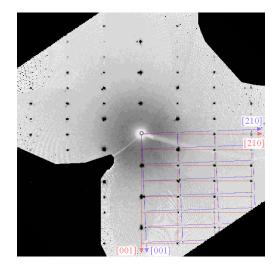




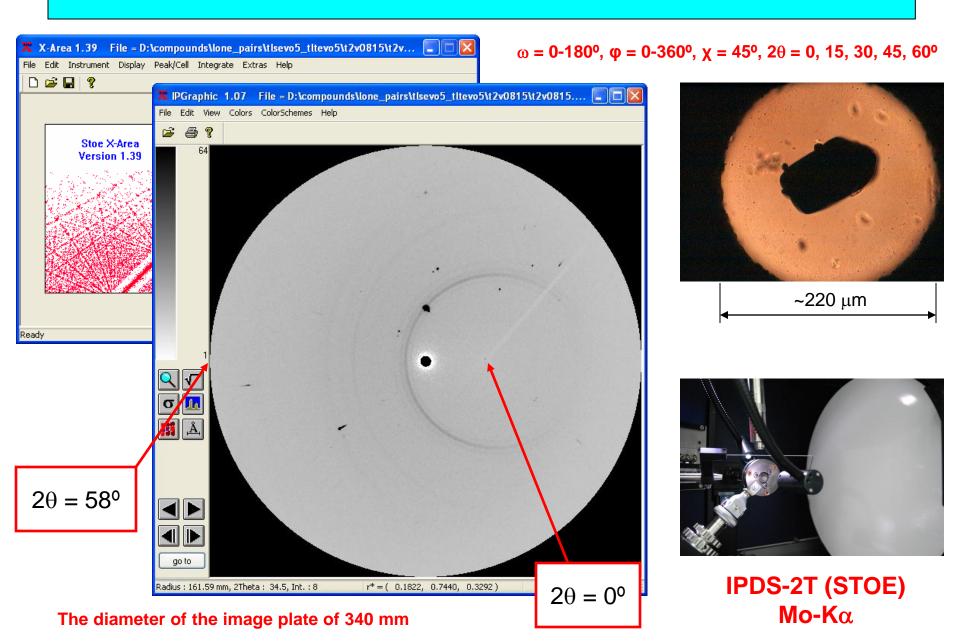
# Crystallographic problems that X-Area handles without any problems

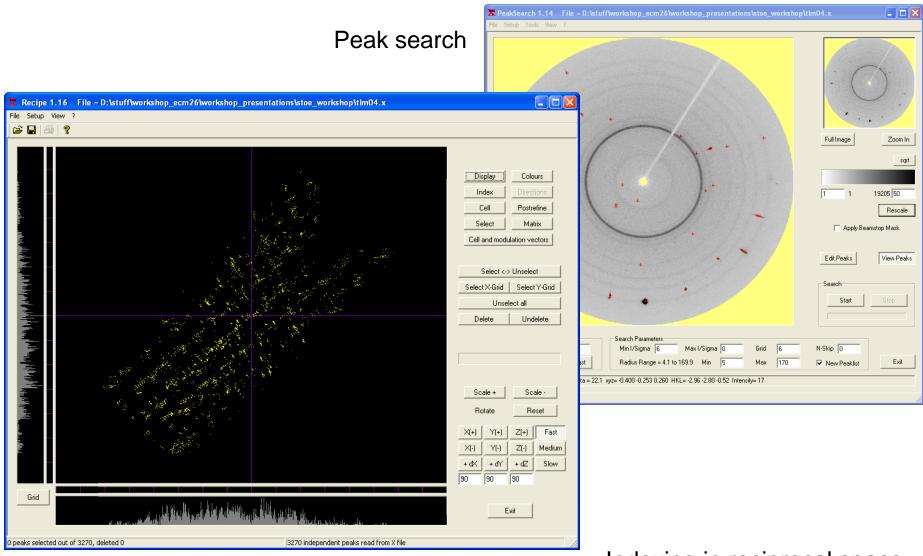
- twinning
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- polytypism
- high-pressure data



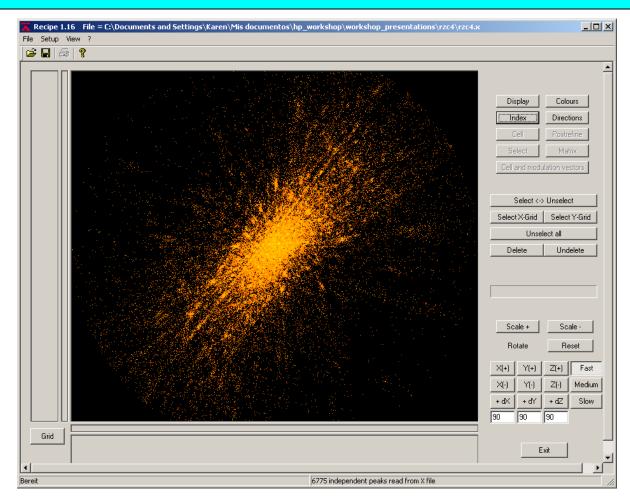




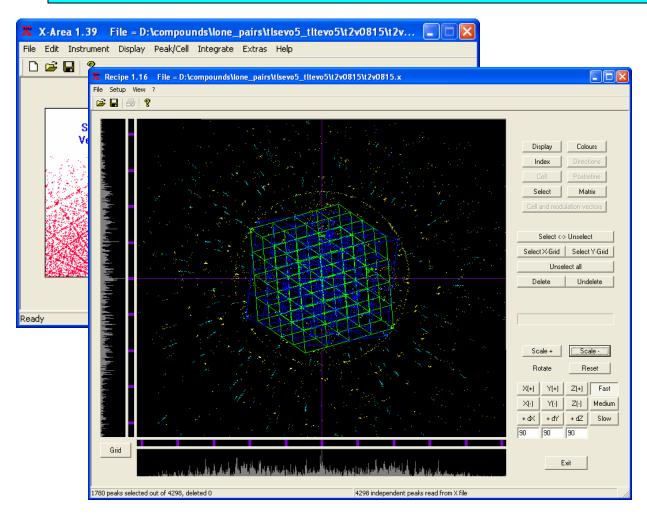




Indexing in reciprocal space



Using the currently selected peaks all difference vectors between all peak positions are calculated, normalised, and projected onto the horizontal plane of the Ewald sphere. When the crystal is a *true* single crystal, a series of sharp "lines" can be seen. Each pixel represents a direction in the reciprocal space. The picture is colour coded, the brighter the pixel the larger the frequency of difference vectors in that direction. Each "line" corresponds to a set of parallel, equally spaced layers in the reciprocal space. In the case of the high-pressure data, the "lines" from the crystal, two diamonds, and grainy spots of the gasket rings are superimposed.



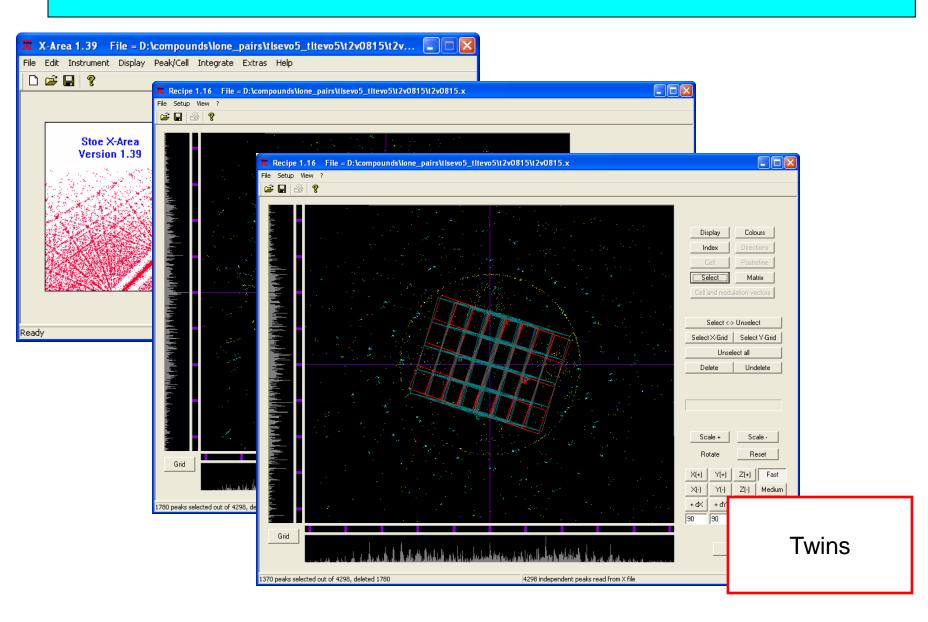
The diamond orientation matrices are useful to check the alignment. They could be used for any other measurements in the same DAC. Hence, it is enough to find them once.



Diamond  $\lambda/2$  reflections

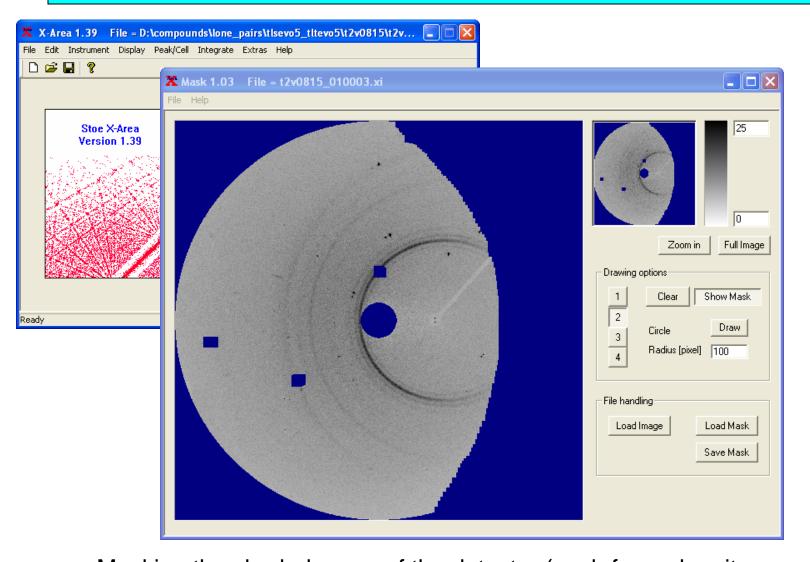
A contamination by the "half-wavelength" component of the Mo spectrum from a tube

both in a crystal-monochromated beam and in a filtered beam.

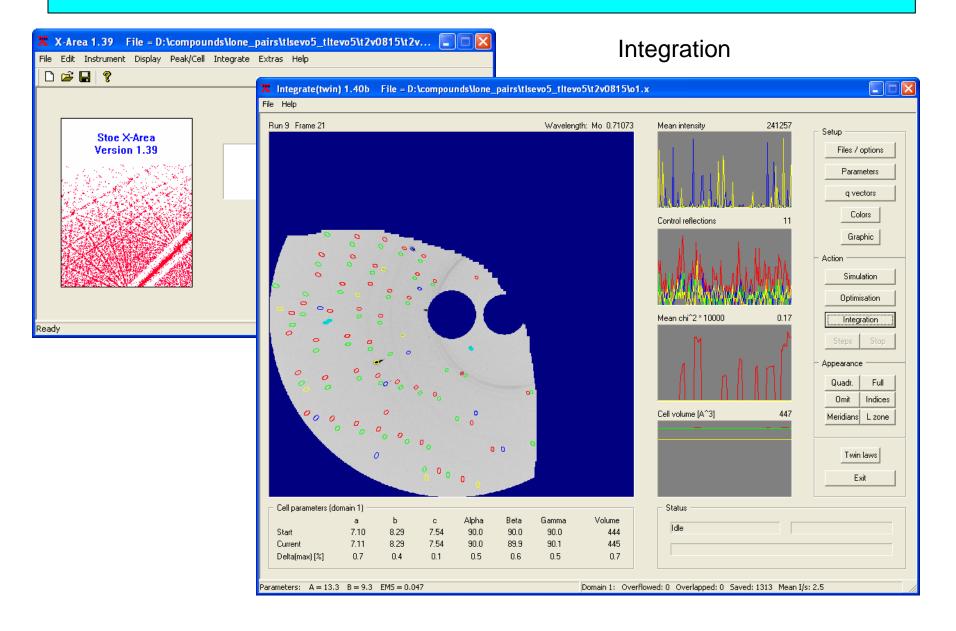


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	Domain 2	diamond_1	Browse	diamond_1	Browse	Graphic
	Domain 3	diamond_2	Browse	diamond_2	Browse	Action
	Domain 4		Browse		Browse	Simulation
						Optimisation
	⊢ Switches =					Integration
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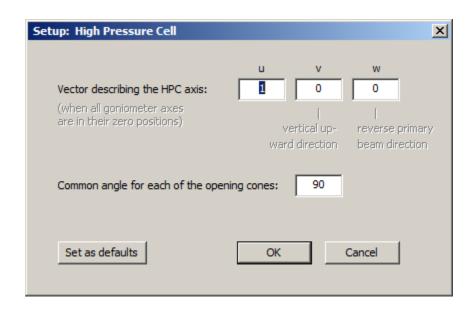
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			Files / options
	Setup: Files / options		Parameters
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	x file(s)	hkl file(s)	Colors
	Domain 1 E:\compounds\lufe2o4\hp_lufe2o4\lfo_E Browse	e2o4\hp_lufe2o4\lfo_5a\average_5a.hkl Browse	
	Domain 2 diamond_1 Browse	diamond_1 Browse	Graphic
	Domain 3 diamond_2 Browse	diamond_2 Browse	Action
	Domain 4 Browse	Browse	Simulation
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	r Switches		Integration
	Use filter frames Apply q vectors	Select runs / frames	Automatic
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Masking the shaded areas of the detector (each frame has its own mask) The masks could be re-used for any other data collected in the same DAC. Hence, it is enough to determine them once. The strong diamond reflections could be masked this way.



Integrate(twin) 1.57.2 File = E:\compounds\lufe2o4\hp_lufe2o4\lfo_5a\ave File Help	erage_5a.x	
No frame loaded	Mean intensity	Setup Files / options
Domain 2   diamond_1   E     Domain 3   diamond_2   E	hkl file(s)         Browse       e2o4\hp_lufe2o4\lfo_5a\average_5a.hkl       Browse         Browse       diamond_1       Browse         Browse       diamond_2       Browse         Browse       Browse       Browse         stors       T       T         ng masks       Control reflections       50         Minimum number       50         Maximum runsber       200	
Cell parameters (domain a b c Alpha Beta Gamma Start Current Delta(max) [%] Parameters: A = 14.0 B = 4.0 EMS = 0.010	Volume	Shading cone instead of masks

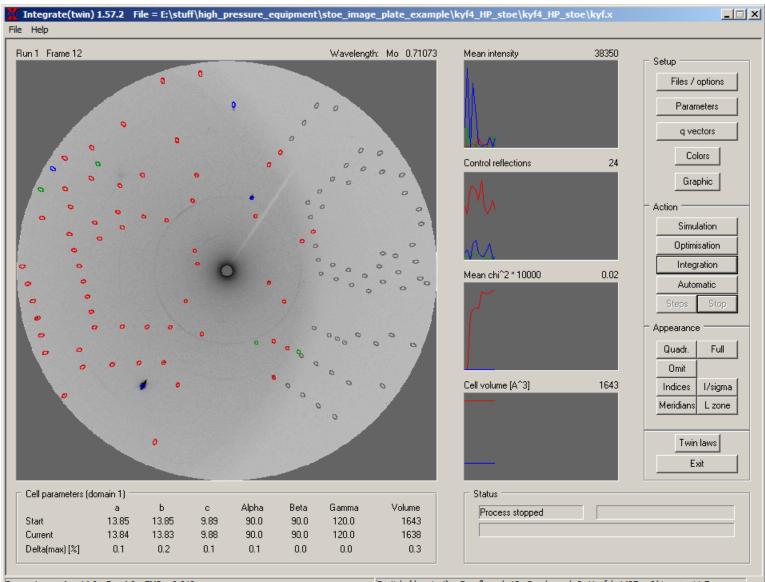


The coordinate system *uvw* is centered on the goniomter:

the vector v is directing upwards vertically, w is directing to the centre of the collimator, and u is oriented in a way that a right-handed Cartesian coordinate system is built.

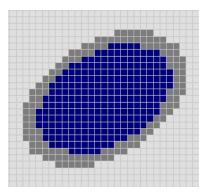
The cone is defined by the vector relating the position of the DAC with respect to the direct beam and the opening angle. When the DAC is perpendicular to the beam at  $\omega = 0^{\circ}$ ,  $\varphi = 0^{\circ}$ , and  $\theta = 0^{\circ}$ , the vector is (1 0 0). For  $\varphi = 90^{\circ}$ , it is (-1 1 0).

The reflections marked in grey on the following picture are the ones that are outside of the cone.



Parameters: A = 14.0 B = 4.0 EMS = 0.010

Partials (domain 1): Overflowed: 12 Overlapped: 2 Useful: 1497 <I/sigma >: 11.7



#### **Integration masks**

Elliptical masks are used to integrate peak and background intensities. For each mask the smallest diameter is given by  $W = A + B \tan\theta$ , the largest diameter is defined by  $W / \cos(2\theta) + \Delta\lambda/\lambda \tan\theta$  (oblique incidence at higher 2 $\theta$  angles and  $\alpha$ 1- $\alpha$ 2 splitting). For obtaining the peak intensity the inner area of the mask is used. For determining the background, the pixels at the border of the ellipse are taken.

**EMS** means *effective mosaic spread* and combines the divergence of the primary beam with the mosaic spread of a crystal. For a given instrument setup (the X-ray source, monochromator, and collimator) the beam divergence is constant. However the mosaic spread varies from crystal to crystal, so that **EMS** should be determined for each measured crystal. **EMS** determines how long a reflection is in the reflecting position when it passes through the surface of the Ewald sphere.

#### The default parameters are A = 14, B = 4, and EMS = 0.01

<pre>Integrate(twin) 1.57.2 File = E:\stuff\high_pressure_equipmer File Help</pre>	nt\stoe_image_plate_example\kyf4_HP_stoe\kyf4_HP_stoe\kyf.x
Run 1 Frame 12	Wavelength: Mo 0.71073 Mean intensity 38350 O O O O O O O O O O O O O O O O O O O
Mode Mode Mode Mode C Semi-automatic C Semi-automatic	Coefficient for profiles)     EMS (effective mosaic spread)     O.02     Automatic
Frames Frames Frames C Single frame	Run: 1 Frame: 1 Quadr. Full Omit Indices I//sigma
e Logfile *.lop	Weights Start Cancel
Ceir parameters (domain 1)         a         b         c         Alpha         Beta           Start         13.85         13.85         9.89         90.0         90.0           Current         13.84         13.83         9.88         90.0         90.0           Delta(max) [%]         0.1         0.2         0.1         0.1         0.0	Gamma Volume Process stopped
Parameters: A = 14.0 B = 4.0 EMS = 0.010	Partials (domain 1): Overflowed: 12 Overlapped: 2 Useful: 1497 <i sigma="">: 11.7</i>

#### Automatic optimization of A, B, and EMS: multiple frames 3-7

It is a very tricky procedure in the case of large overlap of crystal reflections with diamond reflections and gasket rings. You can't use it without having a proper cone or masks for shaded areas of the detector.

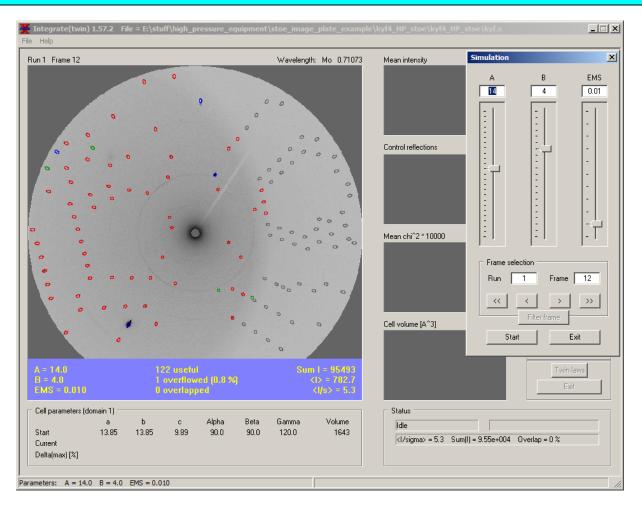
You should never fully trust what this procedure gives you. Visual inspection of the results is imperative.

<b>X Integrate(twin) 1.57.2</b> File = E File Help	:\stuff\high_pressure_e	uipment\stoe_image_plate_example\kyf4_HP_stoe\kyf4	_HP_stoe\kyf.x
Run 1 Frame 12	0 p: Automatic processing	Wavelength: Mo 0.71073 Mean intensity	38350 Files / options Parameters
0		Overall control	q vectors
0 0 0	Cydes 📱	Increment factor 0.5 Frames per trial	30 Graphic
	Values/cycle		Action
0 0	A 3	8 16 4.0	1.0 Automatic
0 0 0	B 3	0 4 2.0	0.5 Appearance
0 0 1	EMS 3	0.008 0.024 0.008	0.002 Quadr. Full Omit
0	Trials 81		Indices I/sigma Meridians L zone
	Set as defaults	Start Cancel	Twin laws Exit
Cell parameters (domain 1) — a b		Beta Gamma Volume Process stopped	
Start 13.85 13. Current 13.84 13. Delta(max) [%] 0.1 0.	83 9.88 90.0	90.0 120.0 1643 90.0 120.0 1638 0.0 0.0 0.3	
Parameters: A = 14.0 B = 4.0 EMS	= 0.010	Partials (domain 1): Overflowed: 12 Ove	rlapped: 2 Useful: 1497 <i sigma="">: 11.7</i>

#### Automatic optimization of A, B, and EMS: searching for the lowest R<sub>int</sub>

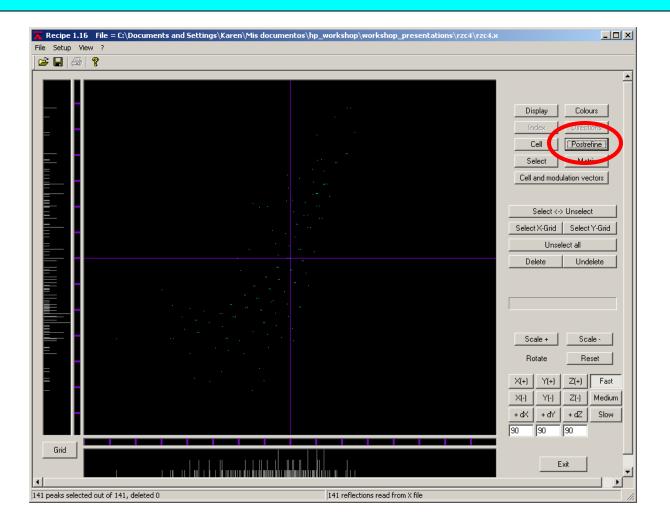
It is a very tricky procedure in the case of large overlap of crystal reflections with diamond reflections and gasket rings. You can't use it without having a proper cone or masks for shaded areas of the detector.

You should never fully trust what this procedure gives you. Visual inspection of the results is imperative.

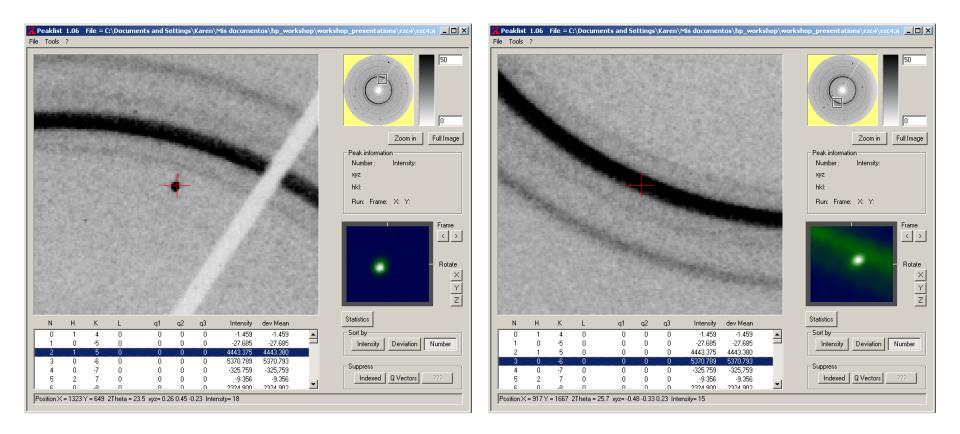


#### Simulation of the integration masks

Usually the A, B, and EMS parameters for a good crystal of an inorganic solid in a DAC hardly ever are larger than 20, 6, and 0.04, respectively. If any of these parameters is bigger, there must be something wrong with your measurement and/or crystal.



**Postrefine** – refinements of the orientation matrices of the crystal and two diamonds after integration



Each reflection could be inspected on the frames in the case you have doubts about its intensity during data reduction and analysis.

BuildSpace 1.31 File = D:\compounds\(nh4)2v3o8\n06_ip	
Runs / frames Available 8 run(s), 177 frames Select Selected 8 run(s), 177 frames	Building and viewing X ViewSpace 1.07 File = D:\compounds\(nh4)2v3o8\n06_ipds2t\
Processed	File Edit Help
Conditions for 3D array [A^-1]         XYZ Min.       XYZ Max.         Increment         Increment         Test         Sphere by frames         2theta(max)         43.4         Pixels (1D)         2267         d*(max) [A^-1]         Subpixels (XY)         Subpixels (XY)         Subpixels (omega)	
Start Interrupt Exit	· · · · · · · ·
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Reconstruction of reciprocal space	

Ready

g reciprocal space

Full Image

Increment 0.005

▼ Use OM

Point 000

10

**Rescale Colours** 

Exit

Square root scale

Zoom In

XZY Max

Distance

Vector 2

Colour Scale

0

1

1

XZY Min

Direction

Vector 1

Max, XY

Build Plane

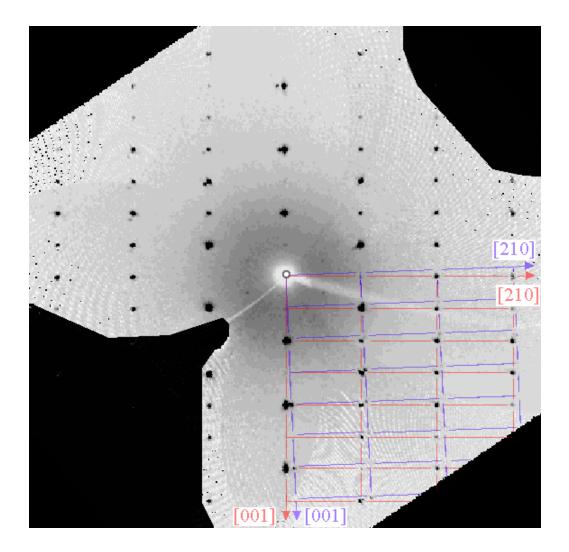
Setup Colours

001 C

-1

on the basis of the measured frames

Intensity = 0 XYZ = 0.4700 0.4819 -0.8418 2Theta = 45.0 HKL = 4.283 -8.839 0.000



# Twinning at high pressures

in the reciprocal space

Some parts of the text and one figure were taken from the X-Area manual written by STOE. It is worth spending some time on reading it carefully. X-area is not a black-box software and requires conscious actions from the user.