

SMART 100CCD Diffractometer Operation Summary

02/14/2012 A.E.

Location: 1709 Gilman Hall

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Safety

All users of SMART 1000 DDC must complete the EH&S "X-ray Safety Fundamentals" course available on-line at www.ehs.iastate.edu. The valid "X-ray Safety Fundamentals" Certificate is mandatory to enter 1709 Gilman Hall X-ray lab.

This class demonstrates the correct procedures for the authorization and use of X-ray producing devices. Topics include X-ray production and hazards, hazard control measures, and regulatory requirements. Certification is awarded upon successful completion of the quiz at the end of the course. You must recertify every year.

All diffractometer enclosures in CIF X-Ray Laboratory are checked by EH&S annually and certified for data collection operations. All systems are also equipped with multiple interlock switches blocking the X-ray shutter to avoid any possible operator exposure.

Training Requirements

Before operating diffractometer, you **MUST** be properly trained and authorized by CIF Supervisor of the X-ray lab!

This training manual includes information about the diffractometer and step-by-step instructions on sample preparation and data collection.

All referenced documents, including the detailed "SMART Software Manual" and the "SMART User's Manual" are available in 1709 Gilman Hall.

1. Before you start

- 1.1. Sign up in the LogBook of SMART CCD
- 1.2. Log in Windows NT on Bruker Frame Buffer PC running under Windows NT:
LOGIN: YOUR LOGIN
PASSWORD: YOUR PASSWORD

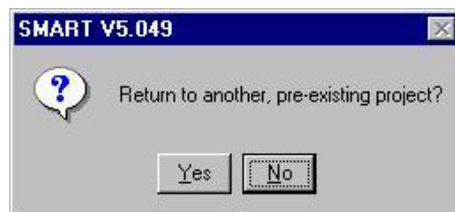
2. Running SMART Software

- 2.1. Click **SMART** icon on a desktop
- 2.2.



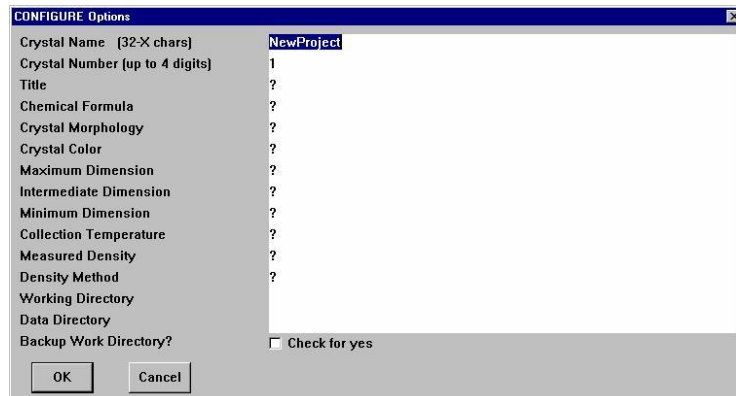
Click **NO**

- 2.3



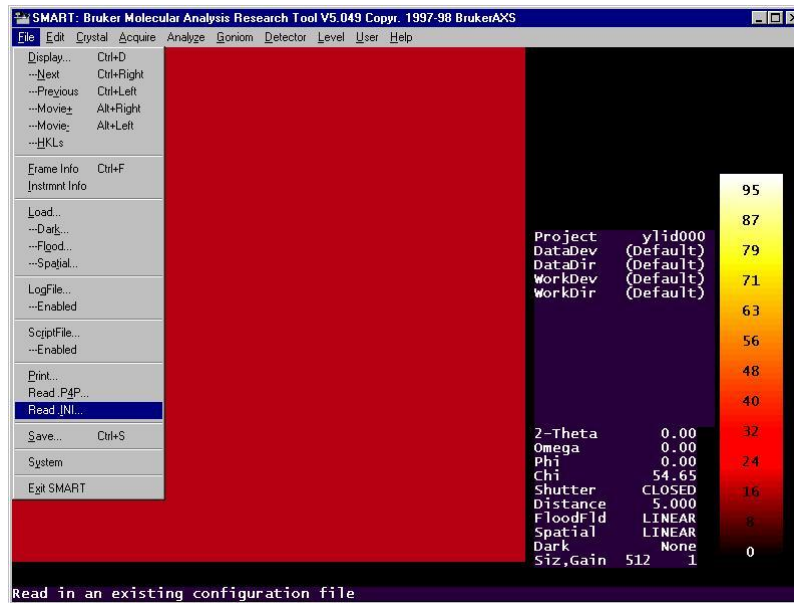
Click **NO**

- 2.4



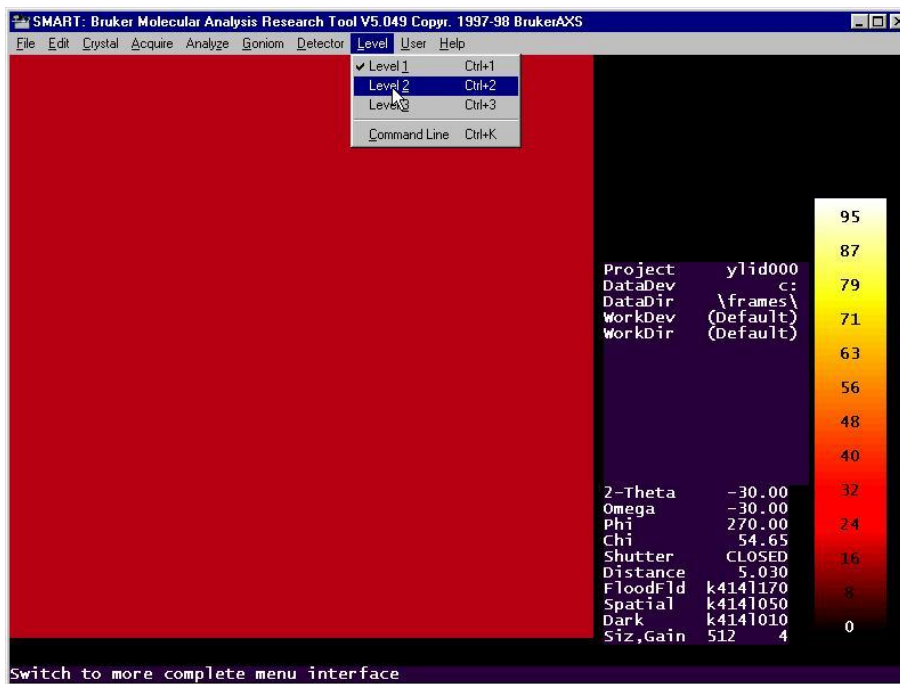
Click **CANCEL**

2.5 Read default configuration file

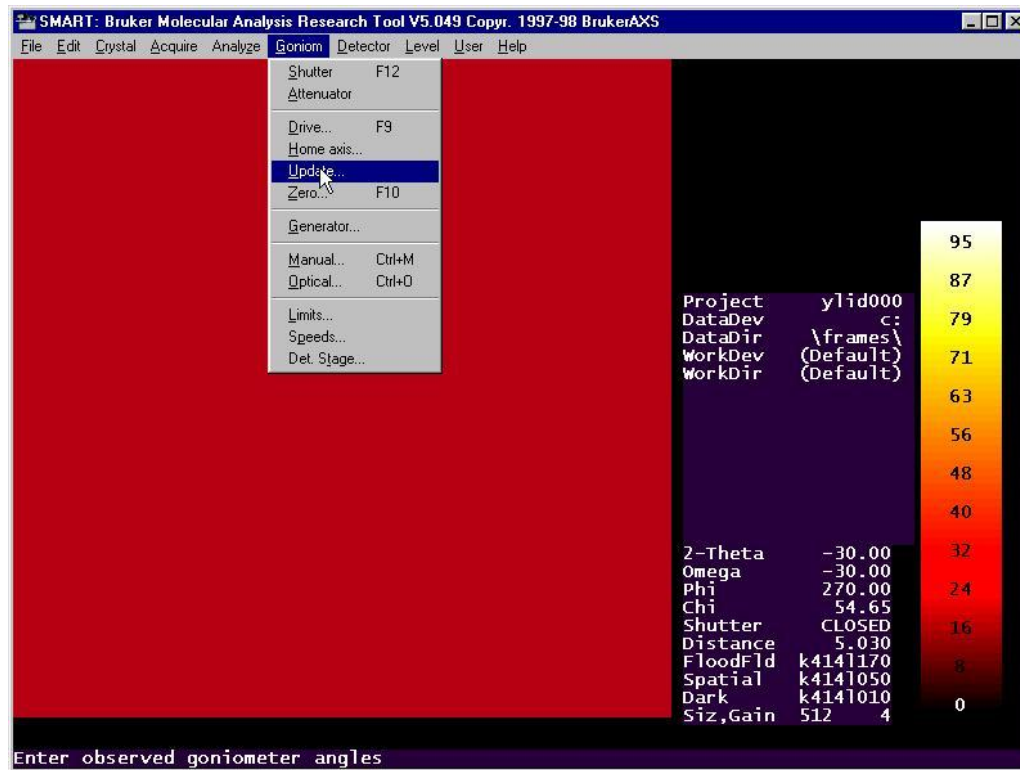


Click **OK**

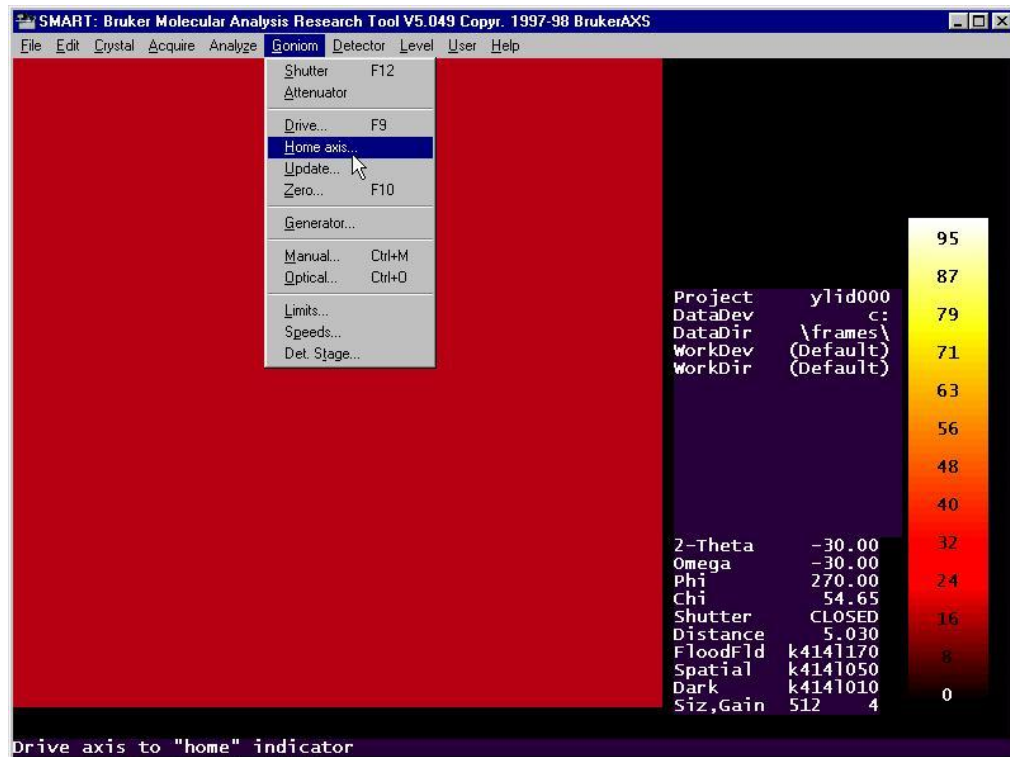
2.6 Change the priority to Level2. Caution!!! You can really make damage of the system at this level



2.7 Update angles.



2.8 Home axes 1.

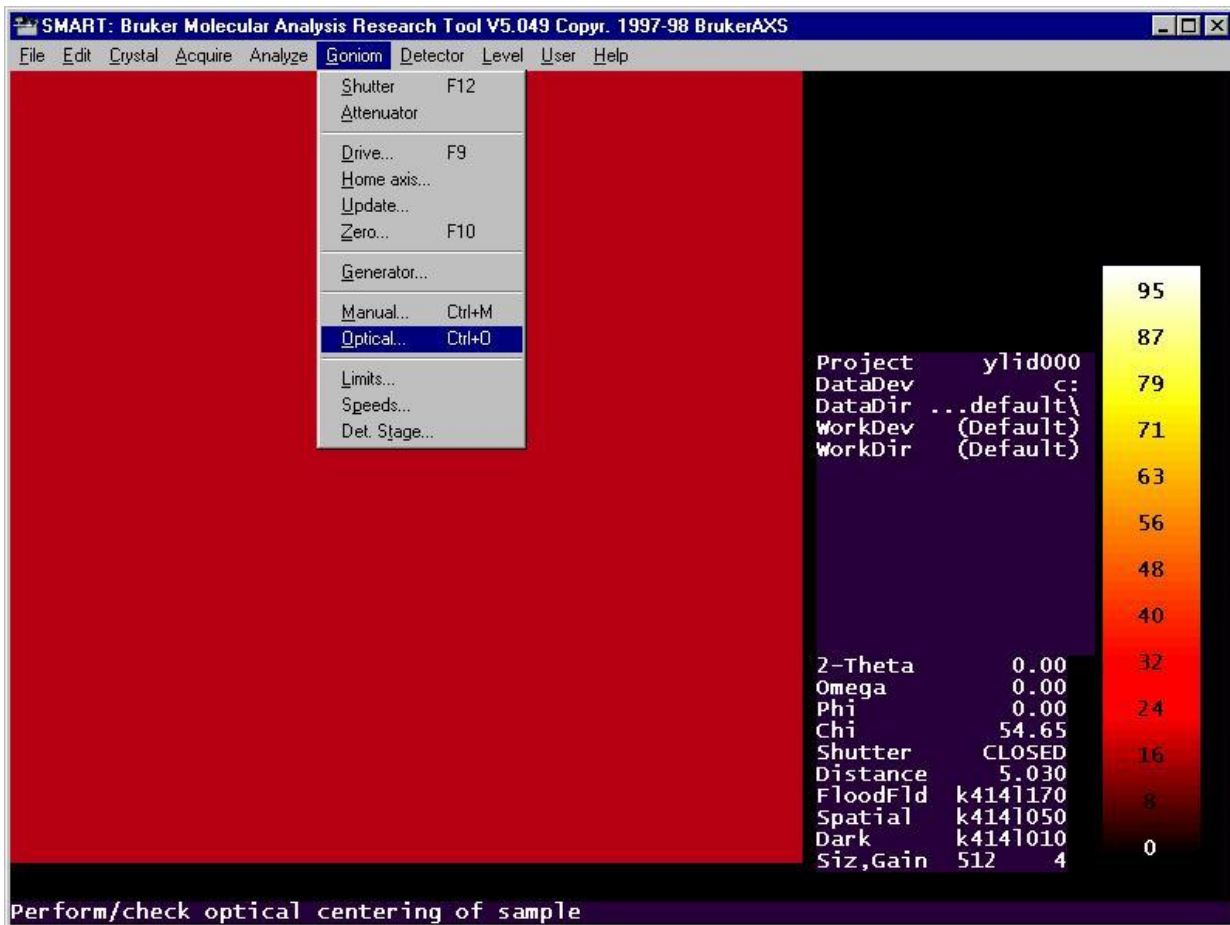




Click **OK** and repeat 2.8 another two times for Axes2 and Axis3.

3. Crystal Optical Alignment

3. 1

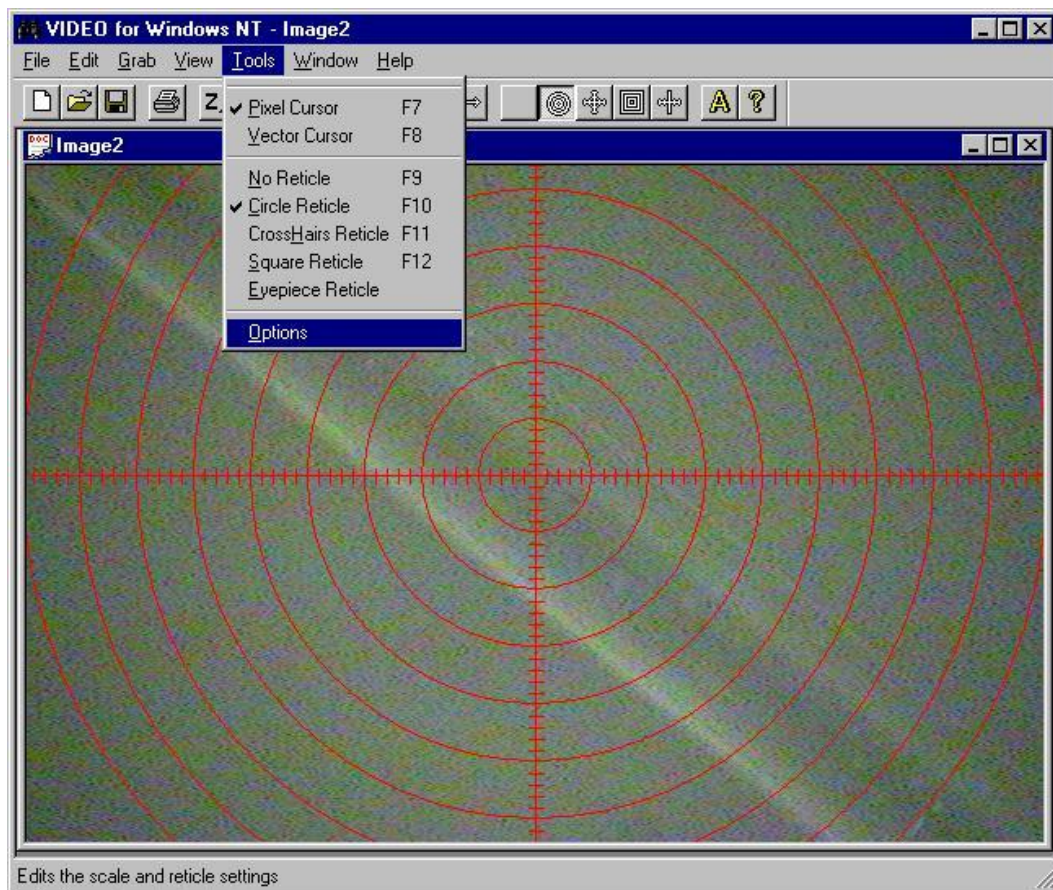


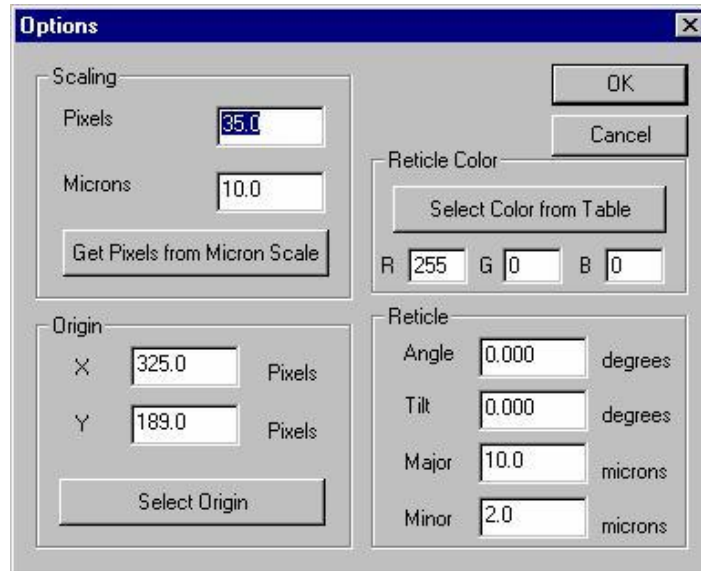
3. 2



Click OK

3. 3 Click **VIDEO** icon on a desktop and open new image: File-New Image
Click on a green arrow on a toolbar to grab the image.





Click **OK**

- 3.4 Using the remote control box press A button and then press Axis Print button. That makes goniometer drive to initial alignment position. Do not attempt to mount a crystal in any other goniometer position. For better view place the white paper on a pin above CCD safety screen.
- 3.5 Adjust the height and horizontal position (located perpendicular to the camera)
- 3.6 Measure two crystal dimensions on a screen using \leftrightarrow arrow
- 3.7 Press **Axis Print** button to drive the PHI axis 180 deg. And check the alignment
- 3.8 Press **B** button and then Axis Print (turns PHI axis 90 deg.) to align crystal in the other direction
- 3.9 Measure the third crystal dimension on screen by using the \leftrightarrow arrow
- 3.10 Press **Axis Print** button to drive the PHI axis 180 deg. And check the alignment
- 3.11 Press **D** button and then Axis Print and verify the height position of the crystal.
- 3.12 Repeat steps 4-12 if required
- 3.13 Close the enclosure of the cabinet and press Shutter Reset button.
- 3.14 Click on a **SMART** window on a PC screen and press ESC to quit the manual/optical alignment mode.

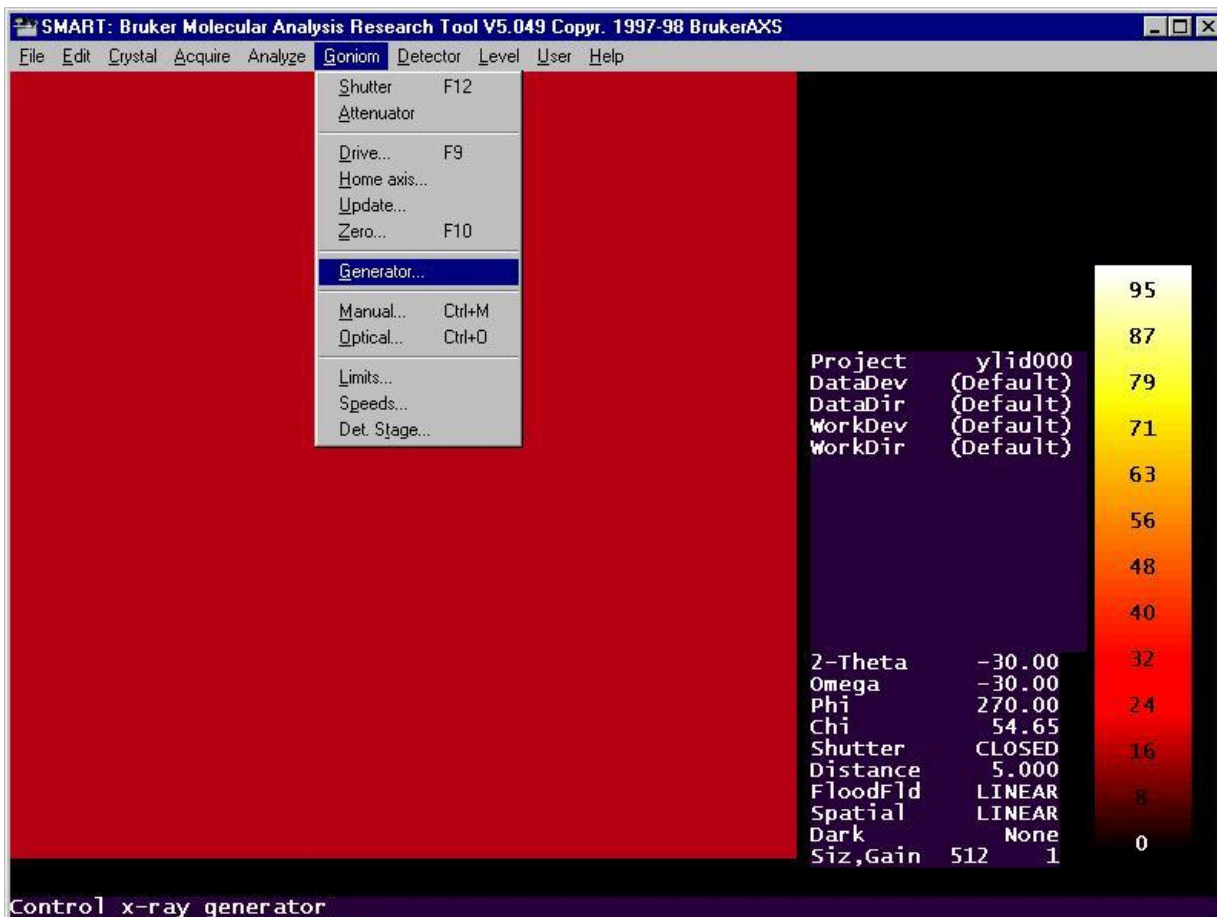
3.15 Close the **VIDEO** window. It takes a lot of memory and should be closed during all other procedures but centering the crystal.



Click **NO**

3. Rotation Photo

4.1 Check the high Voltage on X-ray tube. The default values are shown below.



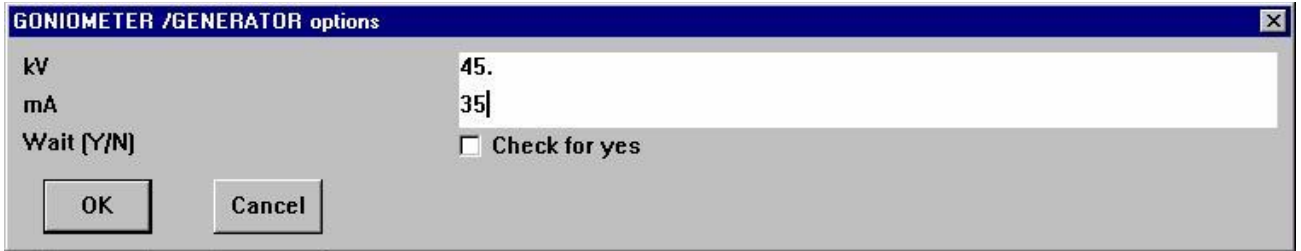
SMART: Bruker Molecular Analysis Research Tool V5.049 Copyr. 1997-98 BrukerAXS

File Edit Crystal Acquire Analyze **Goniom** Detector Level User Help

Shutter F12
Attenuator
Drive... F9
Home axis...
Update...
Zero... F10
Generator...
Manual... Ctrl+M
Optical... Ctrl+D
Limits...
Speeds...
Det. Stage...

Project ylid000
DataDev (Default)
DataDir (Default)
WorkDev (Default)
WorkDir (Default)
2-Theta -30.00
Omega -30.00
Phi 270.00
Chi 54.65
Shutter CLOSED
Distance 5.000
FloodFld LINEAR
Spatial LINEAR
Dark None
Siz,Gain 512 1

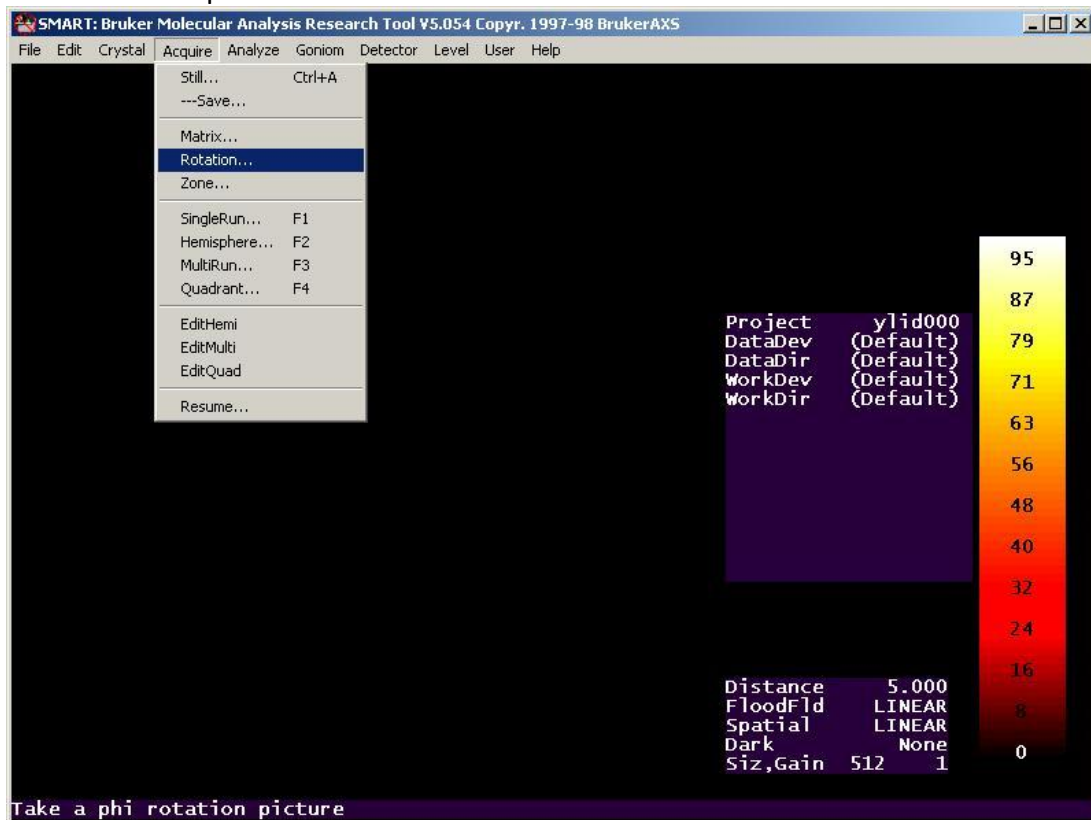
Control x-ray generator



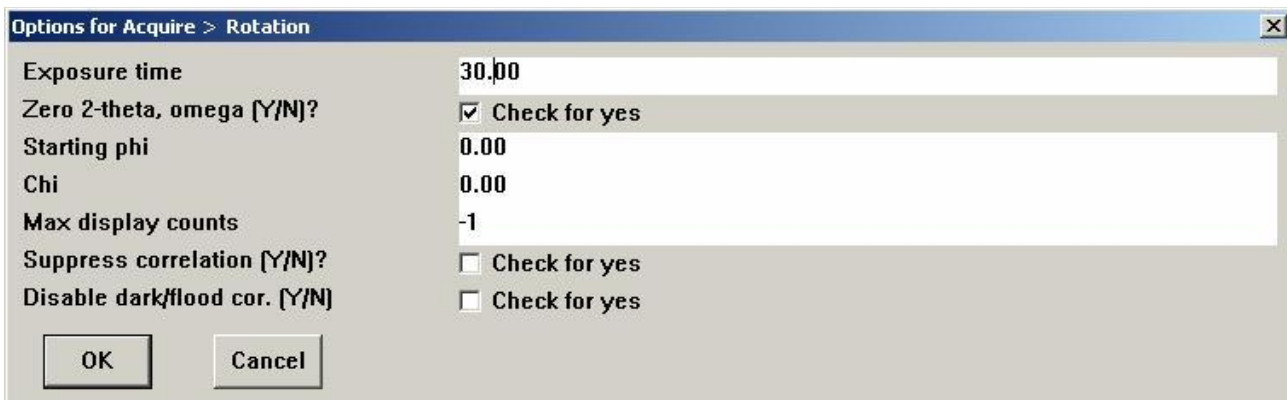
The maximum values 50 KV / 40mA could be used only as exception and this requires consultation with Arkady Ellern tel. 4-5976, 1711 Gillman Hall

Click **OK**

4.2 Make rotation photo.



Exposure time depends upon the scattering of the sample and should be in the range 12-60 sec. The standard value is 30 sec and it has to produce distinct picture.

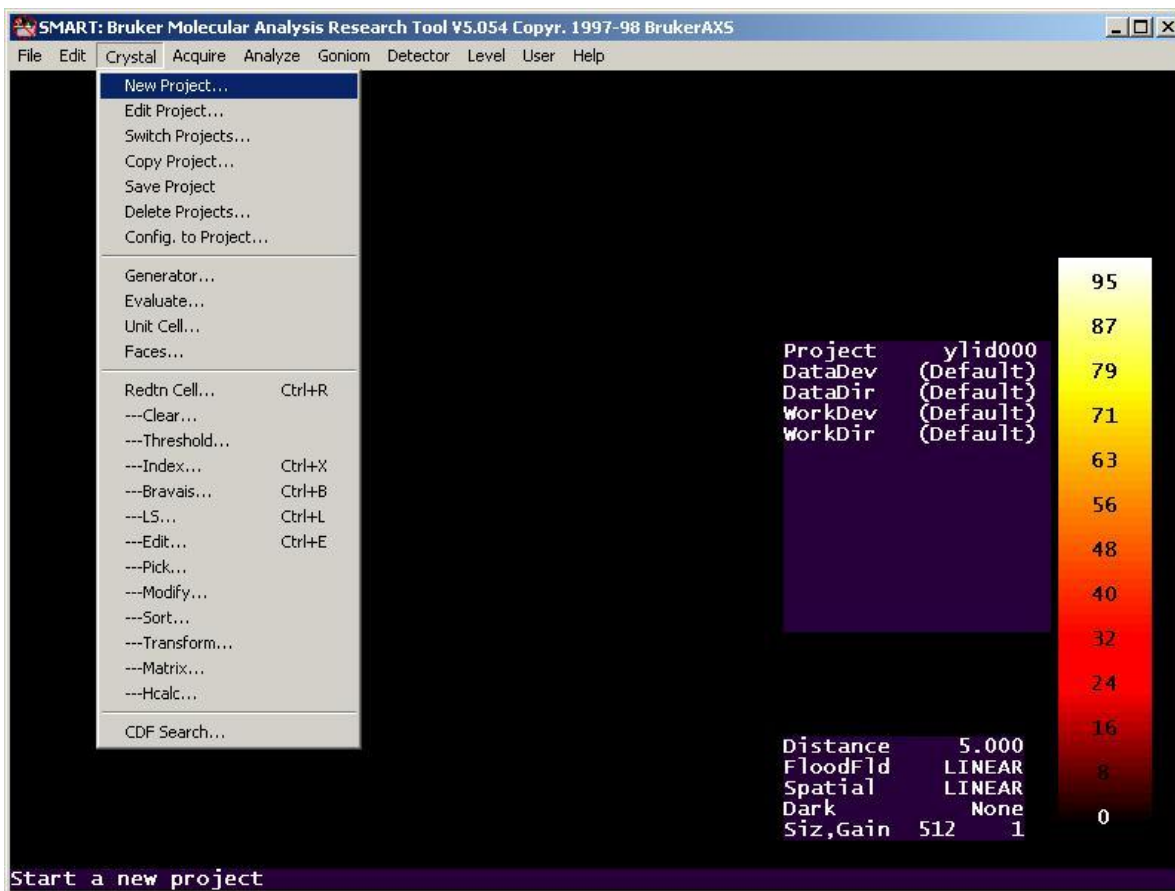


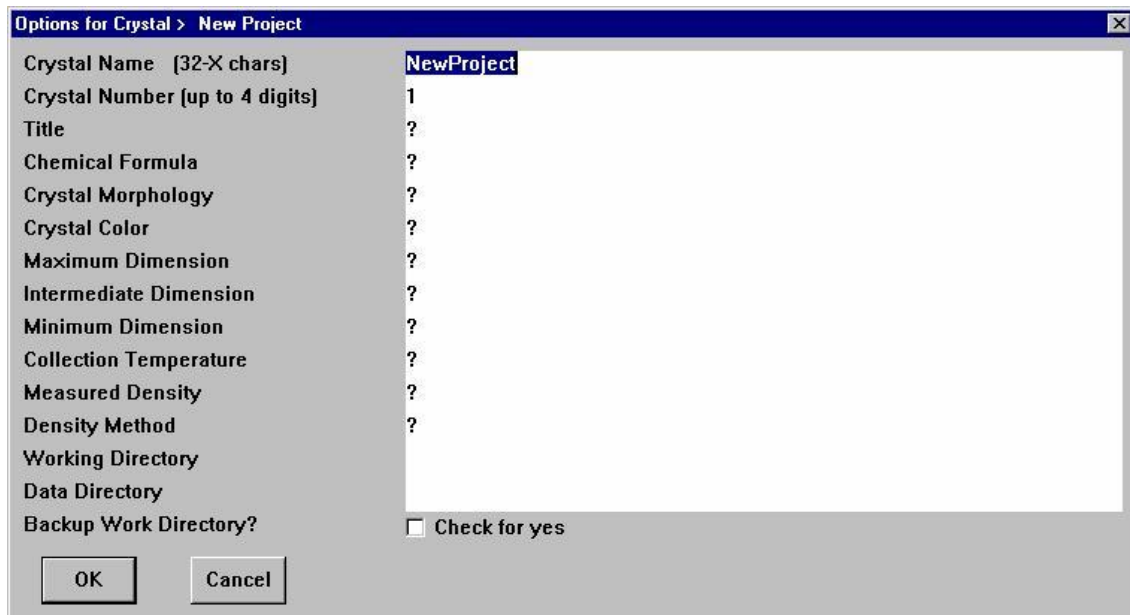
Only if rotation photo indicates the suitable quality of the sample for further attempts one should go for the next step of screening of the quality of the crystal.

Click OK

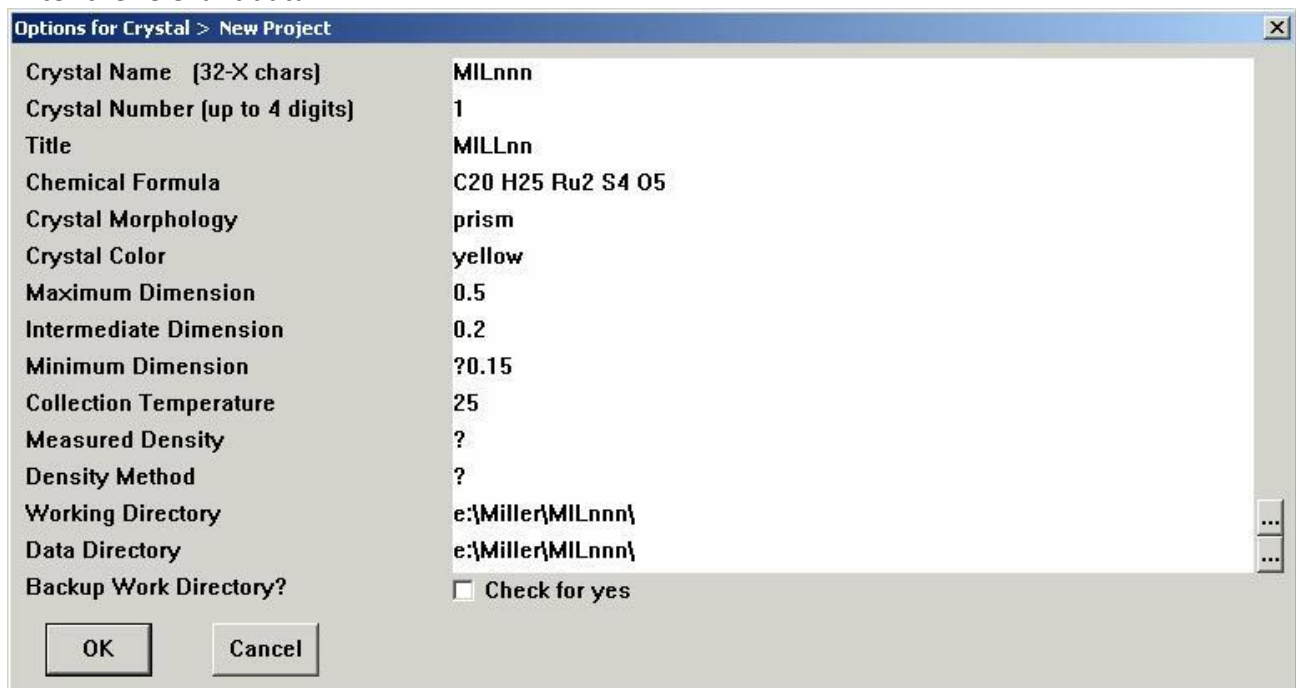
4. Starting New Project

4.1





Enter the relevant data.



Click **OK**

4.2 Important!!!

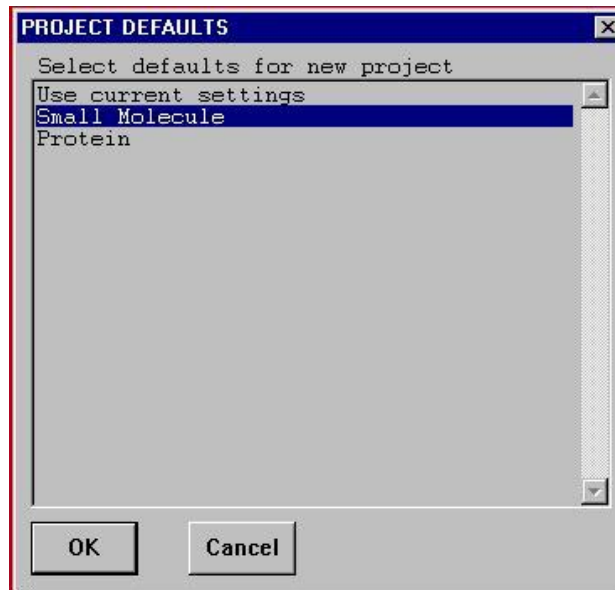
Name and number samples sequentially with your group code

SADnnn , directory e:\Sadow\Sadnnn for Sadow Group

Click **OK**

Save current Configuration **NO**

Clear the face list.....and orientation- **YES**

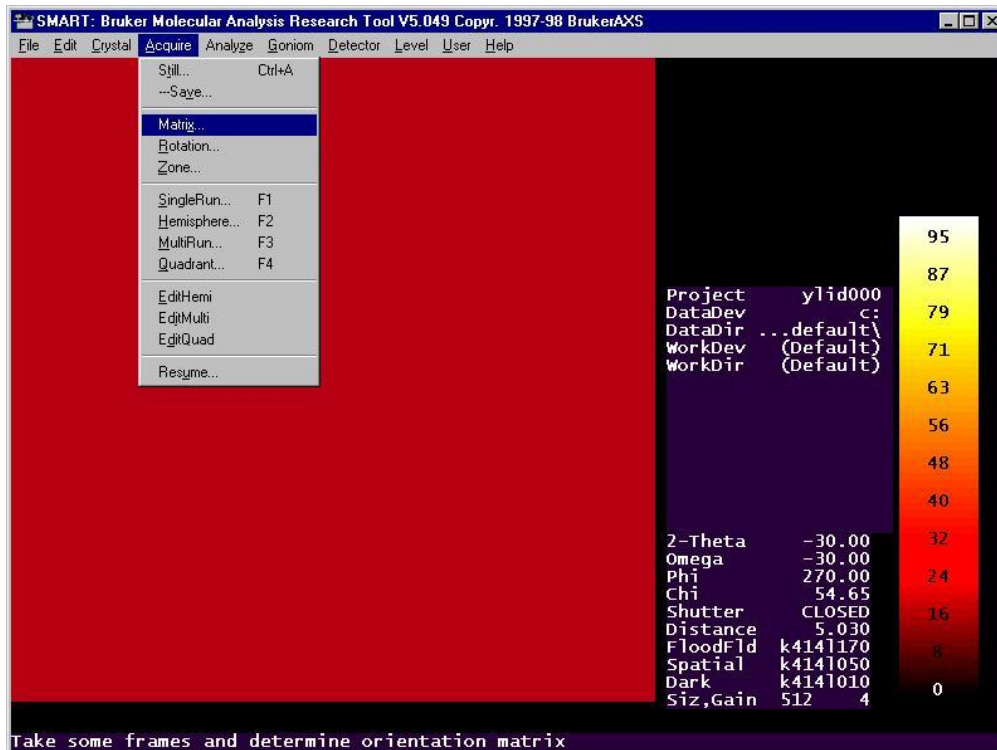


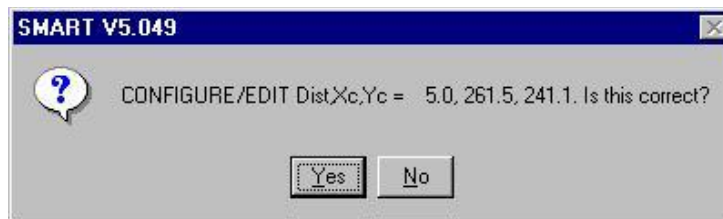
Click OK

4.3 The directory E:\..... does not exist. Create it? - YES

5. MATRIX routine

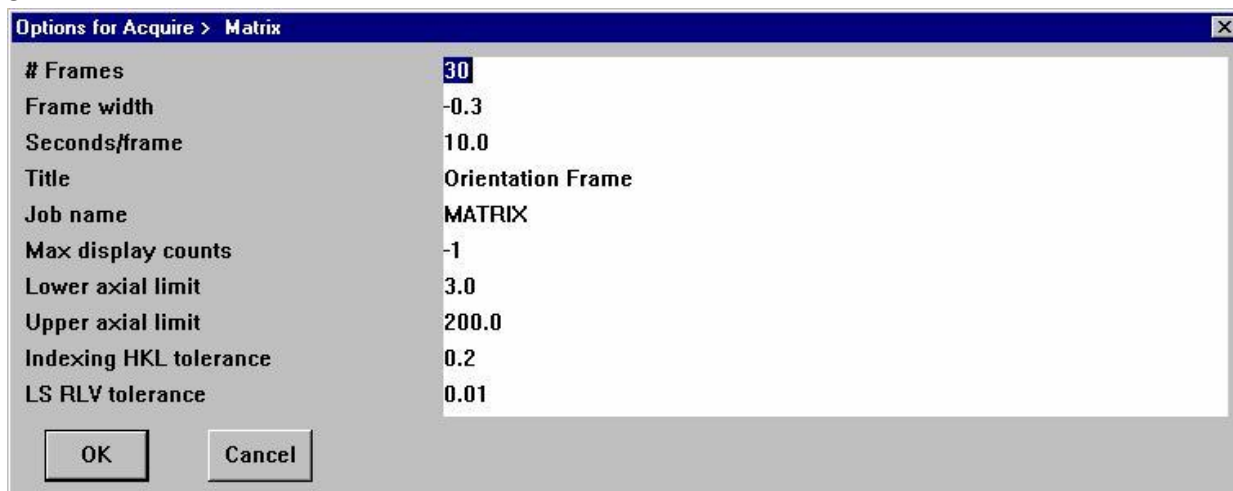
5.1





Click **YES**

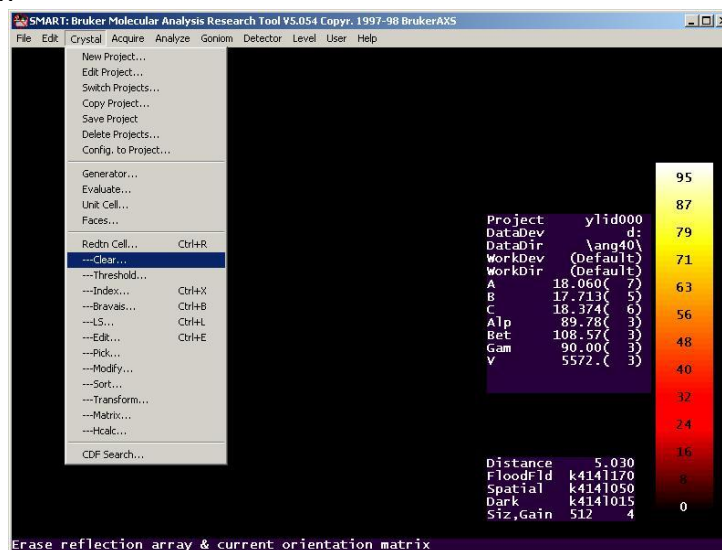
5.2



Click **OK** to make 3 sets of frames to obtain orientation matrix and cell parameter as well as a Bravais lattice. This is a “black-box” closed routine and it works only with a good single crystal. If this procedure fails one should be ready to switch to manual analysis of frames

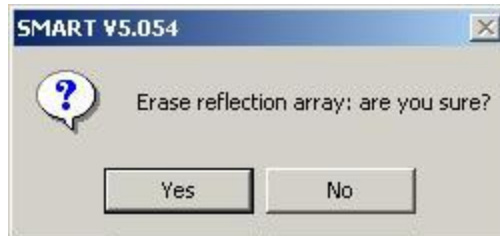
6. Manual Indexing, LS and Bravais Lattice Determination

6.1 Try to reindex



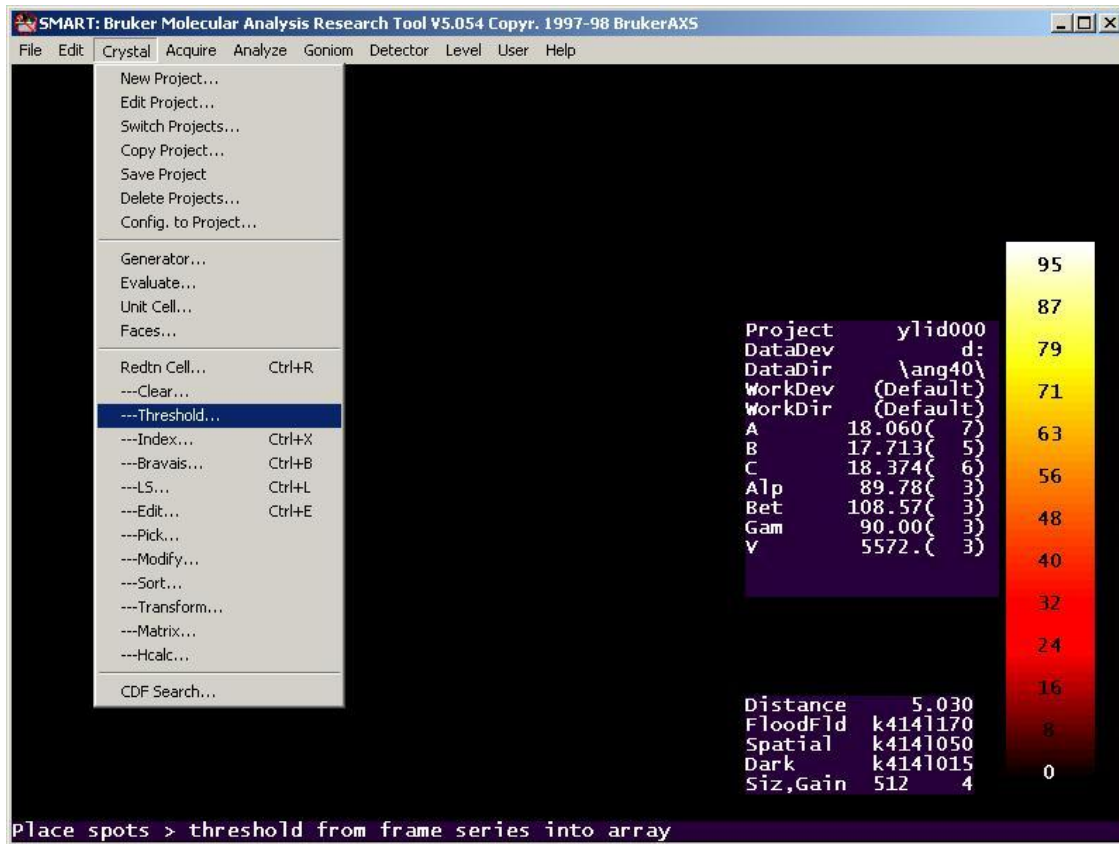


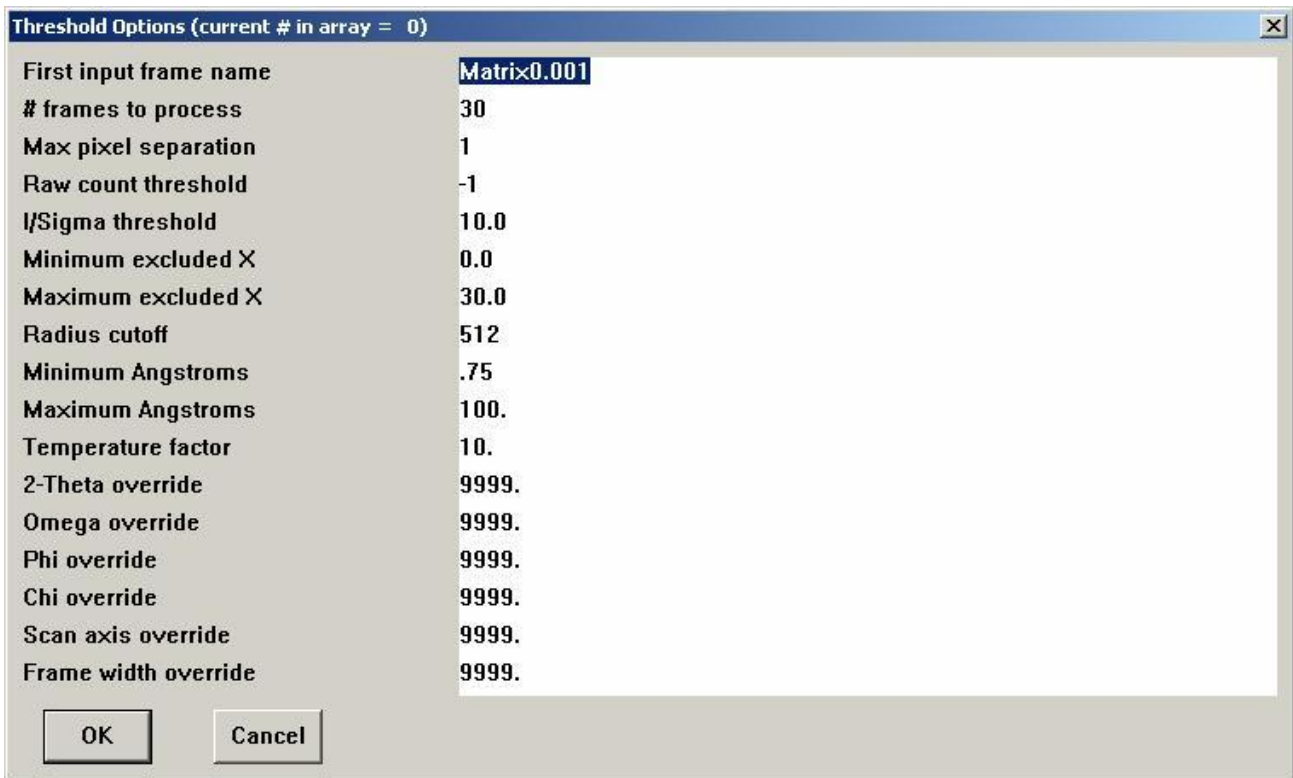
Click **OK**



Click **YES**

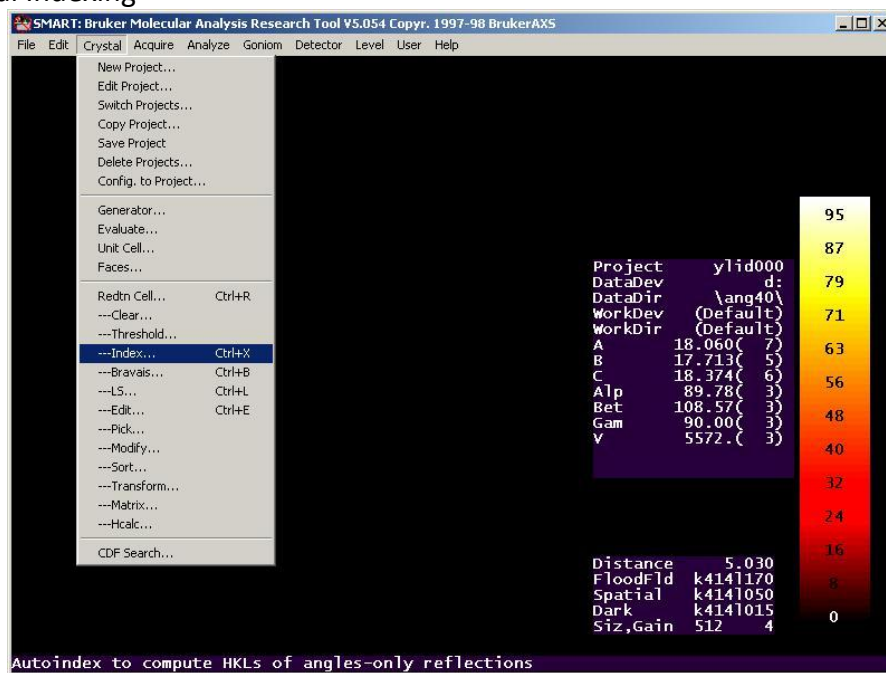
6.2 Threshold data frames

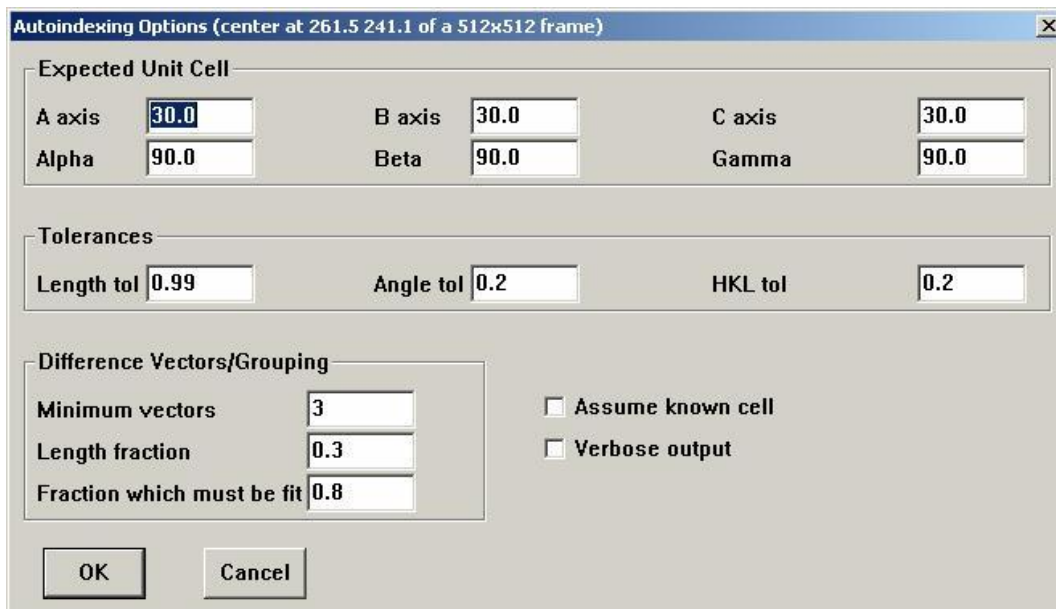




Click **OK**
 Click **OK**
 Click **YES**

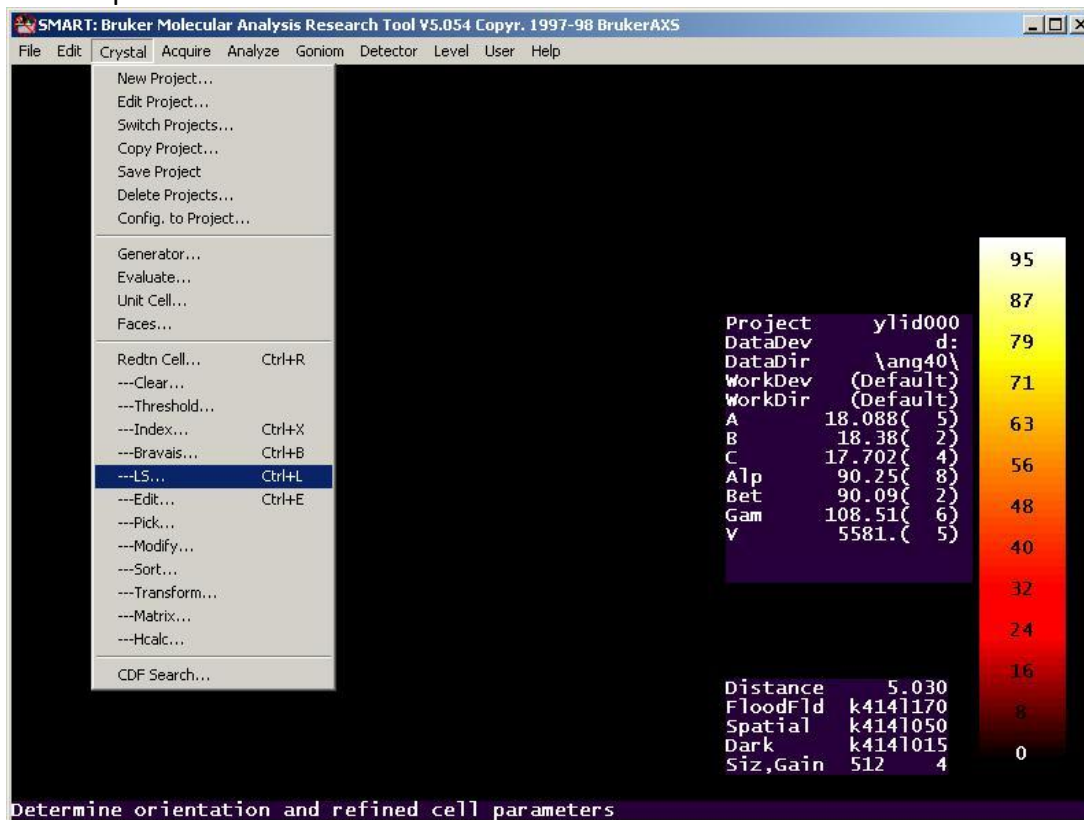
6.3 Manual Indexing

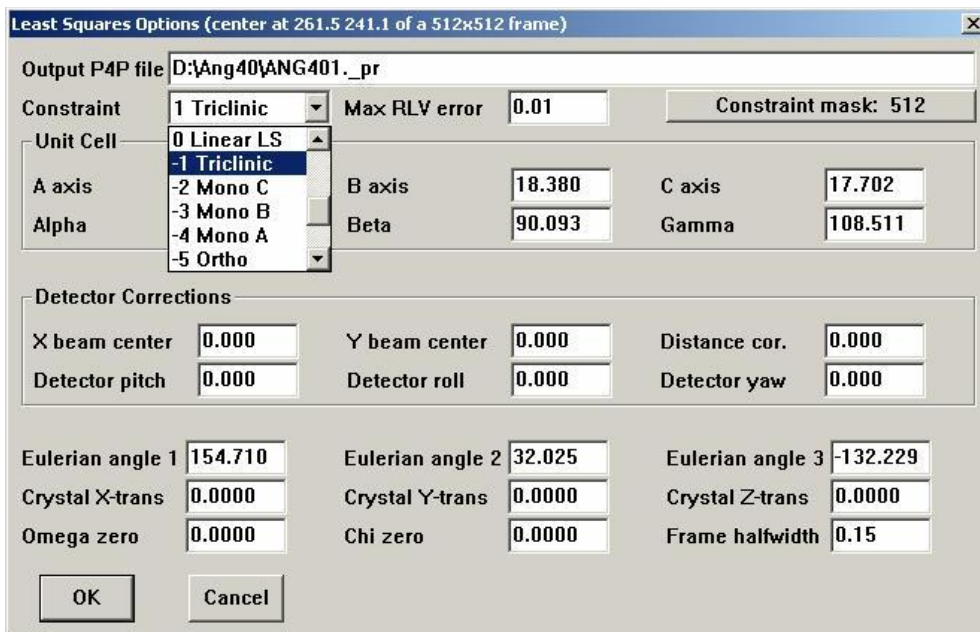




Check the default values and click **OK, OK, YES, OK**

6.4 Least Squares





Click **OK**

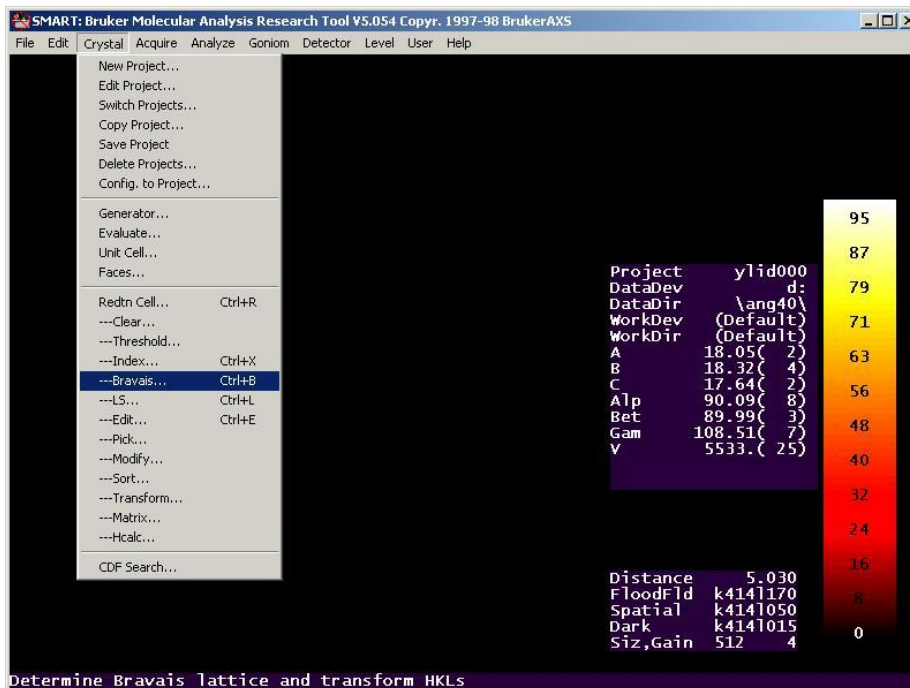
Click **OK**

Click **OK**

Click **YES**

Repeat this step gradually reducing RVL to 0.0025 in 2-3 steps. Unit cell constraint **MUST** be -1

6.5 Bravais Lattice Determination



Bravais Lattice Options

Lattice type: **P** Max sigmas, any: **20.0** Max sigmas, best: **6.0**

Input Unit Cell

Current A axis: **18.050** Current B axis: **18.319** Current C axis: **17.645**
 Current ALPHA: **90.085** Current BETA: **89.987** Current GAMMA: **108.510**

Input Standard Deviations

ESD of A: **0.002** ESD of B: **0.002** ESD of C: **0.002**

OK **Cancel**

Bravais Lattice Output

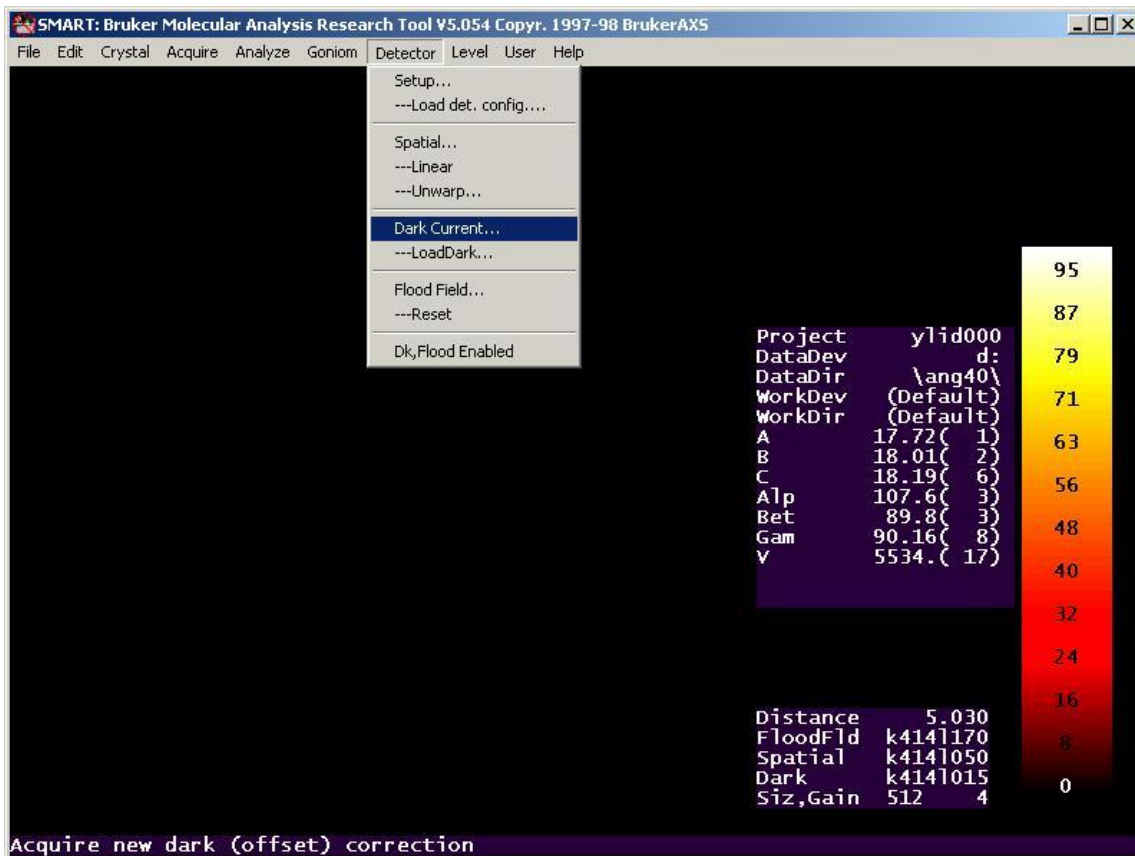
```
#Sigmas for acceptance of any soln, best soln:      20.0      6.0
Input lattice type: P
Input cell and standard deviations:
  18.088  18.380  17.702  90.249  90.093  108.511      5580.58
   0.005  0.005  0.005
Number of solutions = 2
# #Sig A B C Alpha Beta Gamma
MONOCLINIC P
1 11.83 18.088 17.702 18.380 90.249 108.511 90.093
-1.00 0.00 0.00/ 0.00 0.00 -1.00/ 0.00 -1.00 0.00/
TRICLINIC P
>>> 2 0.00 17.702 18.088 18.380 108.511 90.249 90.093
0.00 0.00 1.00/ 1.00 0.00 0.00/ 0.00 1.00 0.00/
```

OK **Print...** **Write...**

Click **OK**.. Refer to manual or ask for additional consultation.

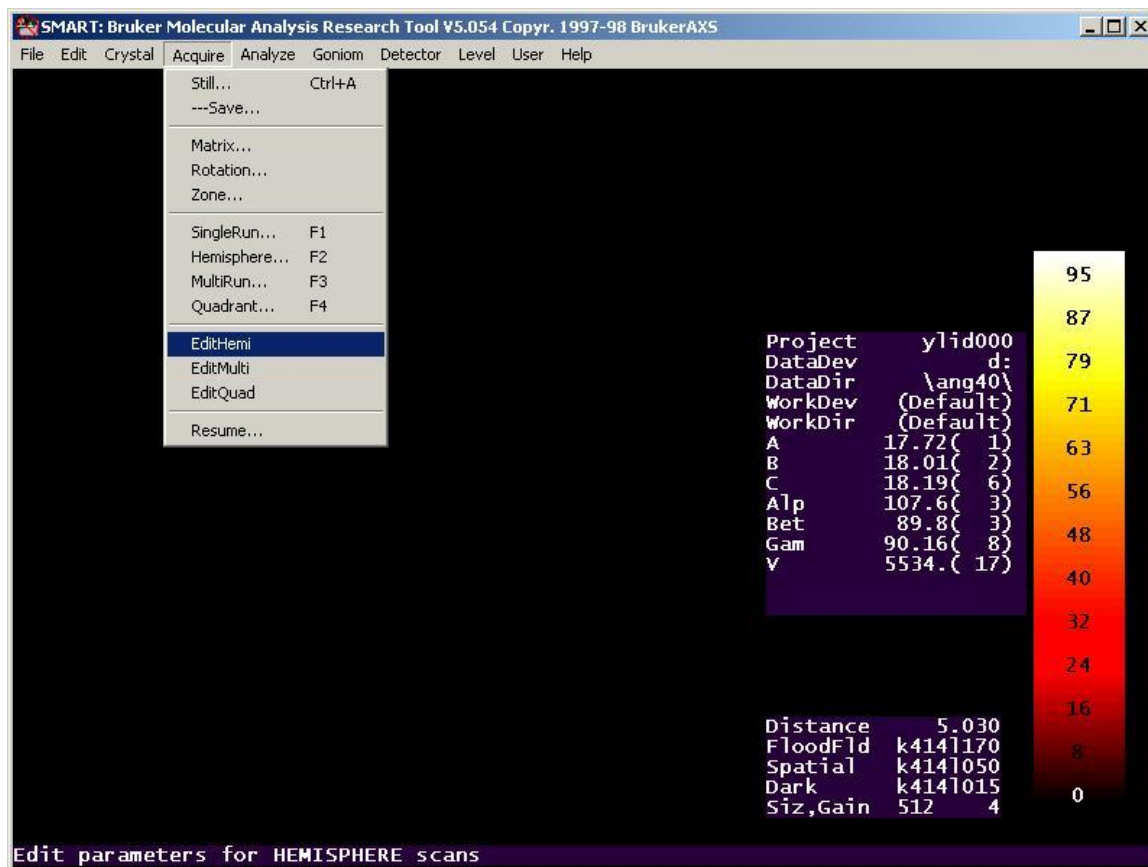
7. Data Collection

7.1 Calculate the new Dark Frame



Click **OK** two times

7.2 For monoclinic and higher cells it is possible to use hemisphere data collection.

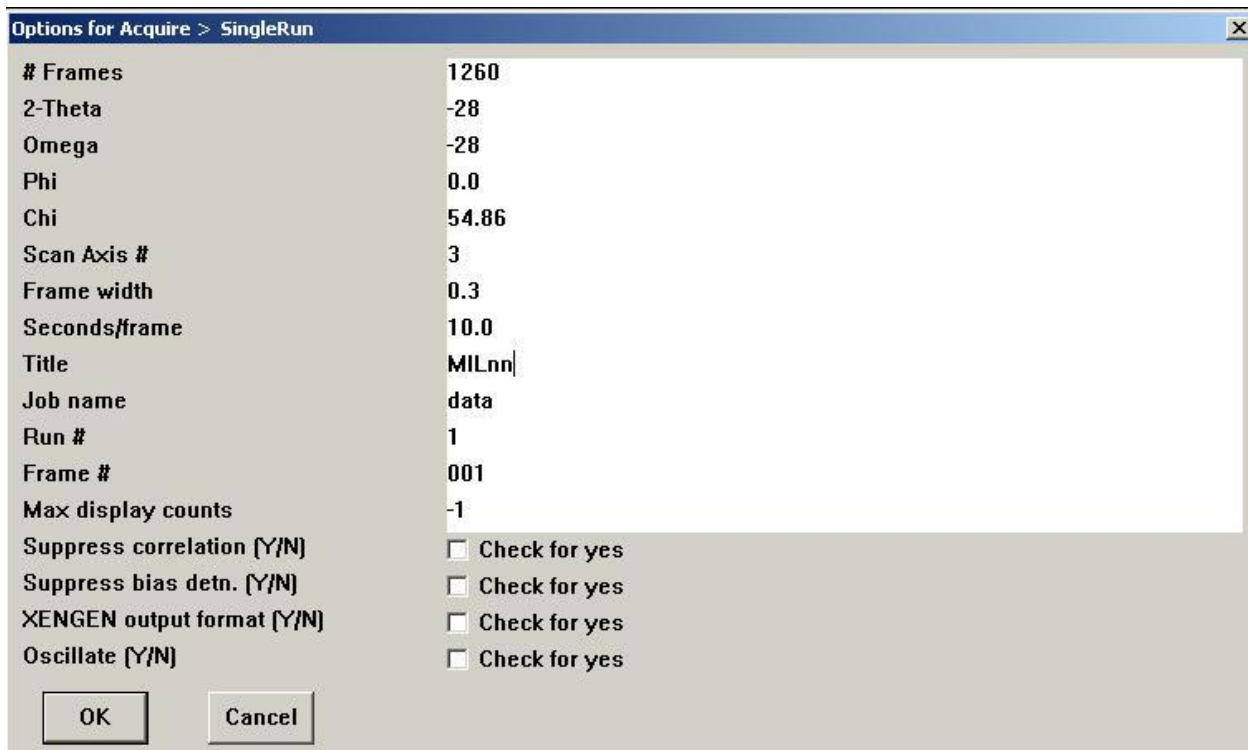
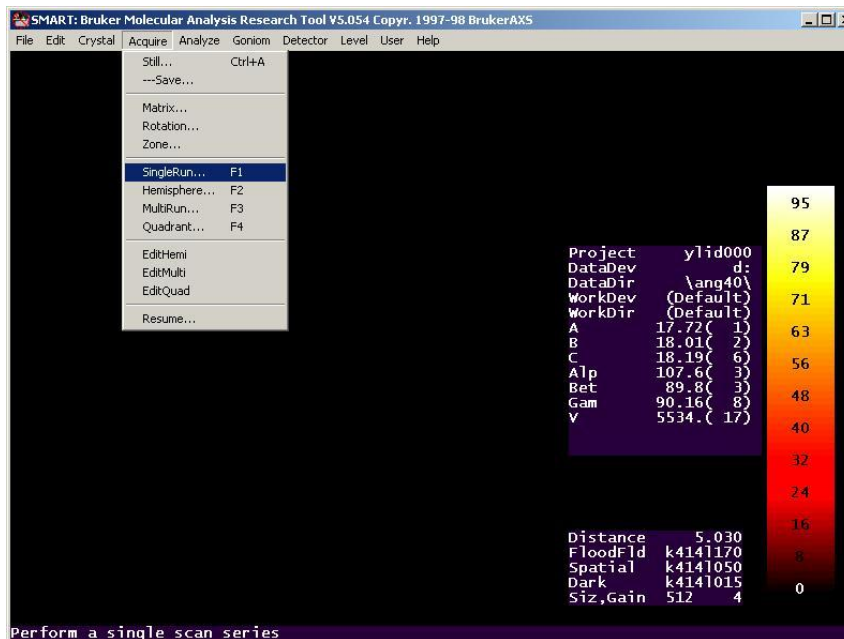


SCAN Hemisphere Run List (8 lines)

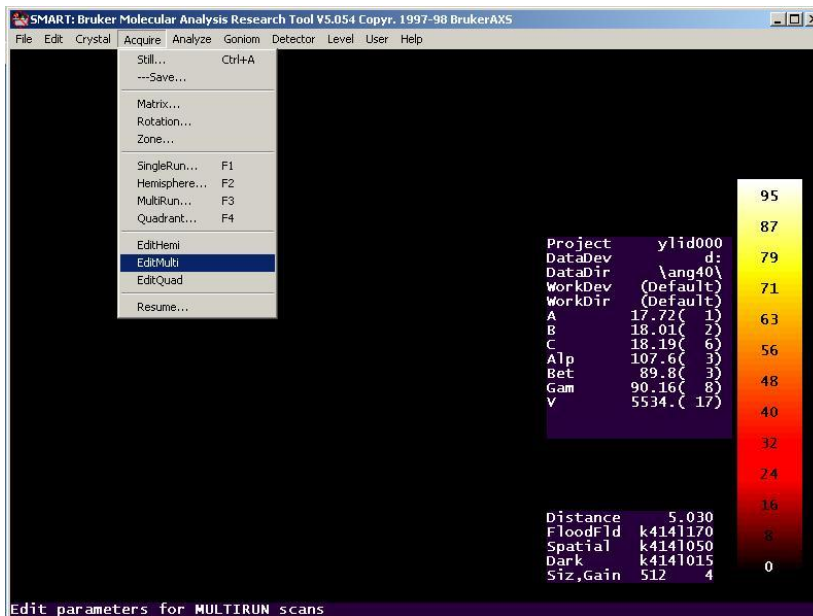
Run#	Frame#	2-Theta	Omega	Phi	Chi	Axis	Width	#Frames	Time
1	001	-28.00	-28.00	0.00	54.86	2	-0.300	606	15.00
2	001	-28.00	-28.00	90.00	54.86	2	-0.300	435	15.00
3	001	-28.00	-28.00	180.00	54.86	2	-0.300	230	15.00
4	001	-28.00	-28.00	0.00	54.86	2	-0.300	50	15.00

OK Cancel Print... Write... Read...

7.3 For crystals in capillaries it is possible to use PHI scan



7.4 For the triclinic crystals and for the best quality of data full sphere has to be collected.



SCAN MultiRun List (50 lines)

Run#	Frame#	2-Theta	Omega	Phi	Chi	Axis	Width	#Frames	Time
1	001	-28.00	-28.00	0.00	54.86	2	-0.300	600	15.00
2	001	-28.00	-28.00	90.00	54.86	2	-0.300	600	15.00
3	001	-28.00	-28.00	180.00	54.86	2	-0.300	600	15.00
4	001	-28.00	-28.00	270.00	54.86	2	-0.300	600	15.00

Buttons: OK, Cancel, Print..., Write..., Read...

Go to 4.1 and change the voltage on a X-ray tube to **20Kv** and **5 mA** AFTER FINISHING DATA COLLECTION.

All your frames data have to be removed from hard disk within 10 days. All files older than 10 days will be removed without any notice.