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
- 6. Home instrument
- 7. Start data collection

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** \rightarrow **New Project**, give new project a name (e.g. grp_a) etc.:

😂 SMART: Bruker Molecular Analysis Research Tool V5.054 Copyr. 1997-98 BrukerAXS	_ 🗆 🗙
File Edit Crystal Acquire Analyze Goniom Detector Level User Help	
Options for Crystal > New Project	
Crystal Name (32-X chars) grp_a	
Crystal Number (up to 4 digits) 1	
Title grp_a	
Chemical Formula C12CIN2O2S2	
Crystal Morphology needle	
Crystal Color colorless	
Maximum Dimension 0.62	
Intermediate Dimension 0.04	
Minimum Dimension 0.04	
Collection Temperature -70	
Measured Density ?	
Density Method ?	
Working Directory d:\frames\grp_a	
Data Directory	
Backup Work Directory? 🔽 Check for yes	
OK Canad	
Distance 5.000	
Spatial LINEAR	
Dark None Siz Cain 512 1	0
Drive:Directory to hold data frames (blank = working directory)	

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

Move all axes to zero position by using **Goniom** \rightarrow **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** \rightarrow **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 $(1/2 \times 1/2 \times 1/2 \dots)$ 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** \rightarrow **Zero** (click **OK** when prompt).

3. Take rotation picture

Open menu Acquire \rightarrow 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** \rightarrow **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 \rightarrow 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:

SMART: Bruker Molecular Analysis	Research 7	Fool V5.054	Copyr. 1997-98 Bruke	rAXS		
Edit Crystal Acquire Analyze Gonio	m Detector	Level User	Help	Unit Ce	11 Determ	nination
Autoindexing Options (center at 2)	54.3 251.0	of a 512x5	12 frame)		<u> </u>	
Expected Unit Cell					3	
<u>A</u> axis 30	<u>B</u> axis	30	<u>C</u> axis	31) <u> </u>	
Al <u>p</u> ha 90.0	B <u>e</u> ta	90.0	<u>G</u> amma	91).0	95
						87
Tolerances				_	a1	79
Length tol 0.95	A <u>ng</u> le tol	0.1	<u>H</u> KL tol	0.	2	71
Difference Vectors/Crouning					1	71
		-	ocume known cell		ž	63
Minimum vectors			/erhose output		5	56
Fraction which must be it 1.0					ģ	48
Traction which must be http://	\mathcal{I}				, i i i i i i i i i i i i i i i i i i i	40
OK Cancel						32
						24
				Shutter	CLOSED	16
				FloodFld	4.943 i430117	8
				Dark	dkcz_20	0
10 U.S. 10			115 - 27 - 193	SIZ,Gain	512 4	

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

Edit Crystal Acquire Analyze Goniom Detector Level User Help Unit Cell Determi	natio
Autoindexing Output	
AUTOINDEXING SUMMARY: A B C Alpha Beta Gamma Three vectors: 8.413 16.721 20.126 90.203 98.462 90.270 Linear L5: 8.324 16.623 20.021 89.911 98.935 90.128	
	95
Axis limits, max index: 1.500 58.500 1.500 58.500 1.500 58.500 15	87
# Length FOM H 1 H 2 H / Reflections fit Angles between solutions	79
1 20.640 0.02 1 0 0 1111111111111111111111111	71
2 16.721 0.01 0 1 0 11111111111111111111111111	63
3 26 493 0 02 1 1 0 111111111111111111111111111	60
39.1 51.2	56
4 26.633 0.03 -1 1 0 111111111111111111111111	48
OK Print Write	40
	32
	24
Shutter CLOSED	16
Distance 4.943 FloodFld i430117 Spatial i430106	
Dark dkcz_20	0

A 1 means a reflection fits the vector, a 0 not. Ideally, you want all reflections fit, with all 1's: 11111111111111...

Click **OK** to close the window.

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

SMART: Bruker Mo	lecular Anal	ysis Research Tool V5.0	154 Copyr. 199	7-98 Bruke	rAXS		_ 0 2
File Edit Crystal Acqui	re Analyze (Soniom Detector Level U	ser Help			·	
					Unit Cell	Determi	nation
Least Squares Opt	ions (center	at 254.3 251.0 of a 51	2x512 frame)			2	
Output P4P file	rp_a2pr					}	
Constraint (1	Triclinic	Max RLV erro	0.01	Сол	straint mask: 512	2	
Unit Cell				6		}	0.F
A axis	8.324	B axis	16.623	C axis	20.021	2	93
Alpha	89.911	Beta	98.935	Gamma	90.128	11	87
						≥: a\	79
Detector Correc	tions					2: a\	71
X beam center	0.000	Y beam center	0.000	Distance	cor. 0.000	1 B	63
Detector pitch	0.000	Detector roll	0.000	Detector	yaw 0.000	ġ	56
						12	48
Eulerian angle 1	44.672	Eulerian angle 2	107.158	Eulerian	angle 3 86.260	j)	40
Crystal X-trans	0.0000	Crystal Y-trans	0.0000	Crystal Z	Z-trans 0.0000		32
Omega zero	0.0000	Chi zero	0.0000	Frame h	alfwidth .15		74
ок	Cancel						16
						3	10
					Spatial i	430106	8
					Dark d Siz,Gain 51	kcz_20 2 4	0
ame of file to	receive	orientation (e.	q., for AST	ro)			

Click OK. Go to menu Crystal \rightarrow 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** \rightarrow **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 \rightarrow LS, choose Constrain to be 1 Triclinic and Max RLV error to be 0.005. Click OK. Go to Crystal \rightarrow 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** \rightarrow **Zero**.

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

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

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

Move all axes to zero position by using **Goniom** \rightarrow **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** \rightarrow **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 \rightarrow Hemisphere:

Edit Crystal Acquire Analyze Goniom	Detector Level User Help	70 DI UNCIANO	کا لگا
		Rotation Frame	
SCAN /HEMISPHERE Options			D
Job name Title Max display counts Suppress correlation (Y/N) Suppress bias detn. (Y/N) XENGEN output format (Y/N) Sequence # of starting run Sequence # of ending run Oscillate (Y/N) OK Cancel	grp_a grp_a -1 Check for yes Check for yes Check for yes 1 9999 Check for yes		
			8
			6
			5
			4
		Shutter CLOSE	3
		FloodFld i43011	1
		Dark dkcz_2 Siz,Gain 512	0

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

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