

Data Collection Beginner

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version 2.0.1_2009

What to know before you begin

Name : _____ Date _____

Office number _____ Telephone _____ Fax _____ e-mail _____

Principle Investigator (Advisor) _____ Dept. _____

Account to be billed _____ to _____ for \$60.00

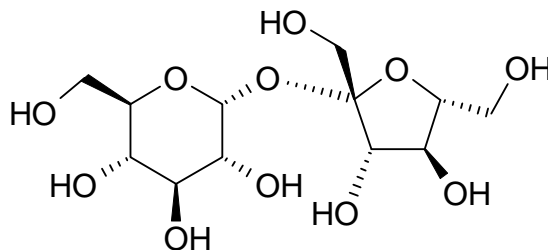
Original Sample Number _____ Location _____

✓ Chemical Formula $C_{12}H_{22}O_{11}$ Formula weight **342.3 g/mole**

Density _____? _____ Solvents used H₂O Sensitivity _____

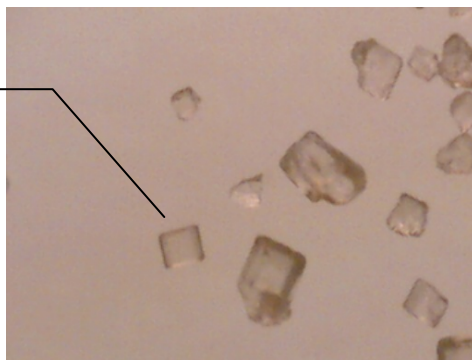
Crystal color Colorless Is the sample chiral or racemic? *chiral (D)*

Draw Structure (label all chiral centers).



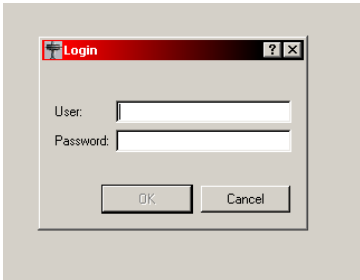
Choosing the crystal

Clear
Sharp edges
Flat faces



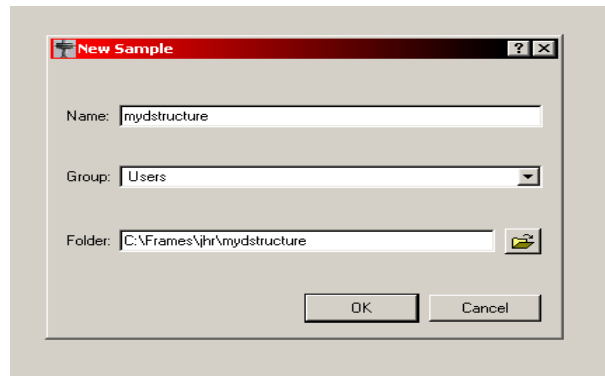
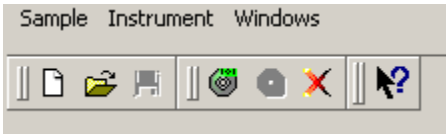
Screening the crystal on the APEXII

1. Login

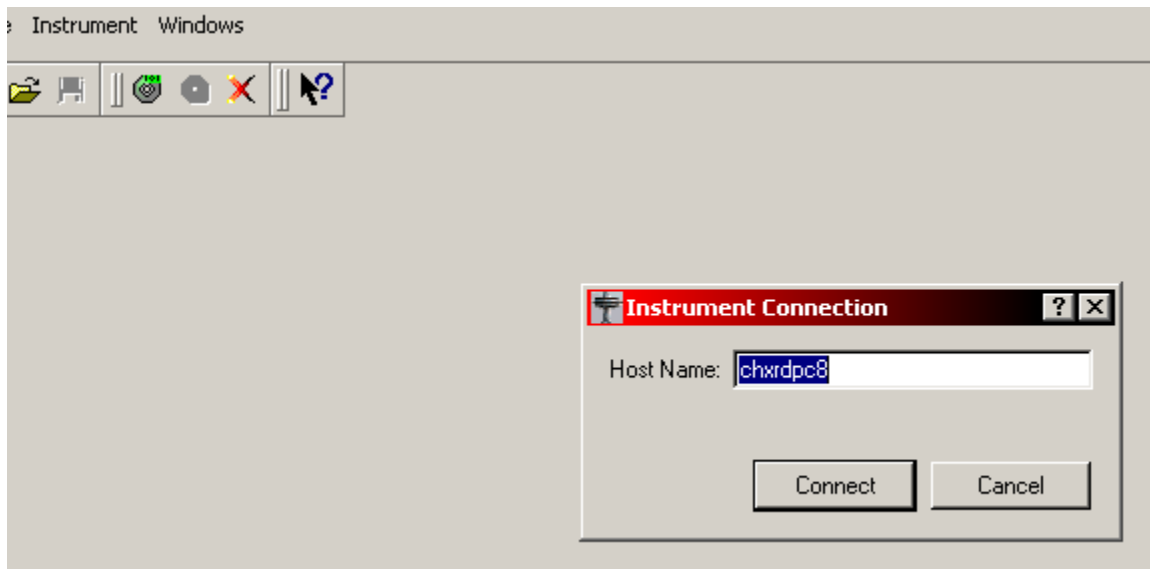


User name and password will be supplied to you

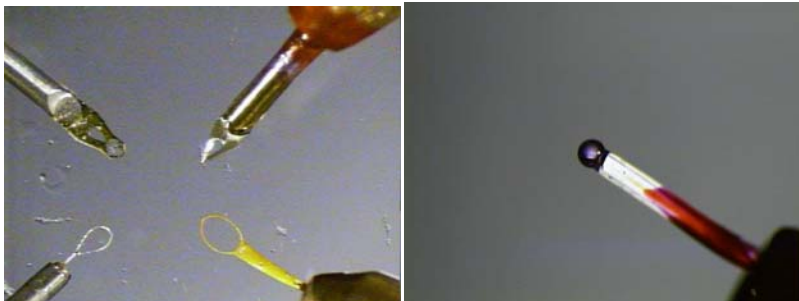
2. Start a new project



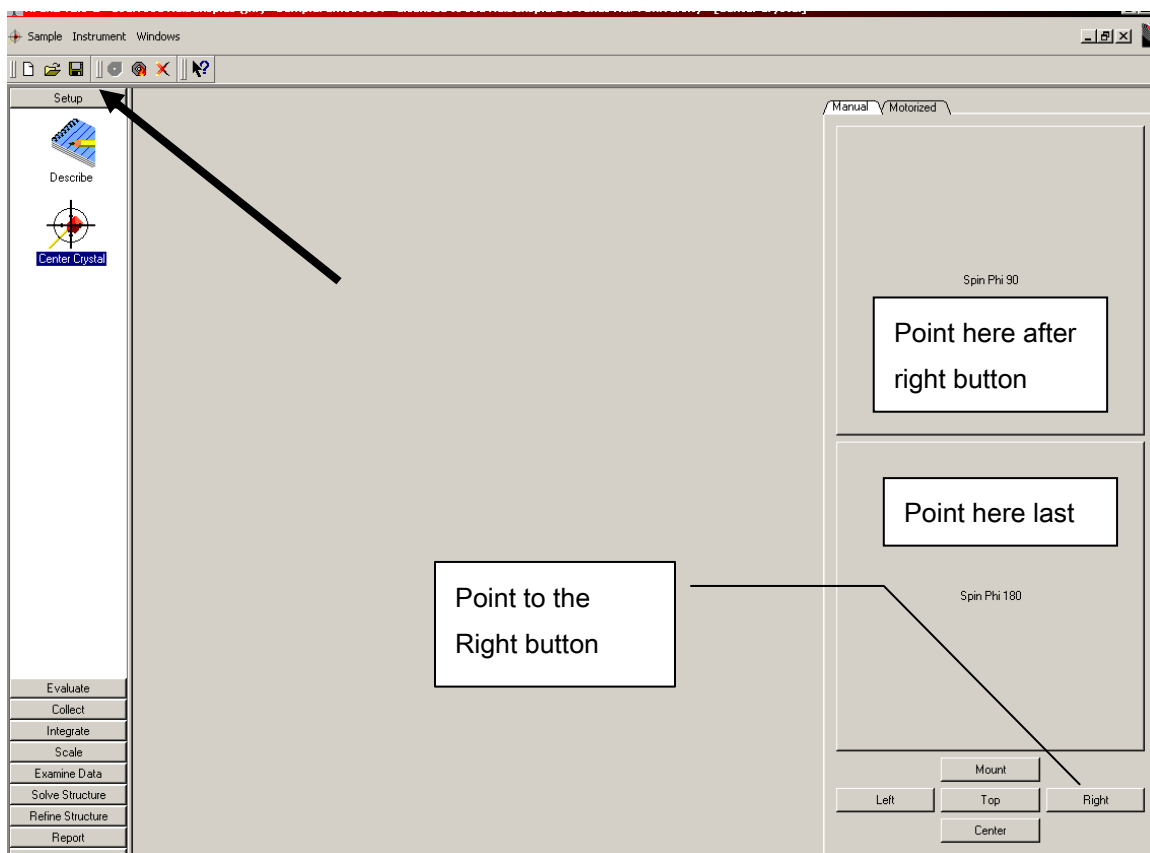
3. Connect the computer



4. Mounting crystal

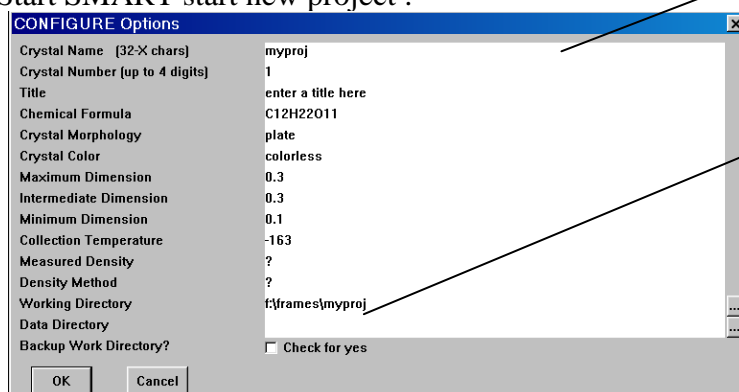


5. Center Crystal



Screening the Crystal in SMART

- Start SMART start new project :

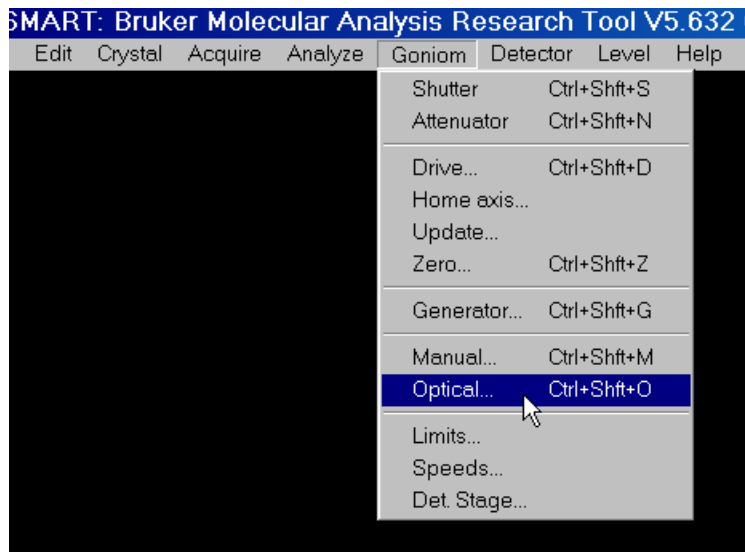


Input the known information

For working directory input
f:\frames\myproj
change f: if needed
use your project name

- Select SMALL MOLECULE when asked

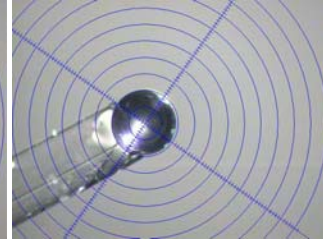
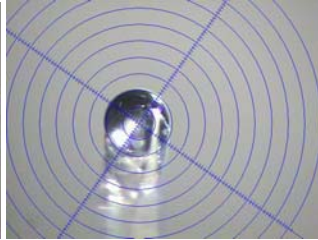
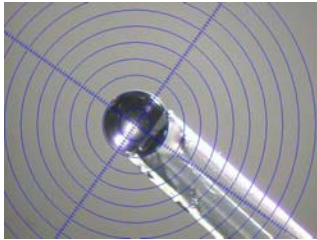
SMART



USE the A, B and C buttons to center crystal

Point to Goniom and Optical. Goto the external control. Select B and push AXIS print. Position the crystal as shown below. Always come from below. Using the "wrench" adjust the height of the crystal so that the crystal is near the crosshairs. Adjust the sledge below the height and adjust the X axis. Punch A and adjust Y axis. Punch C and check the height.

A position



C position

SMART :: When finished punch the "esc" key on the keyboard.

APEXII

APEX2 v2.0.2 - User: Joe Reibenspies (jhr) - Sample: test2 - Licensed to Joe Reibenspies at Texas A&M University - [Determine Unit Cell]

Sample Instrument Windows

Setup Evaluate

Determine Unit Cell

Transform unit cell

Reciprocal Lattice Viewer

View Images

Collect Integrate Scale Examine Data Solve Structure Refine Structure Report Instrument

Automatic Mode
Start at: Collect Data
Stop after: Refine
Run

Manual Mode
Collect Data
Harvest Spots
Index
Bravais
Refine

Unit cells:
Edit...
Delete
Delete All

Reflections:
Edit...
Delete
Delete All

Expected resolution:

	Exposure time [s]	Resolution [Å]
1	20.0	n/a
2	60.0	n/a
3	120.0	n/a
4	600.0	n/a

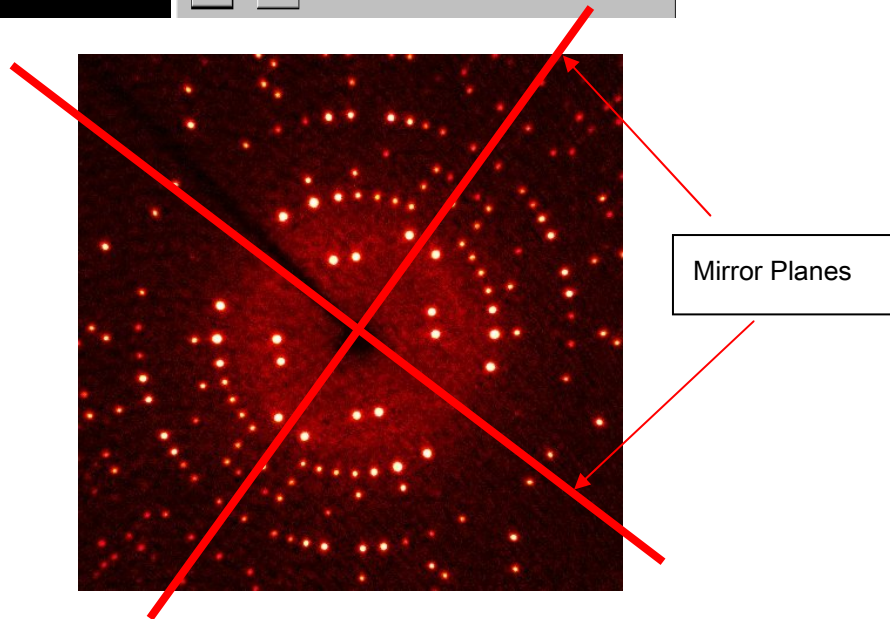
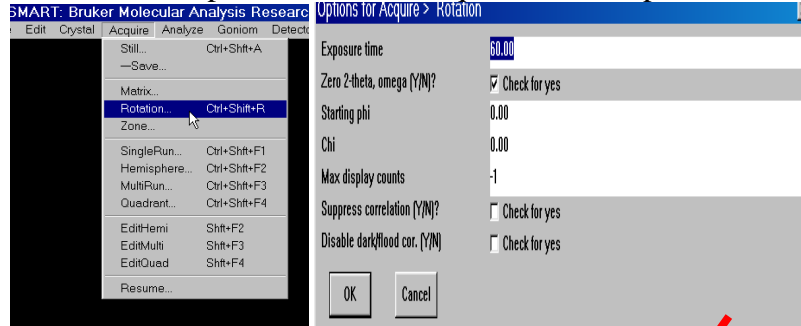
Crystal Mosaicity [°]: 0.40

Cursor
Position [mm]
Position [pixels]
Intensity [counts]
HKL index
Resolution [Å]
2Theta [°]

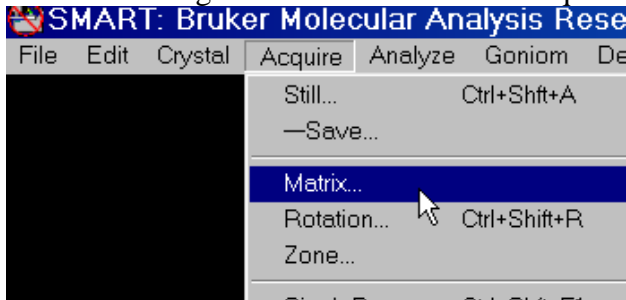
Image Header Tool Editor Cursor Position

SMART : Rotation Photo

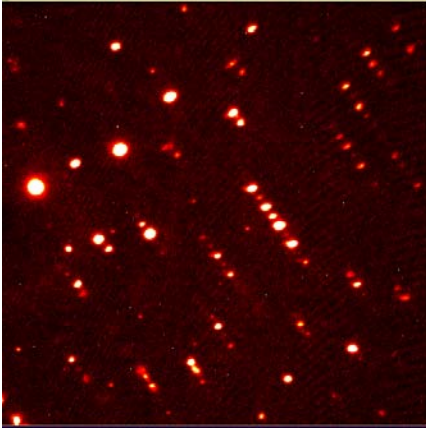
Take a rotation photo. Point to Acquire/Rotation point to OK



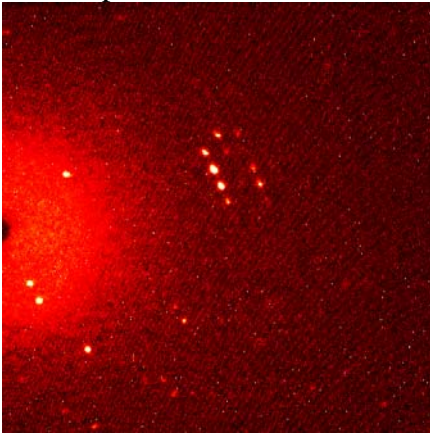
- Determining the Unit Cell. Point to Acquire/Matrix



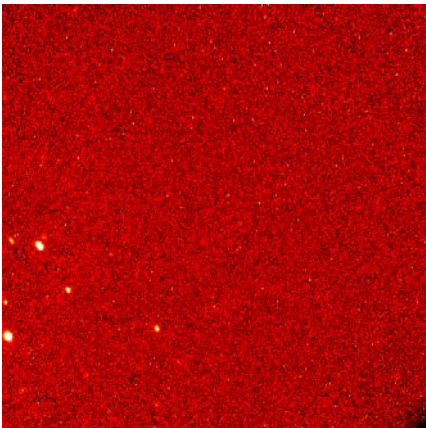
Good frame will look like this



An acceptable frame will look like this



A bad frame will look like this



- Unit cell

good cell

Lattice parameters & Standard deviations:

18.0820	18.6483	18.6871	90.050	90.054	89.958	6301.27
0.0052	0.0055	0.0082	0.029	0.034	0.023	3.94

Standard deviations corrected for GOF:

0.0039	0.0041	0.0061	0.021	0.025	0.017	2.94
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Histograms:

		.00	.05	.10	.15	.20	.25	.30	.35	.40	.45	+Inf
H	57	0	0	0	0	0	0	0	0	0	0	0
K	57	0	0	0	0	0	0	0	0	0	0	0
L	57	0	0	0	0	0	0	0	0	0	0	0
Omega	55	2	0	0	0	0	0	0	0	0	0	0

acceptable cell

Lattice parameters & Standard deviations:

11.2914	11.1571	15.0720	89.760	109.387	90.316	1791.07
0.0046	0.0044	0.0055	0.010	0.011	0.010	2.04

Standard deviations corrected for GOF:

0.0132	0.0128	0.0159	0.029	0.033	0.030	5.89
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Histograms:

		.00	.05	.10	.15	.20	.25	.30	.35	.40	.45	+Inf
H	207	0	0	0	0	0	0	0	0	0	0	0
K	207	0	0	0	0	0	0	0	0	0	0	0
L	201	6	0	0	0	0	0	0	0	0	0	0
Omega	87	70	28	17	2	1	1	1	0	0	0	0

a bad cell

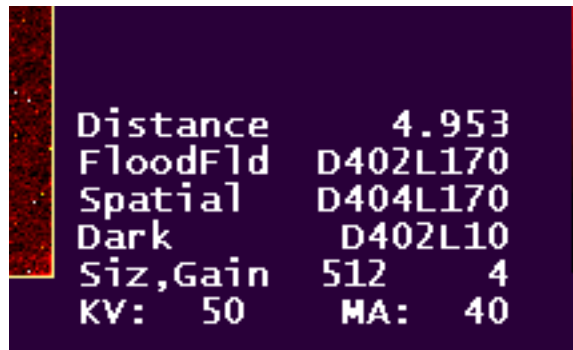
Lattice parameters & Standard deviations:

16.9188	16.7568	17.0997	86.145	86.462	86.316	4818.86
0.6570	0.3144	0.2764	1.543	1.477	0.801	325.74

Standard deviations corrected for GOF:

5.1056	2.4433	2.1478	11.988	11.475	6.226	2531.40
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- Crystal will not index
 - Bad Crystal?
 - check the distance with the detector distance on the instrument.
 - if there not the same the cell will not index properly



○

- Is it the right stuff?
Once you have the unit cell you can guess the Laue group and the Lattice. From this and the unit cell volume you should calculate the density.

$Z(1)$ = non-centrosymmetric: $Z(-1)$ = centrosymmetric

L = Lattice

System	Laue	L	$Z(1)$	$Z(-1)$
Triclinic	-1	P	1	2
Monoclinic	2/m	P	2	4
Monoclinic	2/m	C	4	8
Orthorhombic	mmm	P	4	8
Orthorhombic	mmm	C	8	16
Orthorhombic	mmm	A	8	16
Orthorhombic	mmm	I	8	16
Orthorhombic	mmm	F	16	32
Tetragonal	4/m	P	4	8
Tetragonal	4/m	I	8	16
Tetragonal	4/mmm	P	8	16
Tetragonal	4/mmm	I	16	32
Trigonal	-3	P	3	6
Trigonal	-3	R	9	18
Trigonal	-3m	P	6	12
Trigonal	-3m	R	18	36
Hexagonal	6/m	P	6	12
Hexagonal	6/mmm	P	12	24
Cubic	m-3	P	12	24
Cubic	m-3	I	24	48
Cubic	m-3	F	48	96
Cubic	m-3m	P	24	48
Cubic	m-3m	I	48	96
Cubic	m-3m	F	96	192

$$Density = \frac{FW \times Z}{0.6023 \times V}$$

Type	Density Limits g/ml
C-H	1.1 – 1.3
+ N	1.1 – 1.4
+ O	1.2 – 1.6
+ S	1.2 – 1.7
1 st row TR	1.4 – 1.9
+ Br	1.4 – 2.0
2 nd row TR	1.4 – 2.1
+ I	1.5 – 2.3
3 rd row TR	1.5 – 2.4
+Pb	1.6 – 2.9

For example our sugar molecule with a formula weight of 342 is monoclinic Laue group of 2/m P. The molecule is chiral so the space group will be non-centrosymmetric and $Z=2$. The cell volume is 715 which affords a density of 1.59 g/ml. The density fits the range shown in the table.

Start Data Collection

Point to Collect

Point to Experiment

Find short_runs

Point to Load Table

Operation	Active	3	4	5	6	7	8	9	10	11
1	No Operation	Yes								
2	No Operation	Yes								
3	No Operation	Yes								
4	No Operation	Yes								
5	No Operation	Yes								
6	No Operation	Yes								
7	No Operation	Yes								
8	No Operation	Yes								
9	No Operation	Yes								
10	No Operation	Yes								
11	No Operation	Yes								
12	No Operation	Yes								
13	No Operation	Yes								
14	No Operation	Yes								
15	No Operation	Yes								
16	No Operation	Yes								
17	No Operation	Yes								
18	No Operation	Yes								
19	No Operation	Yes								
20	No Operation	Yes								
21	No Operation	Yes								
22	No Operation	Yes								
23	No Operation	Yes								
24	No Operation	Yes								
25	No Operation	Yes								
26	No Operation	Yes								
27	No Operation	Yes								

Data file name

Change Data collection time

Start Data Collection

Operation	Active	Distance [mm]	2Theta [deg]	Omega [deg]	Phi [deg]	Chi [deg]	Time [sec]	Width [deg]	Sweep [deg]	Direction
1	Generator	Yes	Voltage [kV]	50.000	Current [mA]	40.000				
2	Omega Scan	Yes	60.000	-28.000	-28.000	0.000	54.736	default	default	180.000 negative
3	Omega Scan	Yes	60.000	-28.000	-28.000	90.000	54.736	default	default	180.000 negative
4	Omega Scan	Yes	60.000	-28.000	-28.000	180.000	54.736	default	default	180.000 negative
5	Omega Scan	Yes	60.000	-28.000	-28.000	270.000	54.736	default	default	180.000 negative
6	No Operation	Yes								
7	No Operation	Yes								
8	No Operation	Yes								
9	No Operation	Yes								
10	No Operation	Yes								
11	No Operation	Yes								
12	No Operation	Yes								
13	No Operation	Yes								
14	No Operation	Yes								
15	No Operation	Yes								
16	No Operation	Yes								
17	No Operation	Yes								
18	No Operation	Yes								
19	No Operation	Yes								
20	No Operation	Yes								
21	No Operation	Yes								
22	No Operation	Yes								
23	No Operation	Yes								
24	No Operation	Yes								
25	No Operation	Yes								
26	No Operation	Yes								
27	No Operation	Yes								

Data Collection SMART

point to **Acquire/EditMulti**

Generic Data Collection Strategy

#	2Theta	Omega	Phi	Chi	Axis	Range	#	Time
1	-28	-28	0	54.9	2	-0.3	600	20
2	-28	-28	90	54.9	2	-0.3	600	20
3	-28	-28	180	54.9	2	-0.3	400	20
4	-28	-28	270	54.9	2	-0.3	400	20

About a 12 hour data collection

How fast can you go

Collect a few frames. **Acquire/Multirun**

If you do not see spots near the right hand edge of the frame then
slow the data collection to 30 secs and repeat the collection.

Load the correct dark current for your data collection time

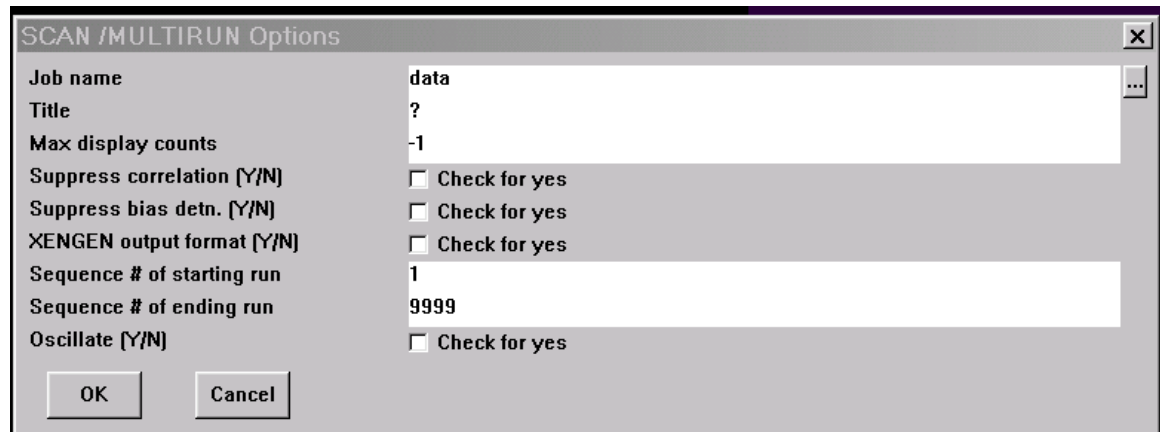
point to **Detector/Dark Current and Load**

the look for *****020.*** for 20 secs ***30.*** for 30 etc.

Start Data Collection

point to **Acquire/MultiRun**

set the JOB name (normally just DATA) and point to OK.



1. Crystal Face

Unit Cell

a=31.93Å, b=16.31Å, c=16.35Å
 $\alpha=90.00^\circ$, $\beta=94.27^\circ$, $\gamma=90.00^\circ$
 V=8489Å³, Monoclinic C

H	K	L	Distance [nm]
0	1	0	0.21
0	-1	0	0.26
0	0	1	0.03
0	0	-1	0.02
1	0	0	0.05
-1	0	0	0.07

Crystal Face
 Miller Indices: 0 0 1
 Distance [nm]: 0.26
 Out-of-plane angle [deg]: 0.74

Max. Miller index: 2
 Max. out-of-plane angle [deg]: 1

Size [mm]: 0.06 x 0.14 x 0.52

Unit Cell

a=31.93Å, b=16.31Å, c=16.35Å
 $\alpha=90.00^\circ$, $\beta=94.27^\circ$, $\gamma=90.00^\circ$
 V=8489Å³, Monoclinic C

H	K	L	Distance [nm]
0	1	0	0.21
0	-1	0	0.26
0	0	1	0.03
0	0	-1	0.02
1	0	0	0.05
-1	0	0	0.07

Crystal Face
 Miller Indices: n/a n/a n/a
 Distance [nm]: n/a
 Out-of-plane angle [deg]: n/a

Max. Miller index: 2
 Max. out-of-plane angle [deg]: 1

Size [mm]: 0.06 x 0.14 x 0.52