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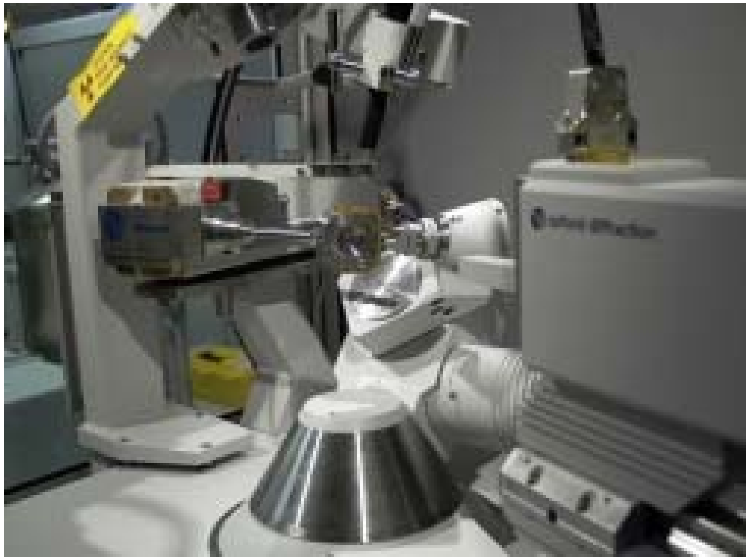
DAC data collection and reduction, Oxford Diffraction Instruments

http://www.oxford-diffraction.com/

Xcalibur ™ CCD



Xcalibur ™ PD

Gemini ™

- *Support for up to 2Kg on the phi axis*
- *Better than 10 micron sphere of confusion*

SeperNova ™

Hi-flux dual wavelength micro-focus
Mo & Cu X-ray sources

Oxford Diffraction diffractometers driving program: CrysAlis^{Pro} ™

CrysAlisPro is accessible either via a graphical user interface or by a command line interface and can be operated under fully automatic, semi-automatic or **completely manual control**. A typical experiment is conducted in three stages:

1) **Automatic crystal screening** - A short pre-experiment of <5 minutes evaluates the crystal quality, providing unit cell and best exposure time information

2) **Strategy computation** - Sophisticated automatic strategy software calculates optimal conditions for fast, high quality, complete data collection

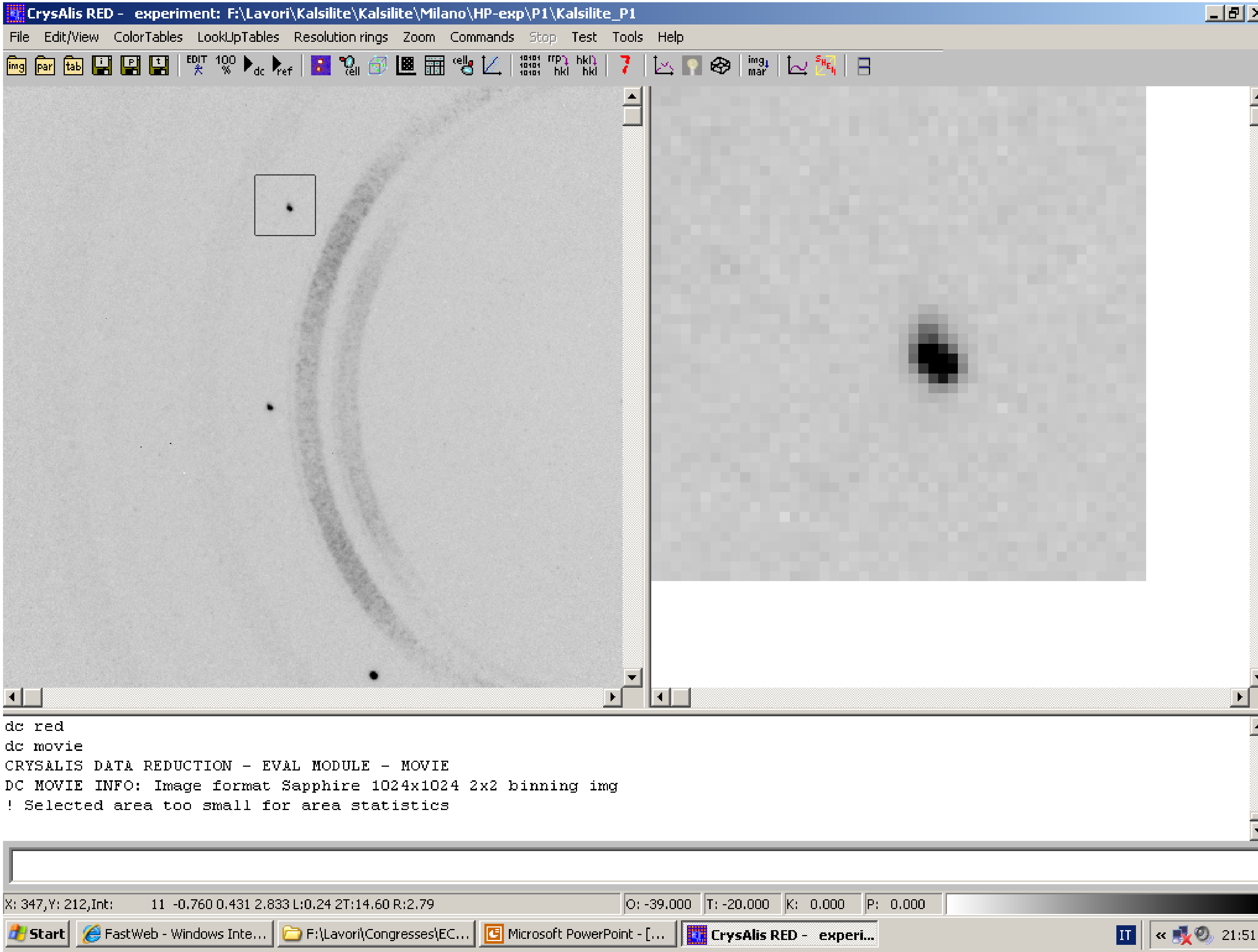
3) **Data collection and concurrent data reduction -** As data is collected, intelligent routines tune the parameters to give the best quality integrated data

http://www.oxford-diffraction.com/

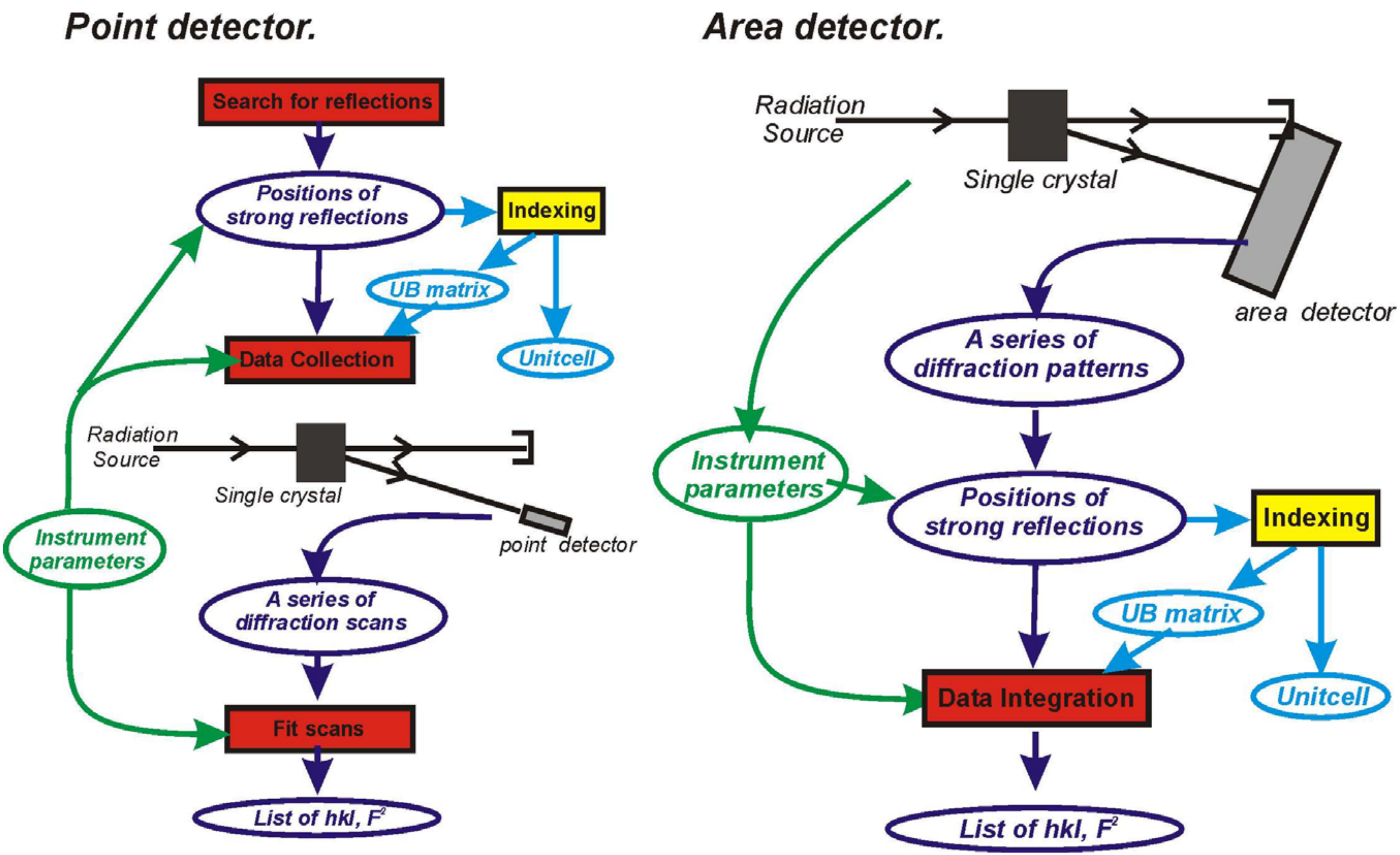
CrysAlisPro also provides several specialist tools for dealing with non-standard and problematic crystals. These include:

* Movie based face indexation absorption correction
* Advanced unit cell finding
* Reciprocal space viewer
* Twinning
* Incommensurates
* High Pressure
* Powders

CrysAlisPro outputs data in HKLF format and interfaces directly with OLEX2, SHELX and third party data reduction packages including MOSFLM and XDS. CrysAlisPro is provided under a multi-site, multi-user licence.


CrysAlis RED - experiment: F:\Lavori\Kalsilite\Kalsilite\Milano\HP-exp\P1\Kalsilite_P1
File Edit/View ColorTables LookUpTables Resolution rings Zoom Commands Stop Test Tools Help
dc red
dc movie
CRYSALIS DATA REDUCTION - EVAL MODULE - MOVIE
DC MOVIE INFO: Image format Sapphire 1024x1024 2x2 binning img
! Selected area too small for area statistics
X: 347,Y: 212,Int: 11 -0.760 0.431 2.833 L:0.24 2T:14.60 R:2.79
O: -39.000 T: -20.000 K: 0.000 P: 0.000
Start FastWeb - Windows Inte... F:\Lavori\Congresses\EC... Microsoft PowerPoint - [... CrysAlis RED - experi...
IT 21:51

Point detector *vs.* CCD experiments

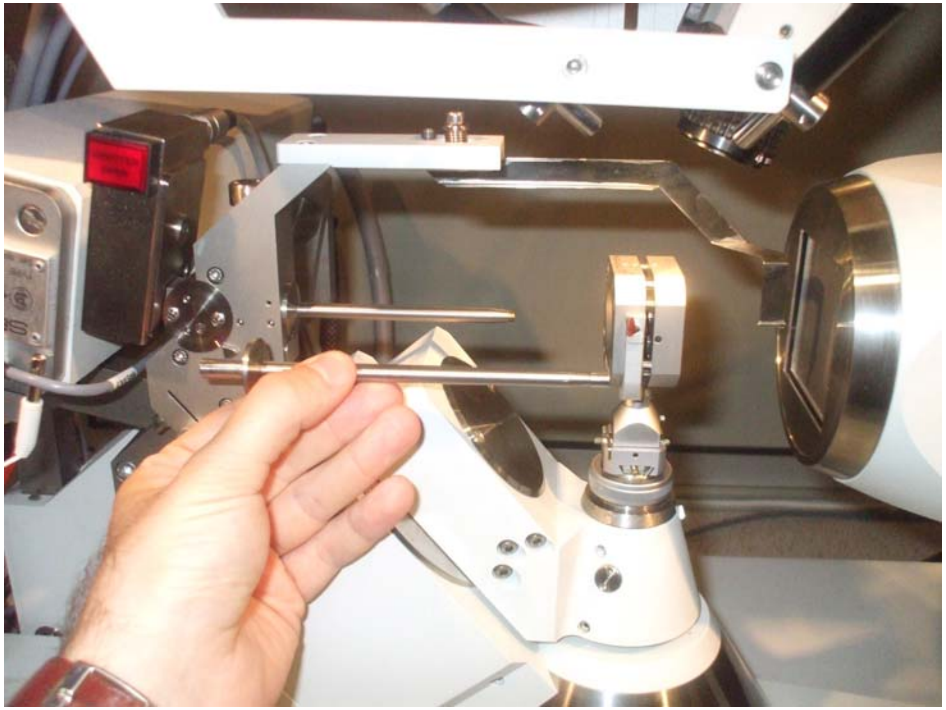


RJA's presentation

Step 1: Diffractometer set-up and physical alignment of DAC

- A short collimator and a modified beam-stop for high-pressure experiment.

- Suggested sample-detector distance of 80 mm (70 minimum for ETH-type DAC)

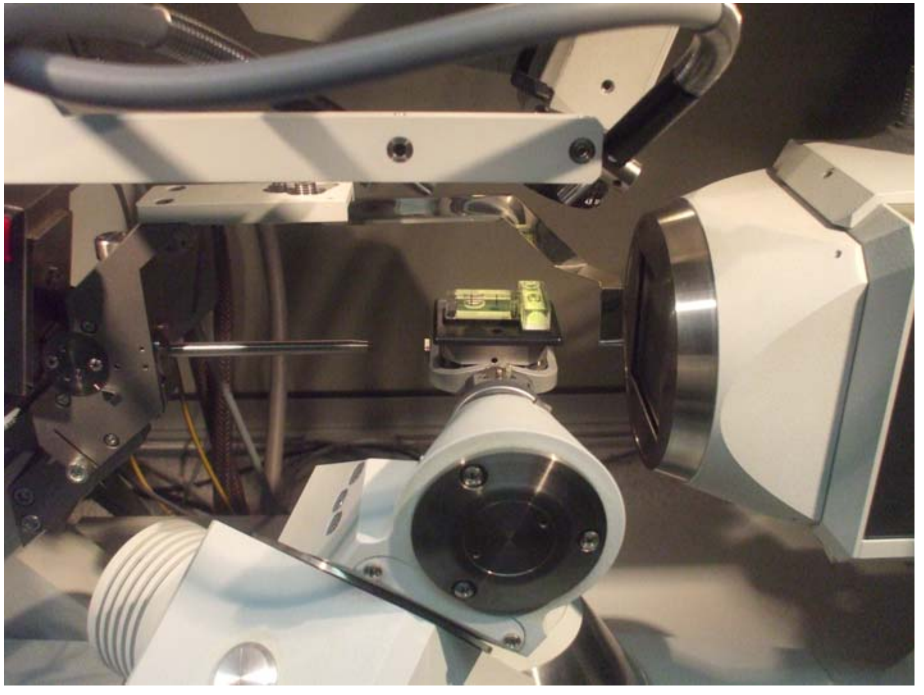


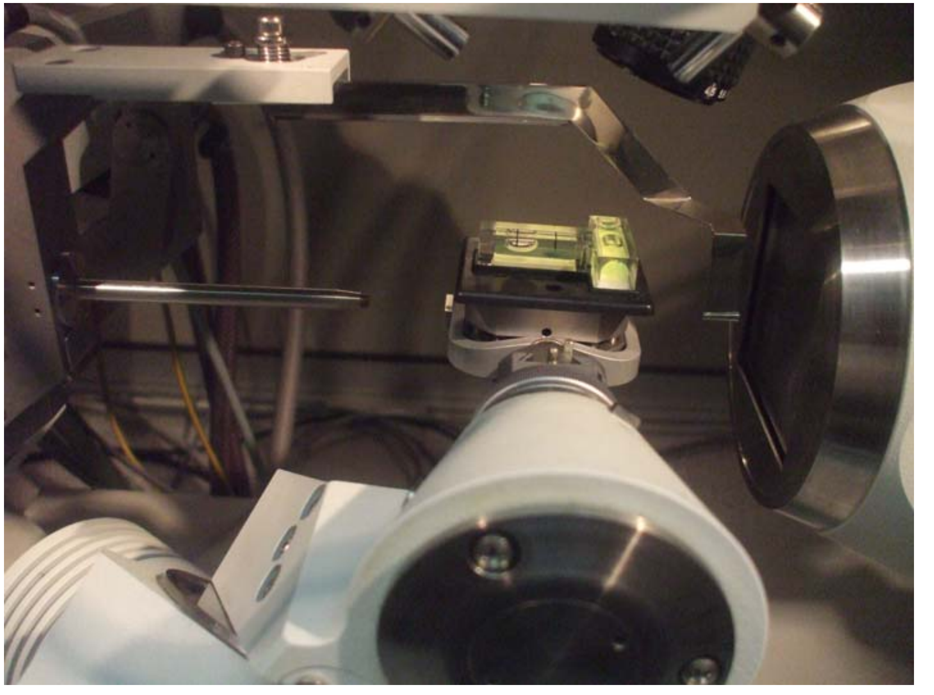
Step 1: Physical Alignment of DAC

Oxf Diffr diffractometers running program: CrysAlis^{Pro} ™

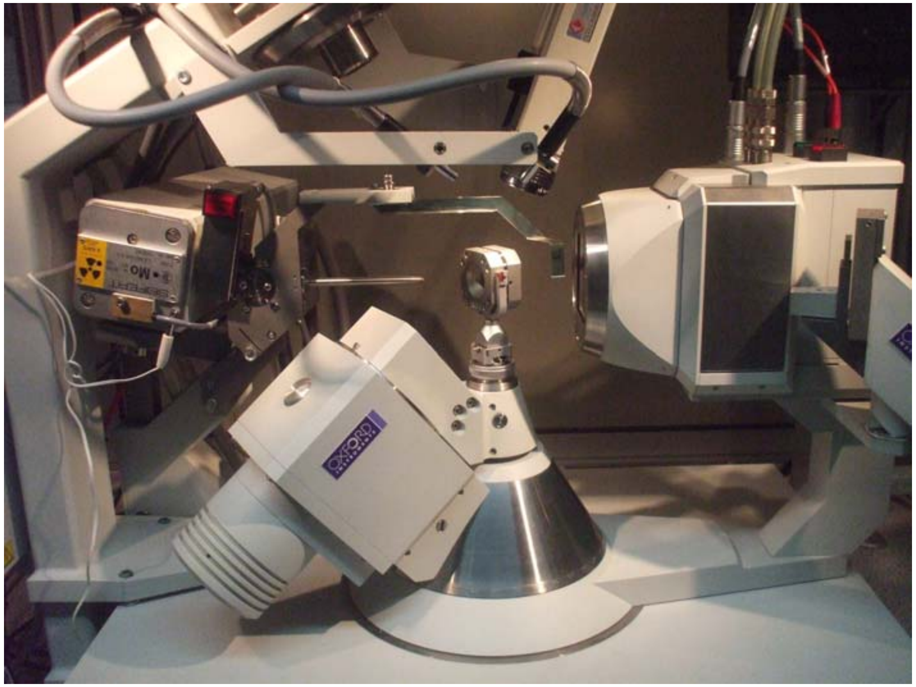
Operation	Command/Action
Switch to HP mode	sw s **2**
Set max Ψ angle	sw a 40
Drive the diffractometer to zero	gt a 0 0 0 0
Load DAC onto diffractometer. Tighten the base screw firmly.	
Align the DAC by eye perpendicular to the beam	Loosen the locking screw for the height adjustment on the goniometer head and rotate the cell until it looks perpendicular to the beam direction.
Accurately align the DAC perpendicular to the beam. (next figure)	**gt e 0 0 90 90** Rotate the DAC until the face of the DAC is exactly horizontal, as measured by a spirit level. Gently tighten the height locking screw on the goniometer head.
Set focus of video microscope, and cell translation along beam	**F12** View image of cell.
Set translation of DAC	**Spin cell by 180 on phi** And compare positions of image of gasket hole. Move the cell and repeat until the center of the gasket hole is in the same place before and after rotation by 180 deg.
Set height of DAC	**Lower position** Observe position of gasket hole centre on video screen. **Upper position** Compare position of gasket hole and adjust height. Repeat until image of gasket hole does not move vertically between these two positions. Tighten height locking screw.

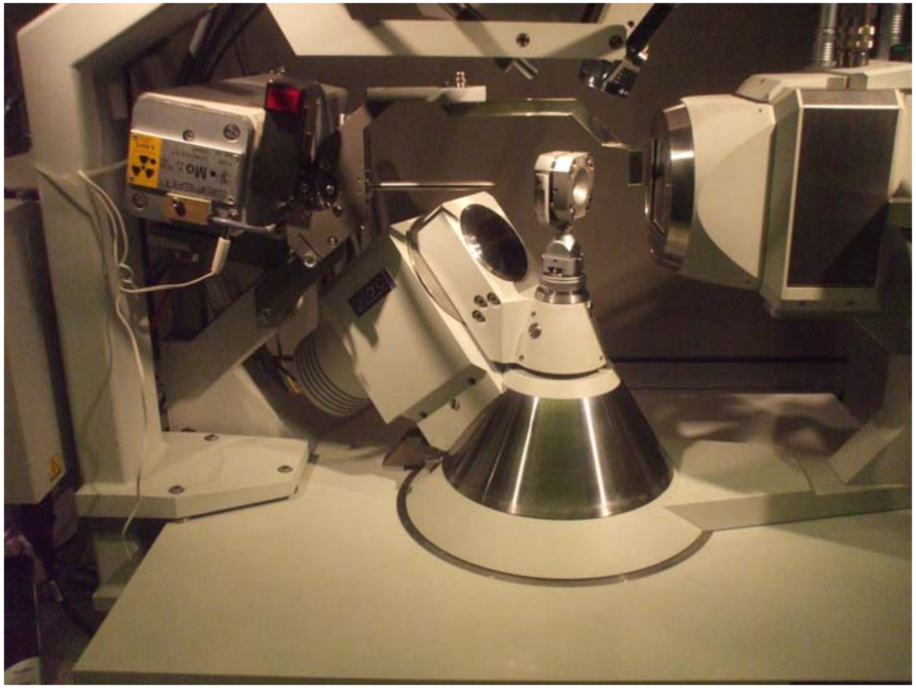
Alignment of the DAC perpendicular to the beam





Use transmitted beam to align cell along beam as follows	
Drive goniometer to zero	**gt a 0 0 0 0**
Reduce generator power to 28 KV and 1mA	
Expose CCD with rotated cell	**gt o –30** **card raw 0.1 on**
Repeat with opposite omega	**gt o 30** **card raw 0.1 on**
Adjust cell along beam until the transmitted spot is in the same position for both ω = +30 and ω = -30.	
Install the long beam stop and align it with the direct beam transmitted through the DAC.	**gt a 0 0 0 0** **card raw 1 on** Repeat with higher generator power up to operating conditions.





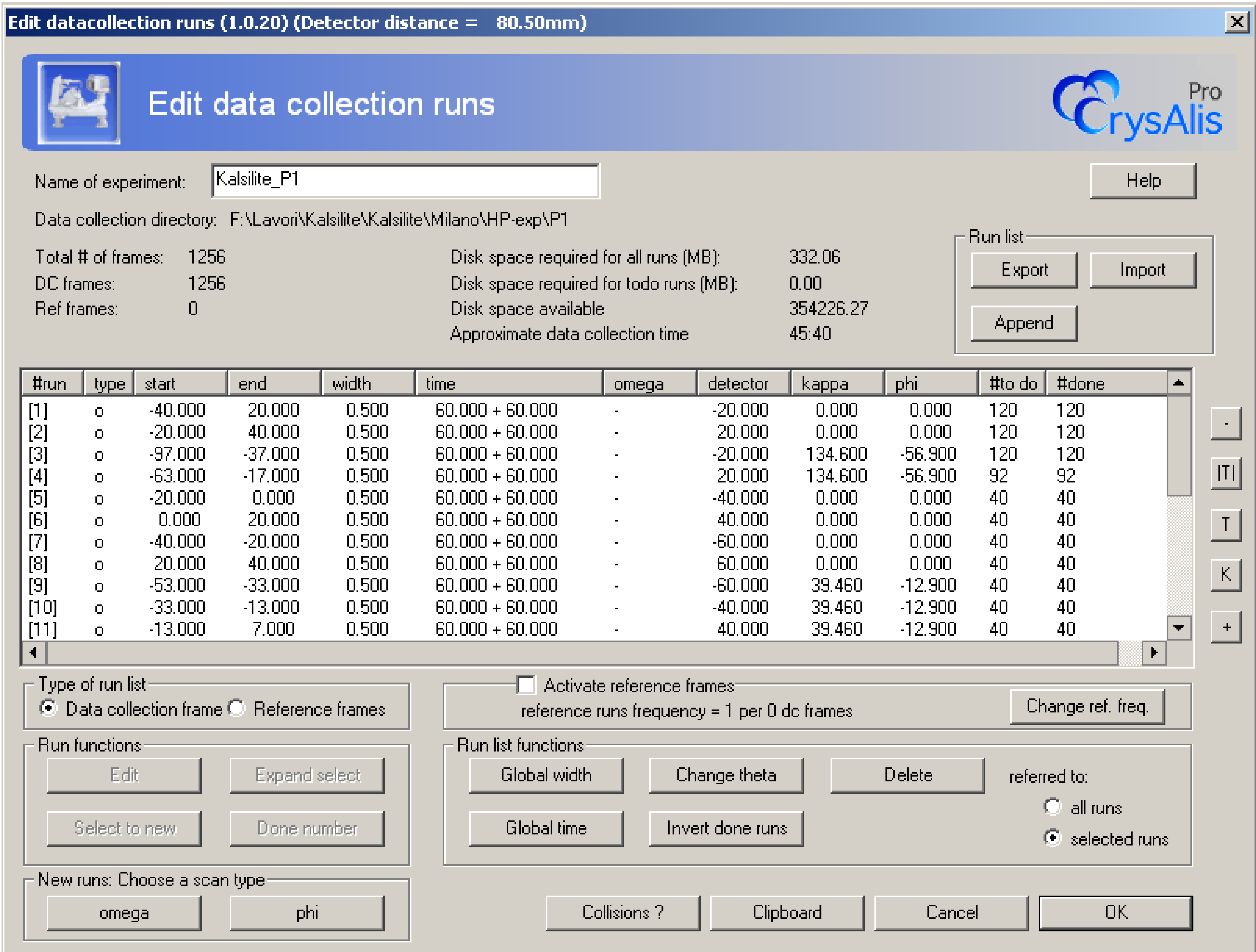
Step 2: Pre-designed run files

We have designed run files for data collection with the Sapphire-3 CCD set at *dd*=80mm and a DAC with a half-opening angle of 40 degrees:

DAC_psi40_dd80_tth60_full_sapphire3.run
DAC_psi40_dd80_tth80_full_sapphire3.run

Both run files attempt to cover all of accessible reciprocal space. If only one-half of that space is required, then the runs at negative values of 2-theta can be deleted.

Step 2: Pre-designed run files



#run	type	start	end	width	time	omega	detector	kappa	phi	#to do	#done
[1]	o	-40.000	20.000	0.500	60.000 + 60.000	-	-20.000	0.000	0.000	120	120
[2]	o	-20.000	40.000	0.500	60.000 + 60.000	-	20.000	0.000	0.000	120	120
[3]	o	-97.000	-37.000	0.500	60.000 + 60.000	-	-20.000	134.600	-56.900	120	120
[4]	o	-63.000	-17.000	0.500	60.000 + 60.000	-	20.000	134.600	-56.900	92	92
[5]	o	-20.000	0.000	0.500	60.000 + 60.000	-	-40.000	0.000	0.000	40	40
[6]	o	0.000	20.000	0.500	60.000 + 60.000	-	40.000	0.000	0.000	40	40
[7]	o	-40.000	-20.000	0.500	60.000 + 60.000	-	-60.000	0.000	0.000	40	40
[8]	o	20.000	40.000	0.500	60.000 + 60.000	-	60.000	0.000	0.000	40	40
[9]	o	-53.000	-33.000	0.500	60.000 + 60.000	-	-60.000	39.460	-12.900	40	40
[10]	o	-33.000	-13.000	0.500	60.000 + 60.000	-	-40.000	39.460	-12.900	40	40
[11]	o	-13.000	7.000	0.500	60.000 + 60.000	-	40.000	39.460	-12.900	40	40

Step 3: Data collection

1. Enter **ccd skipremeasure 1** to prevent remeasuring on diamond reflection overflow.
2. Check the correct detector distance is set in Tools|Options !

3. Enter **dc s**.

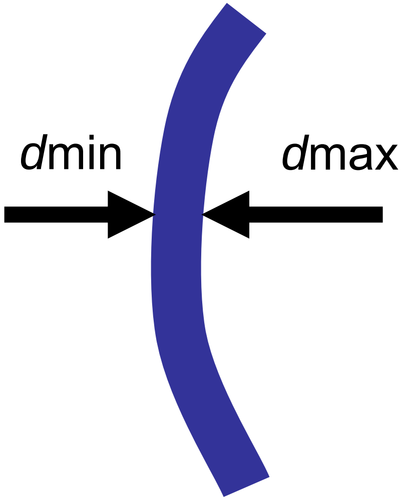
A critical parameter to be checked (first frames):
the exposure time per frame!

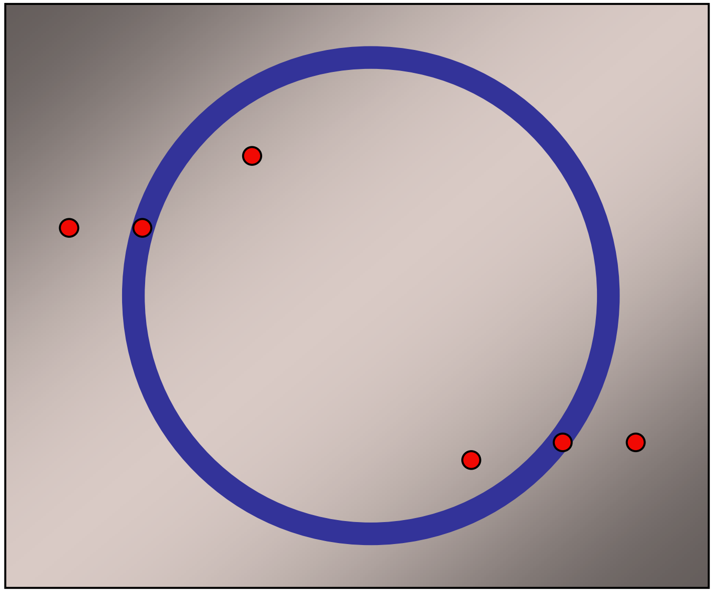
Step 4: Data Integration

1. Start the ***Reduce*** software.
2. Check that the correct high-pressure parameter file is loaded (Tools|Setup File).
3. Turn on DAC mode: **sw s 2** and **sw a Ψmax**. This prevents the software from attempting to search or integrate at peak positions that are obscured by the DAC.
4. Limits to the areas to be searched for peaks with **ph s** can be controlled with the **um skip** commands:

   um skipd dmax dmin prevents peak searching between dmax and dmin

5. Read the necessary d-spacings off some images. Set a skip region for 999.0 down to slightly longer than your unit-cell.
6. Run **ph s**. Use background subtraction with 5,5.

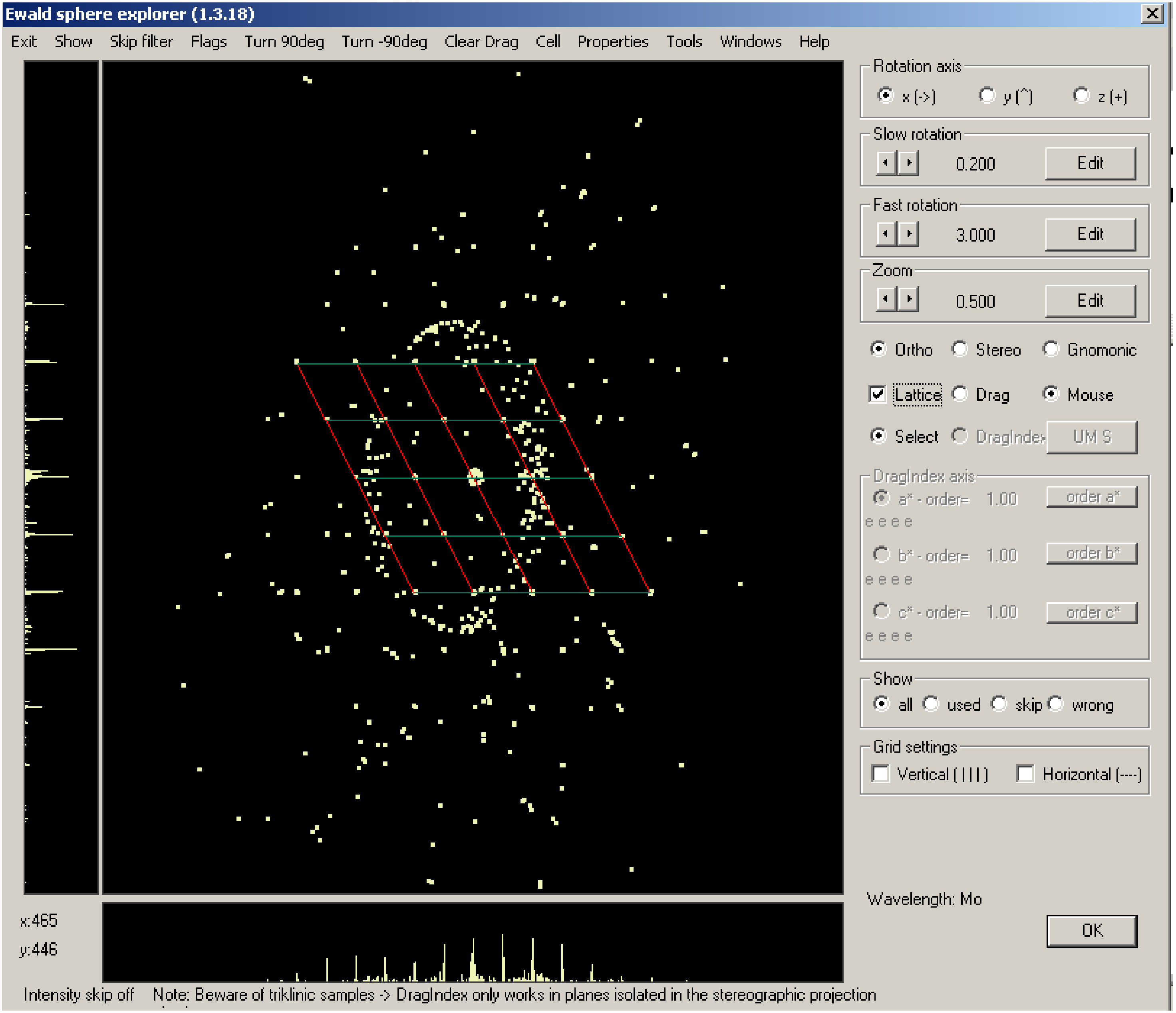




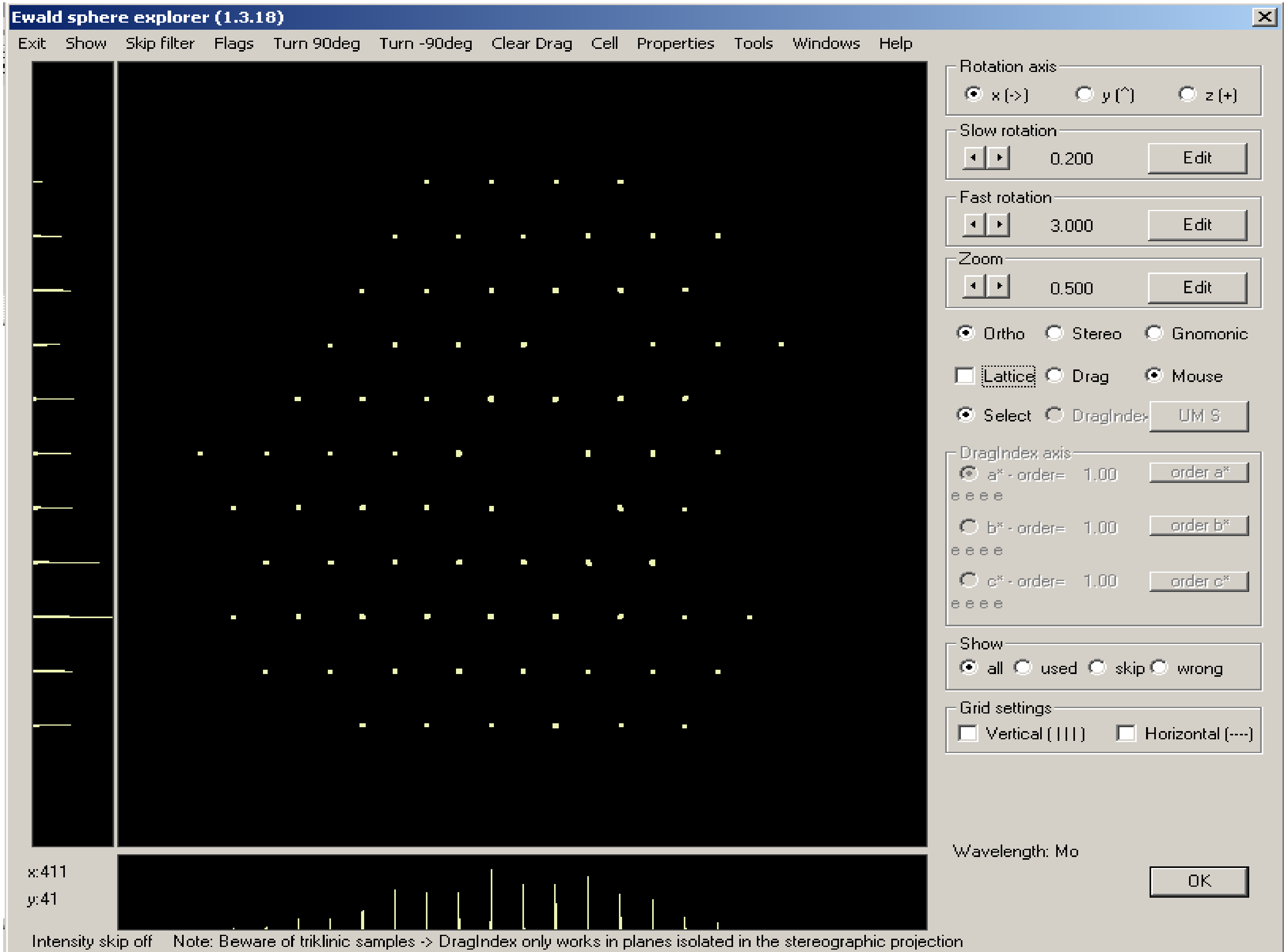
7. Use **pt e** to delete the strongest reflections (usually diamonds). Use **pt ewald** to inspect the peak list. Remove obvious Be rings etc.
8. Attempt indexing: **um searchcell** or **um ttt**. [crystal+calibrant]
9. Run **dc red** (or **dc proffit**):
    a. In step 4, set the background evaluation to 10,5.
    b. In step 5, set the DAC opening angle (in skip filters), the 2theta limit, and set *use background LS plane* (in peak finding).
    c. In step 6, switch off outlier rejection.
    d. In step 7, select the option to produce Shelx direction cosines on the output file.
10. Use **Absorb** and **Average** to correct the intensities for the effects of the DAC, and refine the structure!

http://www.crystal.vt.edu/crystal/software.html
by Ross Angel

Diffraction data of kalsilite (and more!) in the DAC at about 0.2 GPa



Kalsilite diffraction pattern at about 0.2 GPa



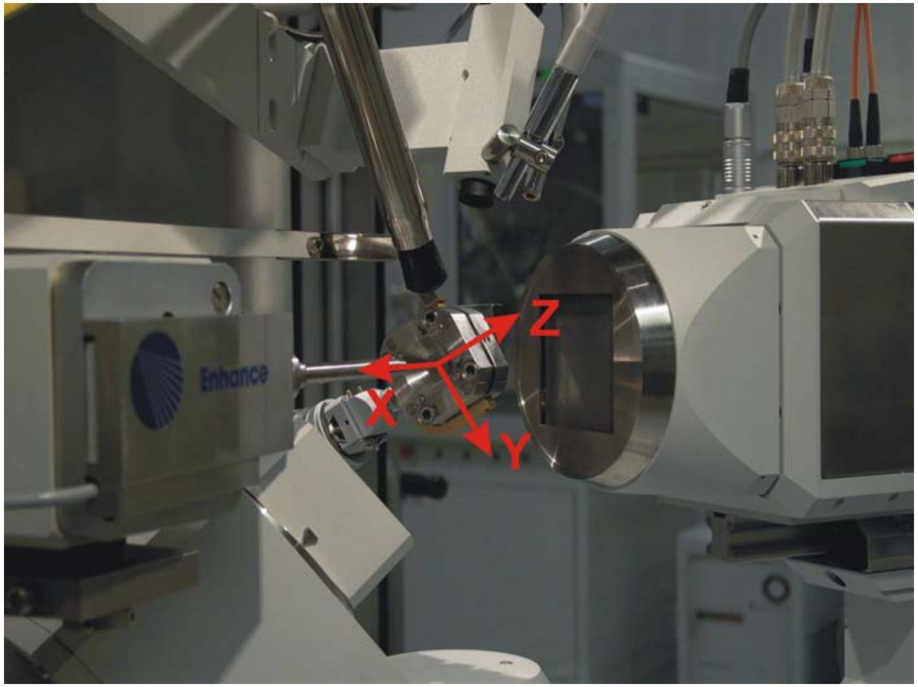
Kalsilite in the DAC at about 0.2 GPa: CRYSALIS DATA REDUCTION

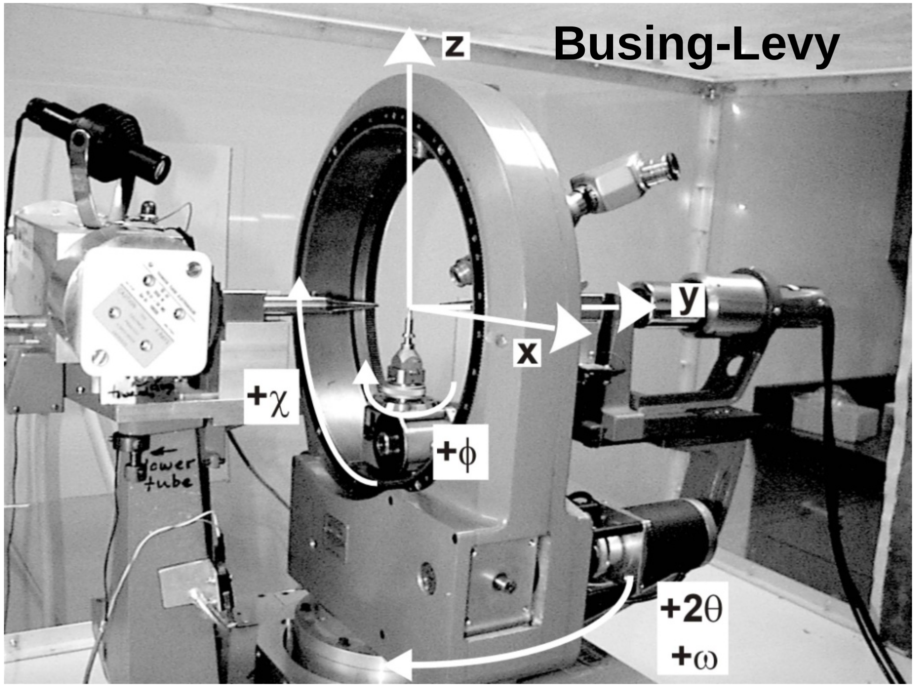
Statistics vs resolution (taking redundancy into account)

resolu-tion(A)	# measured	# unique	# theory	%complete	average redundancy	mean F2	mean F2/sig(F2)	Rint	RsigmaB
inf-1.56	71	26	34	76.5	2.7	1283.22	91.54	0.030	0.011
1.56-1.23	79	26	28	92.9	3.0	922.17	51.69	0.050	0.015
1.23-1.08	65	26	26	100.0	2.5	343.89	21.94	0.115	0.034
1.08-0.97	51	26	26	100.0	2.0	432.77	25.09	0.144	0.039
0.97-0.89	50	26	30	86.7	1.9	399.92	24.58	0.101	0.038
0.89-0.83	52	26	31	83.9	2.0	315.33	21.06	0.112	0.041
0.83-0.78	44	26	30	86.7	1.7	294.20	17.06	0.204	0.051
0.78-0.74	43	26	26	100.0	1.7	776.23	21.36	0.187	0.050
0.74-0.71	35	28	33	84.8	1.3	282.90	11.82	0.308	0.049
inf-0.71	490	236	264	89.4	2.1	614.29	31.79	0.081	0.026
inf-0.80	393	172	192	89.6	2.3	641.46	40.72	0.067	0.023

```
UB fit with 164 obs out of 164
unit cell:
       5.160(3)  5.164(3)   8.723(5)
      90.05(4)  90.10(5)  120.04(6)
      V = 201.2(2)
    unit cell:
       5.1645(13)  5.1645(13)   8.711(2)
      90.0        90.0        120.0
      V = 201.2(2)
```

Xcalibur does not operate following the Busing-Levy configuration!





Axis directions when diffractometer circles at zero

The definition of the phi-axis system, and thus **U** and **UB**, is different in different software!

And you need to know:

- Type of goniometer (kappa or Eulerian)
- Circle parities
- Conventions used by your absorption program

RJA's presentation