Rigaku FR-X Usage

Training on 2015.03.30

FR-X generator: 45 kV, 66 mA, 70 µ cross-section, 180 µ beam, 1.7 miliradians divergence

XG Control tells you the settings values (45 kV and 66 mA). Usually you can start collecting data 1h after setting the values.

Pilatus 300K detector: 3 chips, shorter exposure time, no readout (no shutter closing), almost never overloaded (>1.5 millions), water circulation at room temperature, dry air

```
>thread
```

command on camserver gives the temperature and humidity level Green light at the back of the detector to check before use.

Cryostream should be at 100 K

Close 3 glass doors, push the green light on the left [1]

Run HKL_3000_P300KChi [2]

Collect/Connect to initialize (also open an instrument server window in the background)

0.25° oscillation by default As a "rule of thumb" set the detector distance (in mm) to half the longest unit cell parameter (in Å) [3]

Align/Zero goniostat resets all positions (parameters in /hkl_dc.ini)

2θ always swings away (clockwise)

Set the directory /data/user/project1_crystal1

dtdisplay

- middle click > zoom area
- middle click > reset zoom
- control + left click > move around
- measure /Measure Left 1x, 2x, 3x or 4x (to average)

Never collect at 2θ = 0, use at least 2θ = 2 to offset the center horizontally and avoid thus avoid missing data due to the gaps between detectors

Index

- Refine in triclinic (P1)
- Fit Basic

- Fit All
- Mosaicity (by default it cannot estimate well the mosaicity[4], so you need to select)
- To process up to the corner, you need to index up to the corner!

Strategy

- HKL_3000 strategy only works for single axis
- Rigaku_Strategy is specially made for FR-X on the 4 axis
- Choose I want to make changes
- Completeness 99% (to reduce collect time)
- Redundancy 3.0 (4.0 by default)
- Distance (the distance from the first image is set)
- Suggested distance is not useful
- Edit preferences is possible

You can swing the slider on Time window to adjust/reduce the number of collected frames Don't forget to adjust the Mosaicity value! If anomalous data is required, adjust the redundancy accordingly

For some pins, Chi 60° may be too high and should be restricted to 50°

Close the window and choose *Setup DC* to transfer the information to HKL data collection window
Exposure time is not imported from the strategy and should adjusted manually

multi mode is useful to find the best detector distance and exposure time

Check diffraction images go to the data directory

```
dtdisplayhkl *.img &
```

30 to 50 strong reflections are needed for indexing. If it is not the case, increase the exposure time. It's possible to index non consecutive images if they have the same scan id

At 2θ ω goes from -90° to +90°, so don't put 90° step with 0° as ω start

Open display, do the peak search there, frame ↑ (middle click), the peak search has automatically changed, OK

Chi geometry goniometer, omega angle 180° follows the 2θ angle

[1]
HKL does get info on the shutter status
[2]
the other HKL_3000 version is for data integration only
[3]
2 slits can be tweaked to reduce the divergence with the middle wheel, but at some point the beam decreased and the exposure time will need to be increased to compensate
[4]
default value 0.3

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