Quick Guide for Data Integration on the NIU Bruker Smart CCD

Four simple steps:

1. Switch to your project
2. Index your cell using the data of the whole hemisphere
3. Copy the 2.p4p file to 0.p4p, 1.p4p and 3.p4p
4. Integrate data

In the following Smart program panels, the parameters in red circles are those to be changed.

Individual Steps:

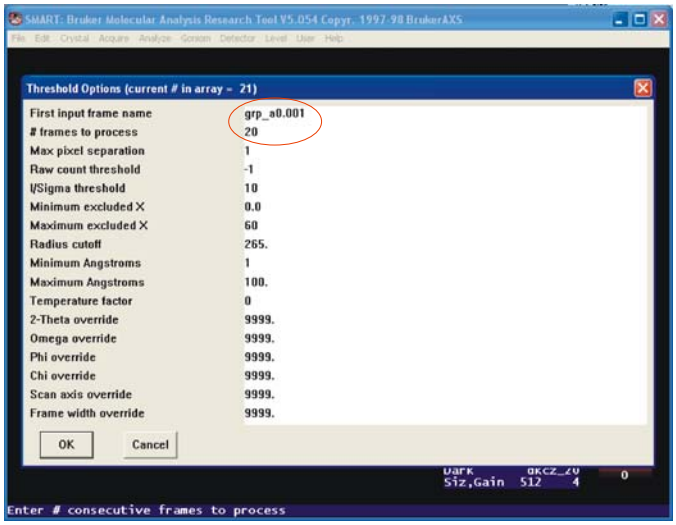
1. Switch to your project

In the program SMART, go to menu **Crystal → Switch Projects** and find your project (e.g. grp_a). Notice that your data have four series: grp_a0.***, grp_a1,***, grp_a2,*** and grp_a3.***. The first three series are ω scan data at different ϕ angles (0, 120 and 240 degrees). The fourth series is the repetition of the first one up to the first 50 frames. You will use the fourth series to judge whether your crystal has moved during data collection. The integration program will also use it to estimate the crystal decay correction, if there is any.

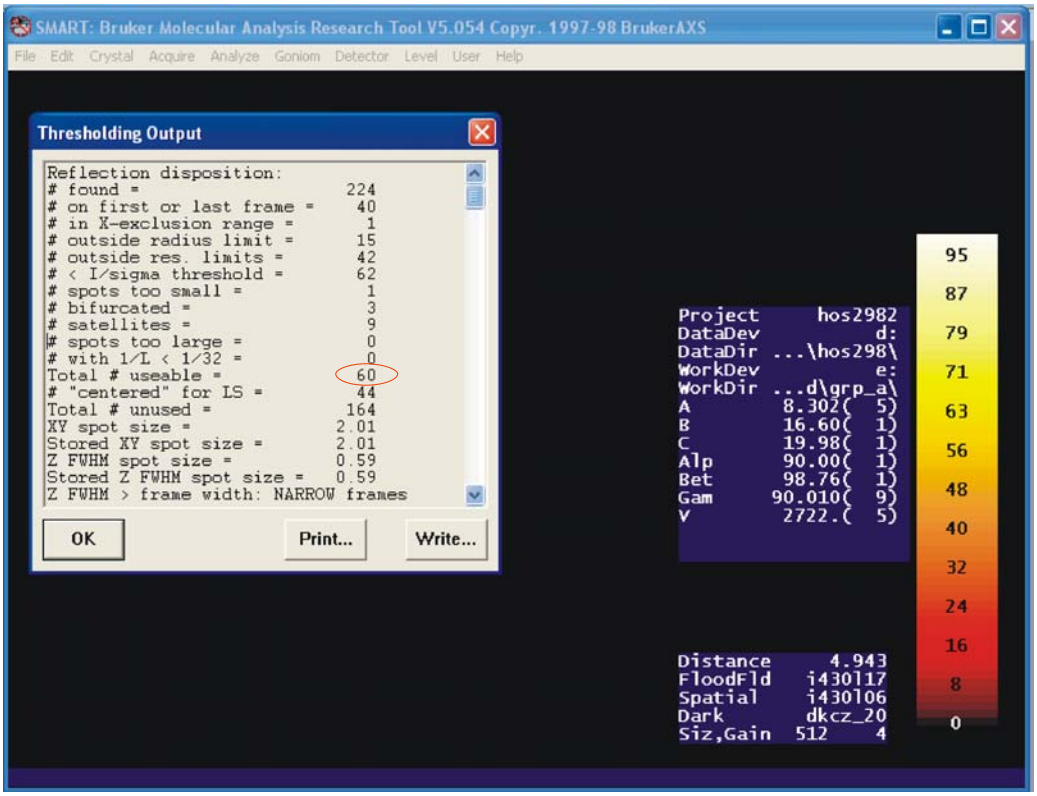
2. Index your cell using the data of the whole hemisphere

Go to menu **Crystal → Edit** to make sure that no reflection is stored in the program. If there are reflections, use **Crystal → Clear** to clear up the memory (**Frame Size 1024**). Now you want to use different data series and extract reflections from 10 to 20 frames evenly distributed in the hemisphere of the reciprocal lattice.

Go to menu **Crystal → Threshold**, extract 20 frames from series grp_a0.001:

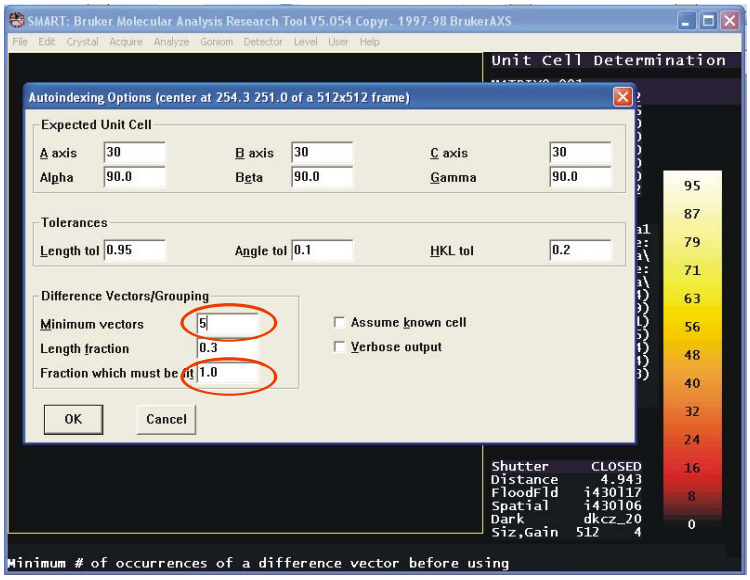


Click **OK.** The program will find between 50 to 100 usable reflections.

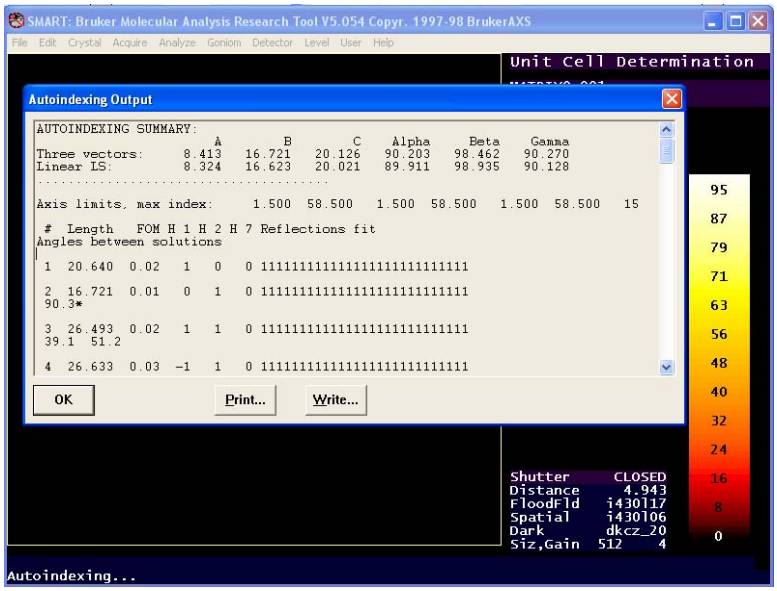


Click OK two times when prompted. If the number of reflections is not within this range, start over again, and change the **# of frames to process parameter** (only once for the first frame set grp_a0.001) to other values (but not larger than 50). If you do so, remember to clear the memory first (**Crystal → Clear** with 1024 Frame Size).

Repeat the threshold procedure eight more times, each time change the **First input frame name** parameter to (remember to substitute grp_a with your project name):
grp_a0.201; grp_a0.401; grp_a1.001; grp_a1.201; grp_a1.401; grp_a2.001; grp_a2.201; grp_a2.401.
(You can use the program macro called SLAM to do this in one command. But it is for more advanced users)
Now we will carry out the indexing procedure: 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:



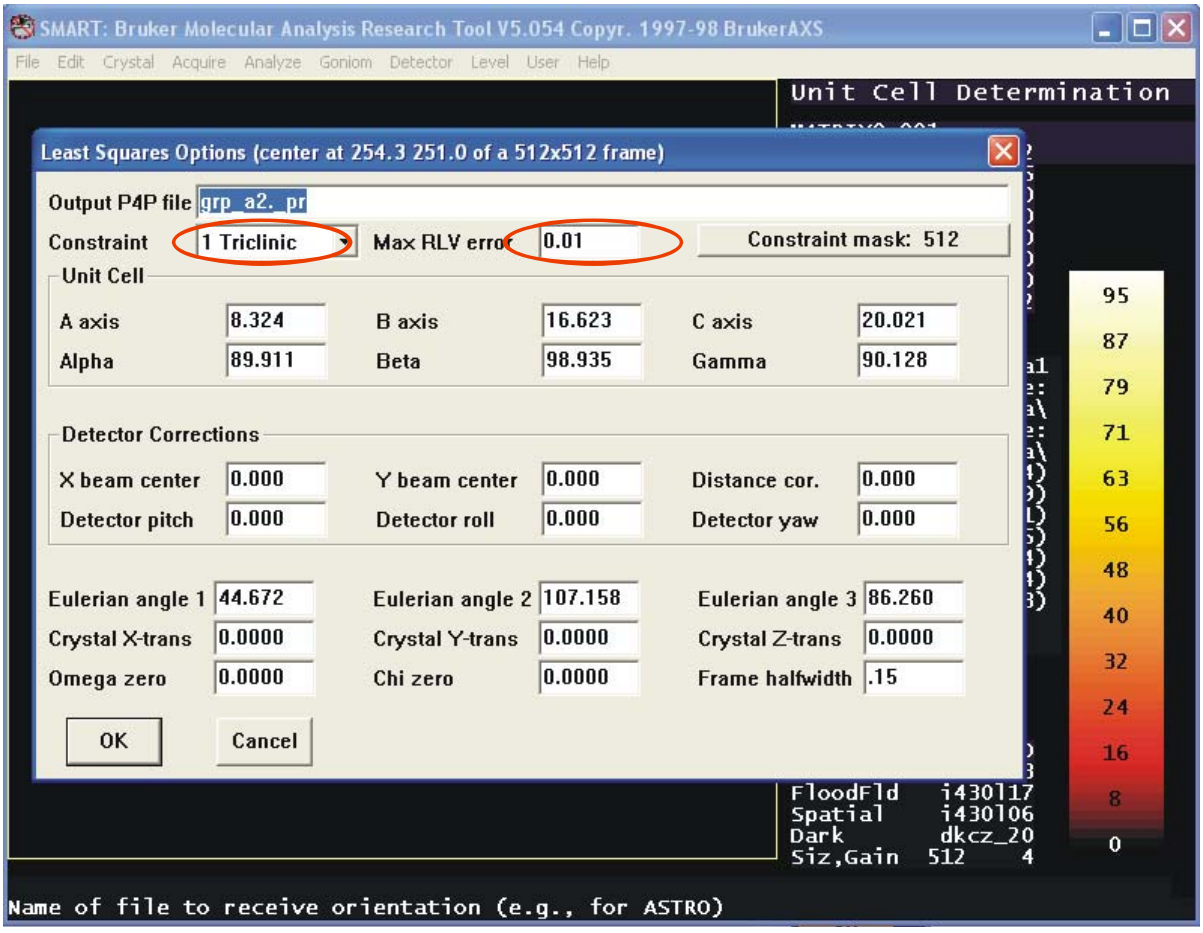
If the crystal can be indexed, you will see a screen like this:



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. Pay attention to the lattice type given by the program.

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.

Now we want to make sure that the crystal did not move during data collection. Go to menu **Analyze→Display,** type in the first file name in the File name menu: **grp_a0.001**, remember a few reflection positions. Now go to menu **Analyze→Display,** type in the corresponding file name of the last series in the File name menu: **grp_a3.001**, compare the reflection positions with the grp_a0.001 frame. If all the corresponding positions are the same, the crystal has not moved. You can also compare other corresponding frames such as **grp_a0.030** and **grp_a3.030**, etc. We can now proceed with data integration.

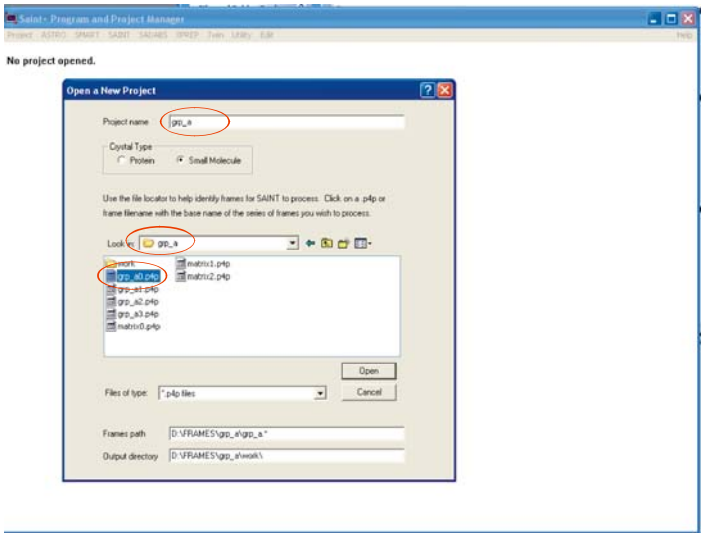
3. Copy the 2.p4p file to 0.p4p, 1.p4p and 3.p4p

The .p4p files are the orientation matrix files that contain the cell geometry transformation information. The program SAINT will use this information to convert the frame data to hkl data. After you have index the crystal, the information is stored in 2.p4p file which is the last series that you have used for threshold. For the 0.***, 1.*** and 3.*** series, we don't have the .p4p file. Therefore we want to copy the 2.p4p file so that the program SAINT has the .p4p file for all series.

Open a DOS window, go to the data directory (cd d:\frames\grp_a) (substitute grp_a with your own project name), and copy three files:
copy grp_a2.p4p grp_a0.p4p
copy grp_a2.p4p grp_a1.p4p
copy grp_a2.p4p grp_a3.p4p

4. Integrate data

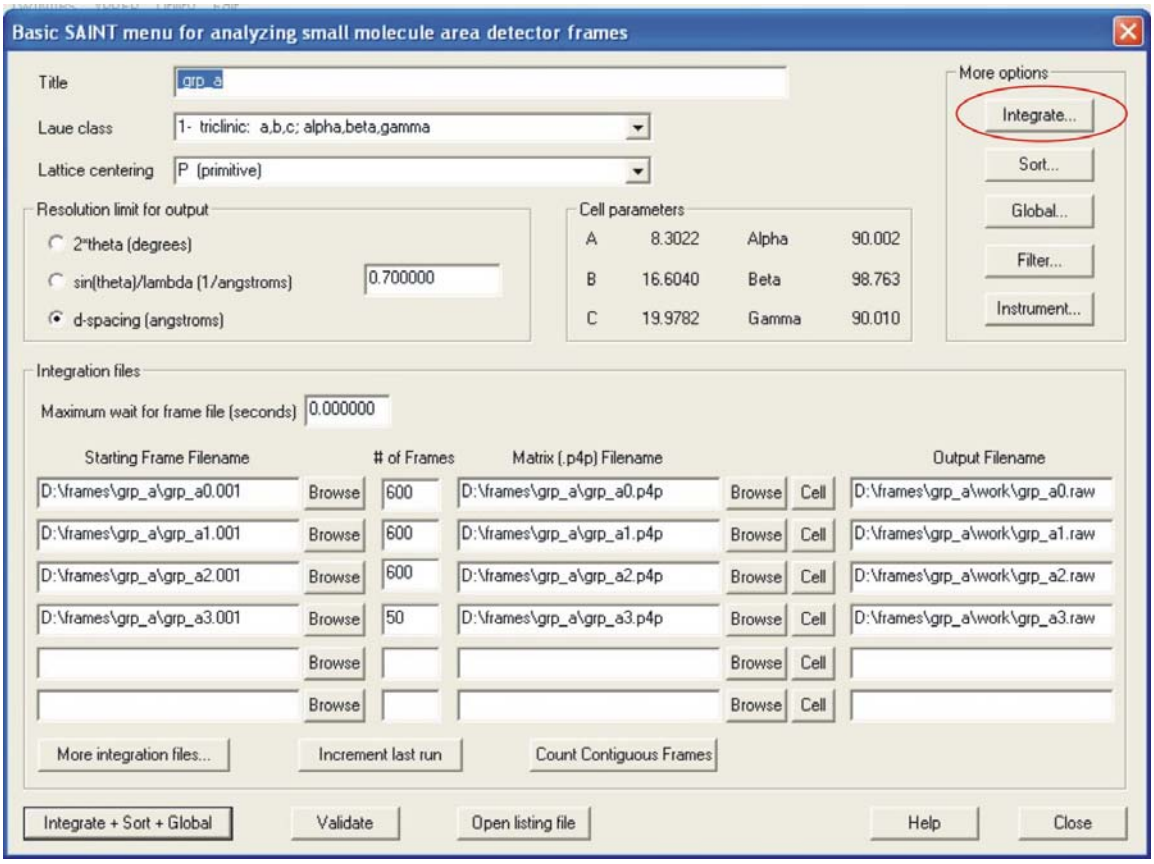
Lunch the program SAINTPLUS, open menu **Project→New,** give a project name (e.g., **grp_a**), locate the data directory, select the grp_a0.p4p file:



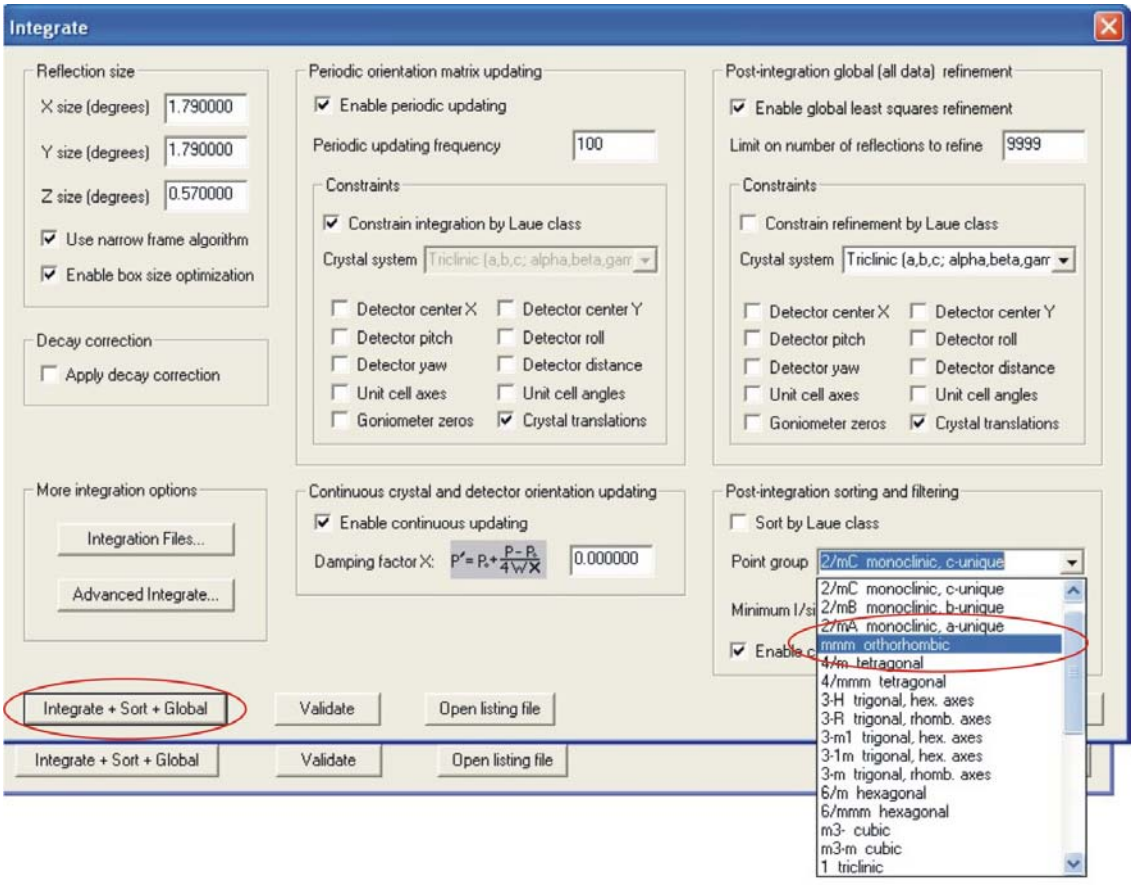
Click Open to close the window.
Go to menu **Saint→Initialize** (the process will finish in a second), and then **Saint→Execute**. In the next window, make sure that your cell constants, cell type and the resolution range (0.7 Å) are

correct.

Click the **Integrate**… button.



Choose the correct point group for your crystal:



Click **Integrate+Sort+Global** button. The integration now starts. Close this window and the

SAINTPLUS window (but not the DOS window that the program opens).

After 30 minutes, you will have the integrated data (the *.raw files) in the work directory.