Quick Guide for Data Collection on the NIU Bruker Smart CCD

1. Create a new project
2. Optically align the crystal
3. Take rotation picture
4. Collect matrix to determine unit cell
5. Refine unit cell
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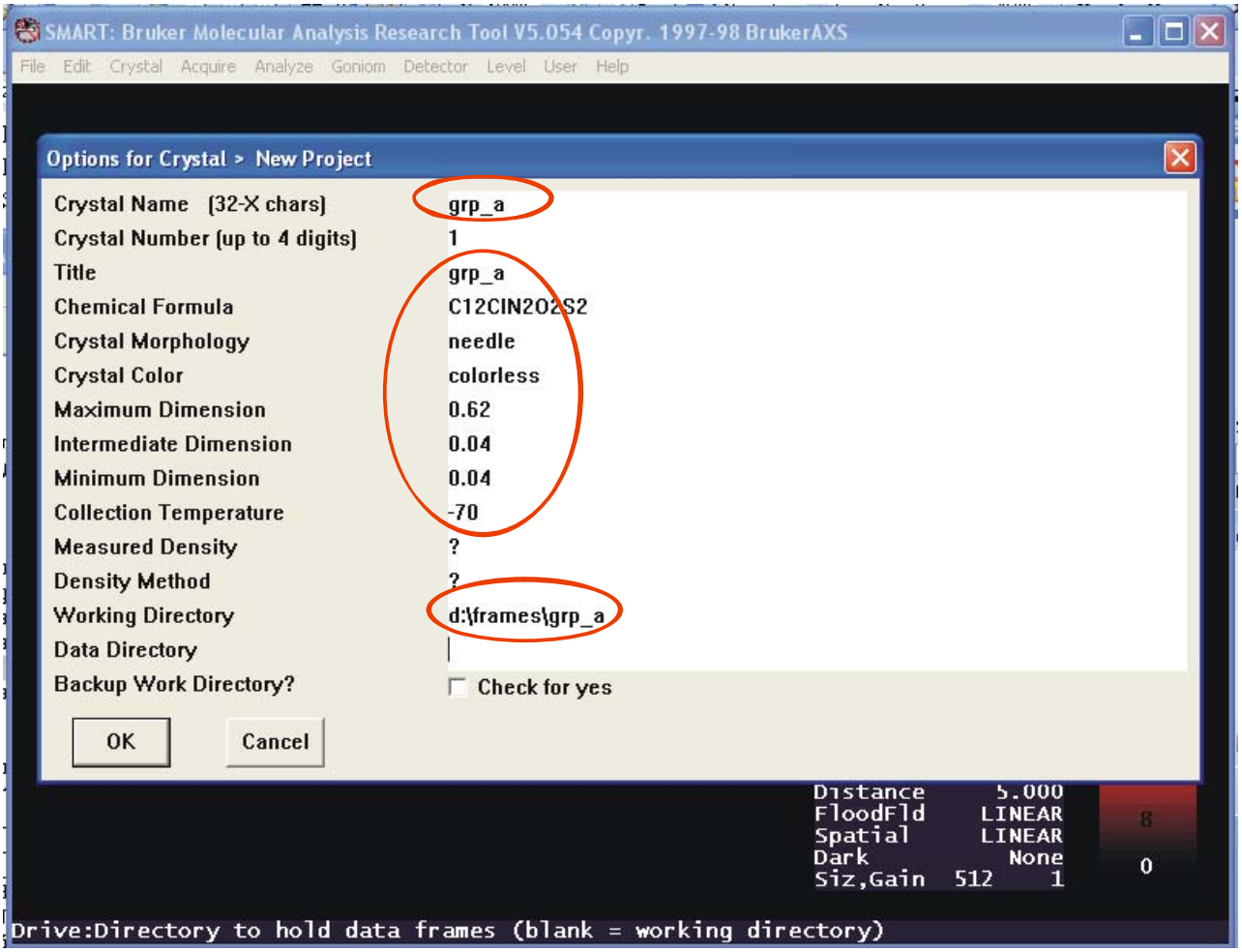
In the following Smart program panels, the parameters in red circles are those to be changed.

Individual Steps

1. Create a new project

Under menu **Crystal → New Project,** give new project a name (e.g. grp_a) etc.:



Click **OK** three times. In the fourth prompt, use **Small Molecule** setting.

Move all axes to zero position by using **Goniom → Zero** (click **OK** when prompt).

2. Optically align the crystal

Mount the crystal to the goniomenter head. Screw the goniometer head on to the diffractometer.

Open menu **Goniom → Optical**, accept the default parameters. Now the goniometer is in manual mode.

Use the goniometer manual control box, align the crystal. First press the **B** button, then the **Print Axis** button. The goniometer will move the mount position.

Adjust the height of the crystal so that it is at the center of the cross in the microscope.

Adjust the lateral Y position so that the crystal is at the center of the cross in the microscope.

a) Press the **A** button and then the **Print Axis** button, the crystal will rotate 90 degrees. Adjust the lateral X position so that the crystal is at the center of the cross in the microscope.

Press the **Print Axis** button, the crystal will rotate 180 degrees. If the crystal is of the center, adjust it only half way toward the correct position.

Press the **Print Axis** button again, the crystal will rotate another 180 degrees. If the crystal is off center, adjust it only half way toward the correct position. Repeat the above two procedures until the crystal is not moving when rotated.

The reason that we adjust the crystal only half way toward the correct position is that sometimes the cross in the microscope is not the true center. By using the infinitesimal （$1/2 \times 1/2 \times 1/2 \ldots$）approach we can find the true center.

b) Press the **B** button and then the **Print Axis** button, the crystal will rotate 90 degrees. Adjust the lateral Y position so that the crystal is at the center of the cross in the microscope.

Press the **Print Axis** button again, the crystal will rotate another 180 degrees. If the crystal is off center, adjust it only half way toward the correct position. Repeat the above procedure until the crystal is not moving when rotated.

Repeat steps a) and b) if necessary until the crystal is centered.

Return the manual control box to the side, close the radiation protection windows until all window status lights in the front panel turn green, push the small **Reset** button. All window indicator lights must be green before the shutter can be opened.

Press the **Esc** key on the computer to return to the automatic mode. Move all axes to zero position by using menu **Goniom → Zero** (click **OK** when prompt).

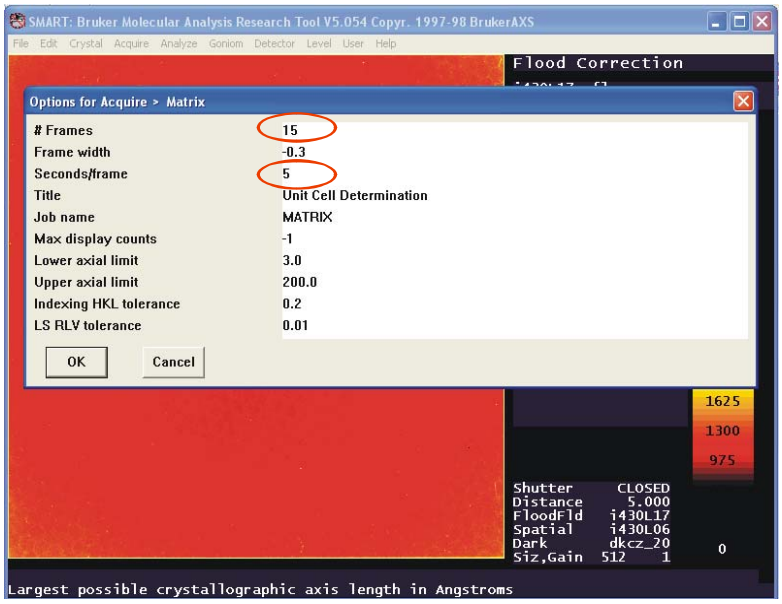
3. Take rotation picture

Open menu **Acquire → Rotation**, choose a 30 second exposure time, click **OK**. **DO NOT** use exposure time more than 60 seconds without consulting with an expert. You may damage the CCD detector!

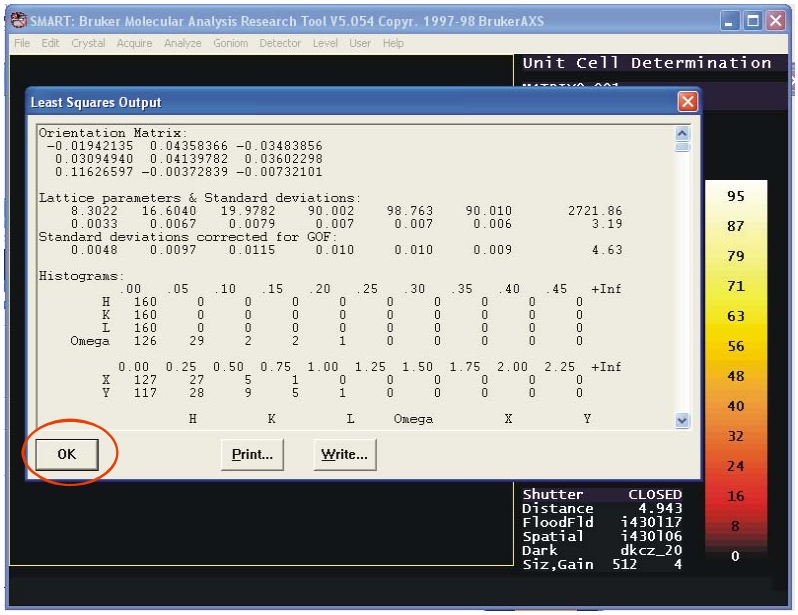
Exam the rotation picture (remember the Ewald construction?) to make sure that the crystal quality is good.

4. Collect matrix to determine unit cell

Open menu **Acquire → Matrix.** Depending on the quality of the crystal, we choose appropriate number of frames to collect and the exposure time per frame. For most organic crystals, 15 frames and 5 seconds/frame exposure time is good. Leave other parameters at their default values.

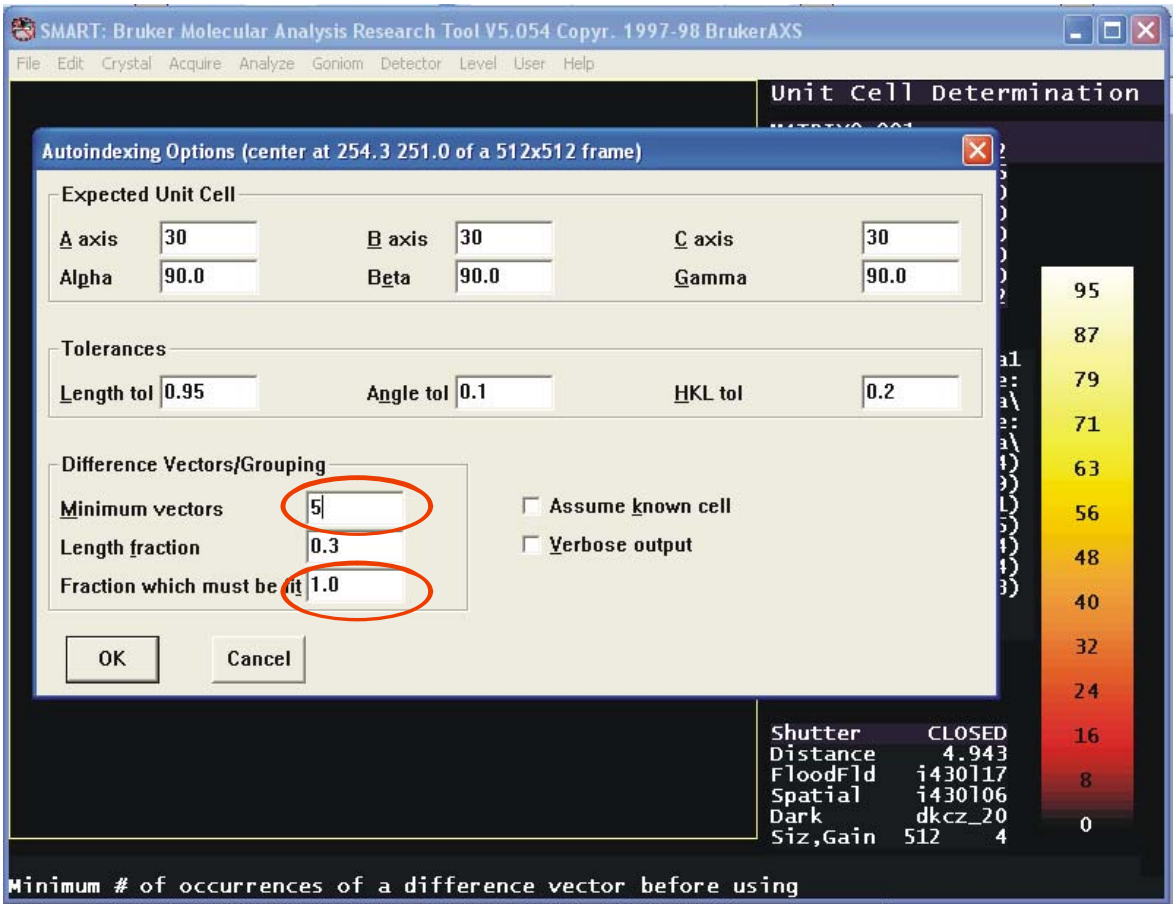


This will take about 7 minute time, after which you may see a screen like this:

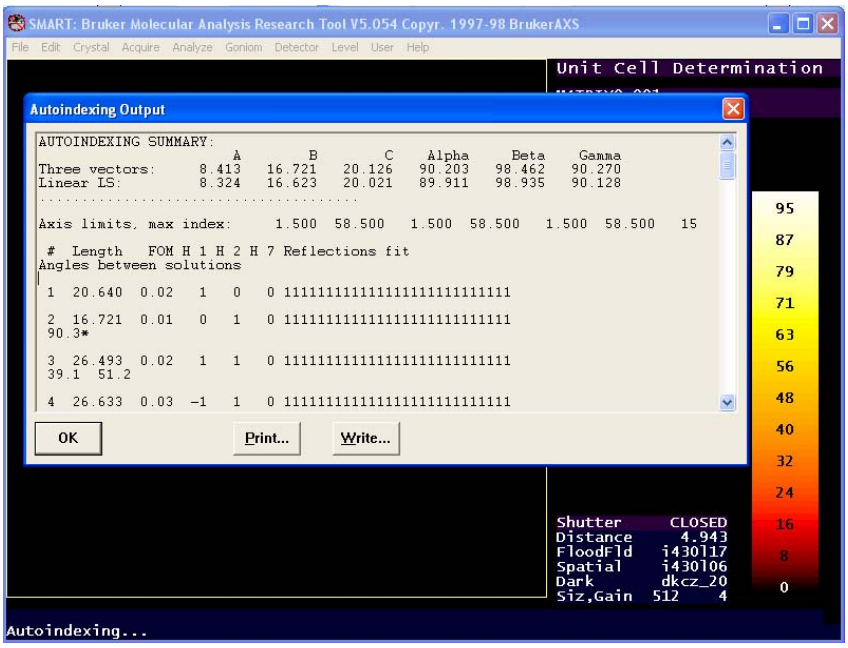


Click **OK** to close the window.

Go to menu **Crystal → Index**, change **Minimum vectors** to **5**, and **Fractional which must be fit** to **1.0**, click **OK**. If index fails, reduce the fraction to **0.9, 0.85, 0.8** etc. Below **0.8**, the crystal is no good, change to another crystal:



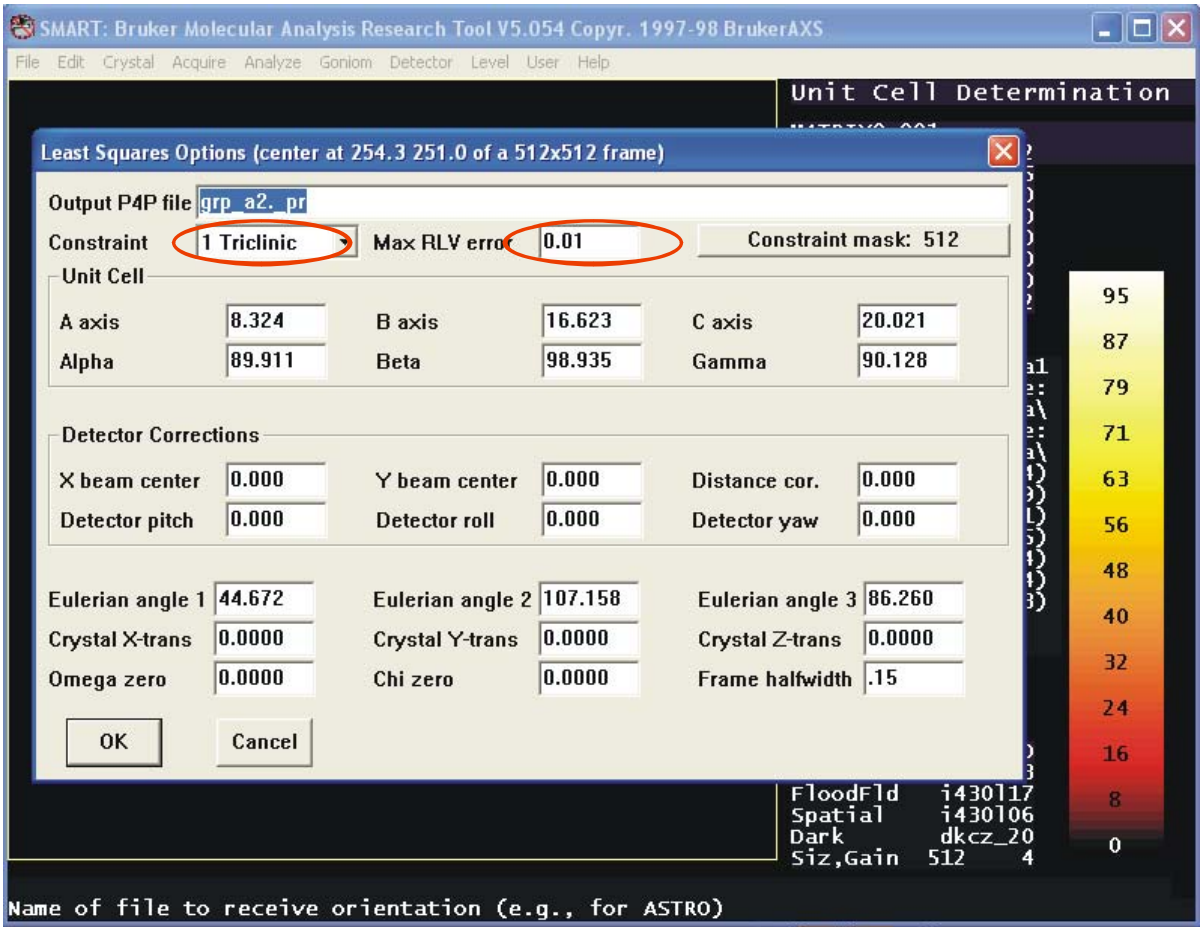
If the crystal can be indexed, you will see a screen like this:



A 1 means a reflection fits the vector, a 0 not. Ideally, you want all reflections fit, with all 1's: 111111111111111…

Click **OK** to close the window.

Go to menu **Crystal → LS** to do least square refinement of the cell. Choose **Constrain** to be **1 Triclinic** and **Max RLV error** to be **0.01**:



Click OK. Go to menu **Crystal → LS** again, now choose **Constrain** to be **-1 Triclinic** and **Max RLV error** to be **0.005** (we do not use any bias for a unknown crystal, therefore we choose triclinic lattice. 1 is for linear LS, -1 for nonlinear LS fitting).

Go to menu **Crystal → Bravais** to choose Bravais lattice (the index part gives only primitive lattice). Accept all default values for most crystals.

Do cell refinement again: Go to menu **Crystal → LS**, choose **Constrain** to be **1 Triclinic** and **Max RLV error** to be **0.005**. Click **OK**. Go to **Crystal → LS** the last time, choose **Constrain** to be **-1 Triclinic** and **Max RLV error** to be **0.003**. Click **OK**.

Now you have the cell constants. Record the values of a, b, c, α, β, γ, lattice type, first histogram column, X-cent, Y-cent, Dist, Omega, Chi zeros, GOF and total numbers of reflections used in the refinement. The absolute values of X-cent, Y-cent, Dist should be less than 0.5 for a correctly centered crystal, GOF should be close to 1 for a correctly index crystal. The number of reflections in the histograms should be 50% or greater of the total number of reflections used in the cell refinement.

8. Home instrument

Since there are accumulate errors in the instrument gears, we need to reset the motor reference positions each time we collect data. This procedure is called Home axes.

First move all axes to zero position by using **Goniom → Zero**.

Home the first (θ) axis by using menu **Goniom → Home axis**.

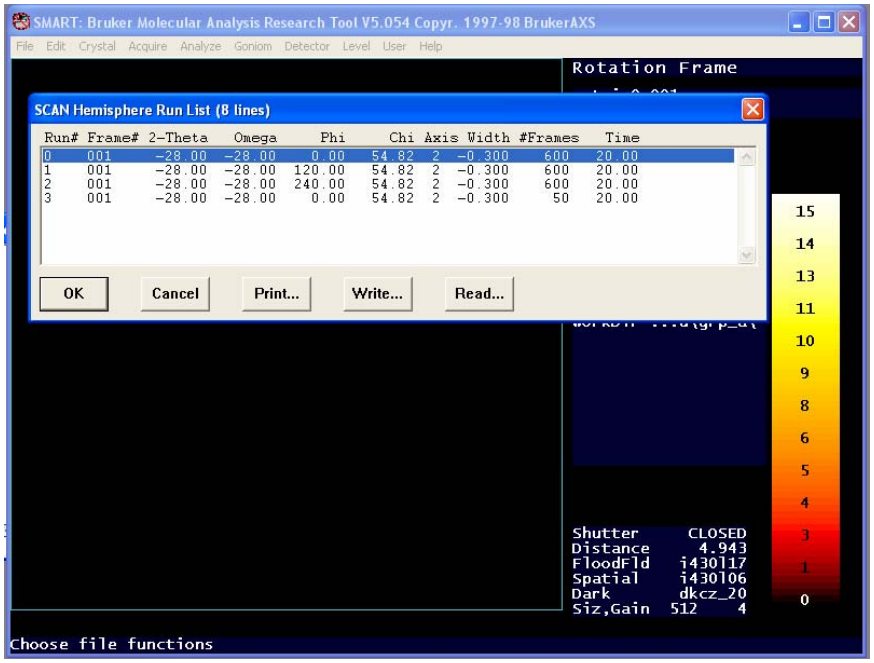
Home the second (ω) axis by using menu **Goniom → Home axis** (the axis number will automatically change to 2).

Home the third (ϕ) axis by using menu **Goniom → Home axis** (the axis number will automatically change to 3).

Move all axes to zero position by using **Goniom → Zero**.

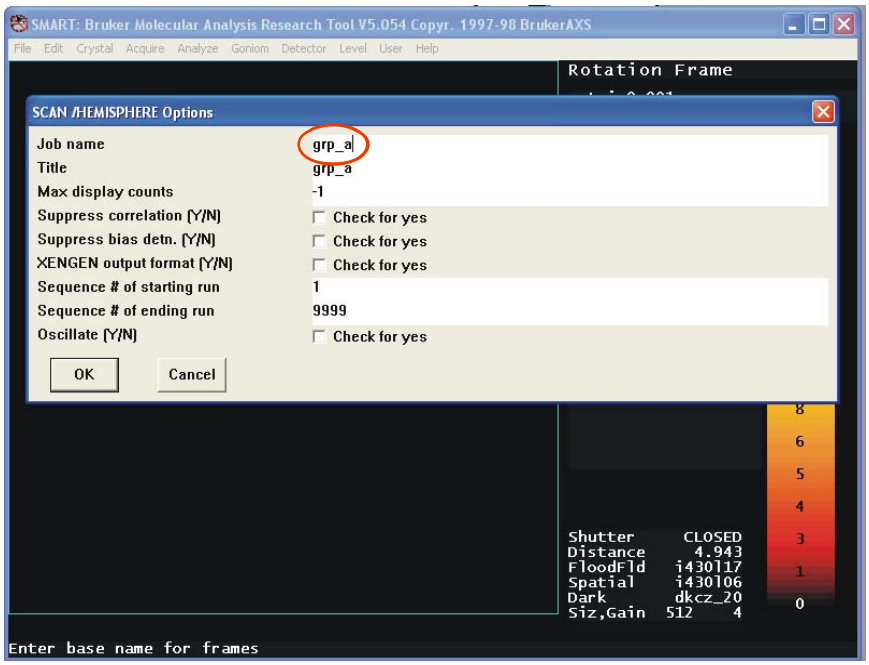
9. Start data collection

Normally we collect the reflections of the reciprocal lattice points inside the hemisphere of the Ewald sphere. Because of the Friedel relation $I_{hkl} = I_{-h-kl}$, these reflections are complete even for the lattice with the lowest symmetry (P 1). For lattice with higher symmetry, the high redundancy will give better signal to noise ratio (I/σ) after averaging. Given the geometry of our diffractometer, the data collection strategy is given in menu **Acquire → EditHemi**:



Click **OK** to close the window. For strongly diffracting crystals, you can reduce the exposure time from 20 seconds to 10 seconds. For weakly diffracting crystals, you can increase the exposure time from 20 seconds to 30 seconds. **DO NOT** use exposure time more than 50 seconds without consulting with an expert. You may damage the CCD detector!

Now and finally, we can collect data by using menu **Acquire → Hemisphere**:



Click **OK** to close the window. Accept all the default parameters when prompt.

Data collection using 20 second/frame will last 13 hours.