Powder X-ray Diffraction Patterns

using

Bruker D8 Adv GADDS instrument.

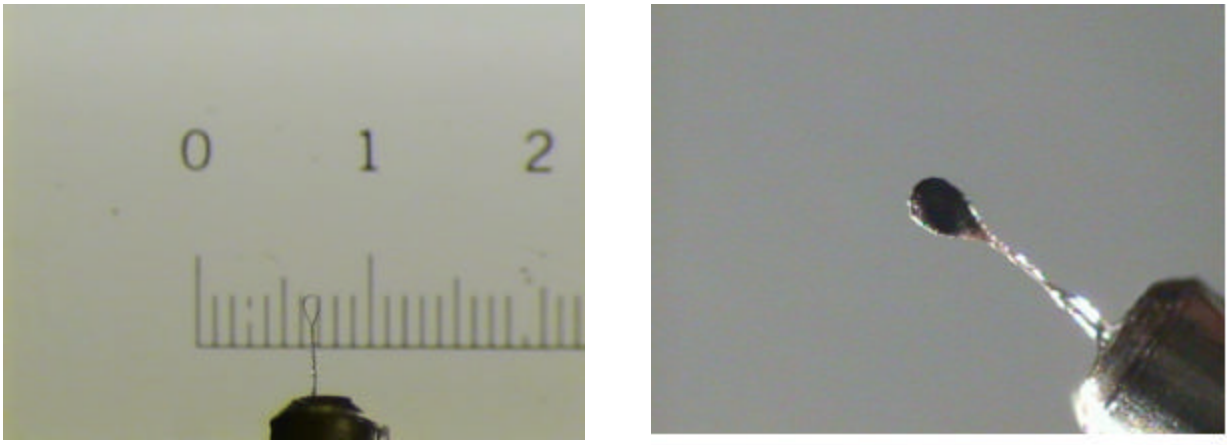
A Tutorial for Beginners

I. Mounting the Sample on the Loop:

Following procedures can be used for sample preparation:

1. The loop is "wetted" with mineral oil, and the excess oil is carefully removed leaving only a trace of oil on the loop itself. It is then carefully dragged over the sample under a microscope allowing the sample to collect in the area encircled by the loop. The sample should be gently rolled to form a near spherical mass. A fully loaded 10 μm loop (0.1mm diameter) would contain about a microgram of the material (depending on the density of the sample and packing).

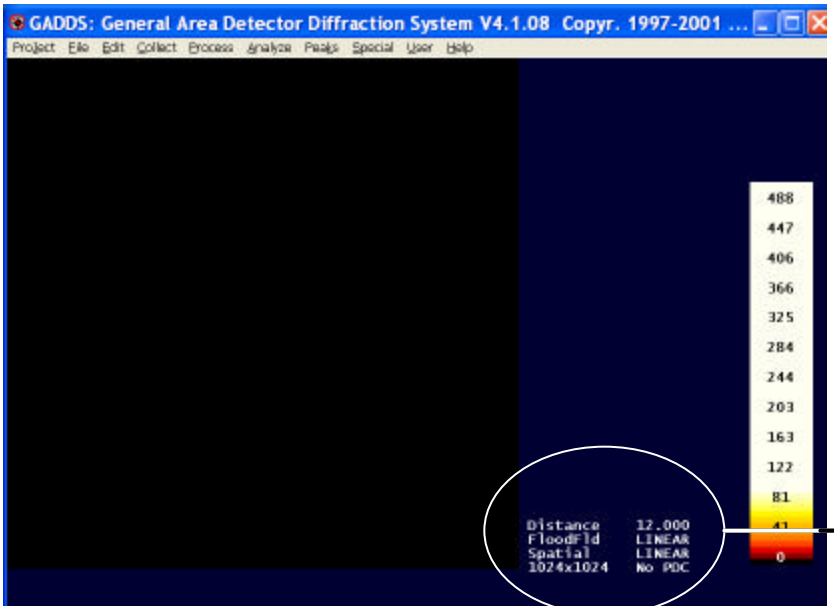
2. Alternately, very small amount (5 to 10 micrograms) of the sample should be made into a paste using mineral oil under the microscope, and the sample is scooped using the loop.



Enlarged images of (a) empty loop (0.1 mm diameter), and (b) loop (0.1 mm diameter) filled with silicon powder prepared by the method 2 above.

II. Opening GADDS and Creating a New Project

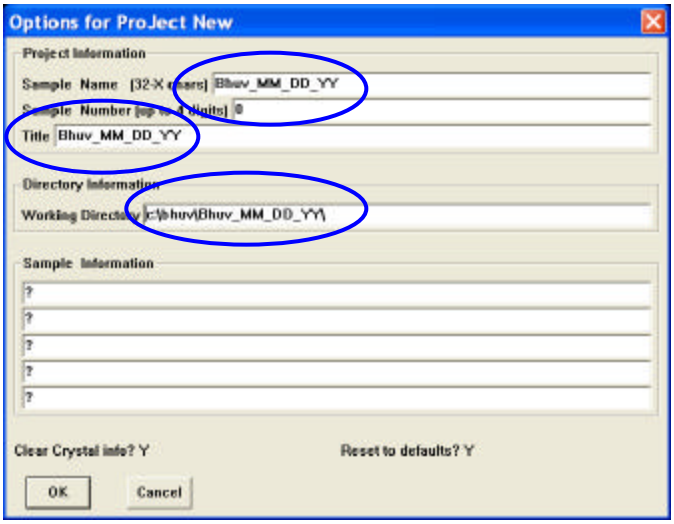
1. Double Click on the "GADDS" icon from the Desktop to start the program. After you answer few questions you will see a screen as below.



Check Distance FloodFld, Spatial, and the Resolution, after opening a new project.

Refer next page for details

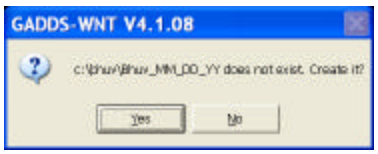
2. Project ⟶ New, will give you the following screen



- Fill in the Sample Name (eg., Bhuv_MM_DD_YY), Title (eg., Bhuv_MM_DD_YY), and working directory (e.g., C:\Bhuv\Powder\Bhuv_MM_DD_YY\).

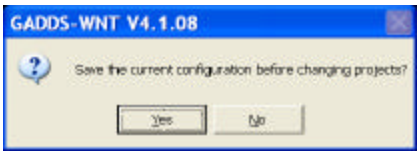
- Click OK

The program will ask



Say, Yes, if it is a new directory

To the screen,



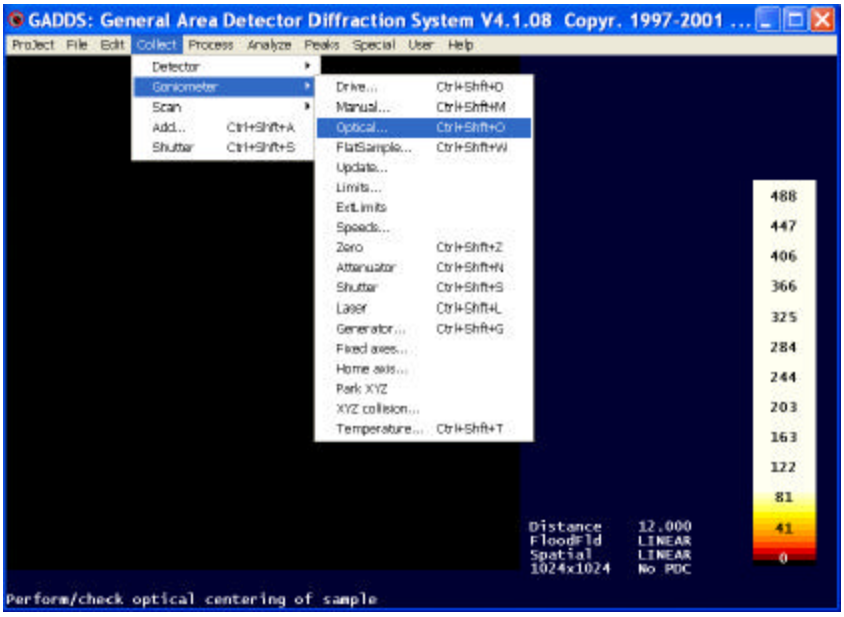
Click on Yes

Check: FloodFld and Spatial **SHOULD <u>NOT</u>** be linear. If they are linear, load the right FloodFld and Spatial files (If you do not know anything about FloodFld and Spatial, ask an expert to avoid problems with your data, and eventually repeating the data collection after!!). Also, verify the Detector Distance (in the instrument) and see to that the FloodFld and Spatial files are agreeing to the proper distance.

III. Sample Mounting and Centering:

In the GADDS program,

1. Collect ⟶ Goniometer ⟶ Optical (Two theta = -30, Omega = -30)



2. Now open the **INSTRUMENT DOOR,**

SAFETY CHECK: EVERY TIME YOU OPEN THE INSTRUMENT DOOR

Check if the Shutter Lights (in the rear frame of the instrument) are in the Shutter Closed Position

3. Open the Video Program from the Desktop.

Open a New File icon [?] and Click on the GRAB icon [?]. Now you can see the sample in the screen, with a Cross Wire. If you don't, rotate the base of the sample slowly to see the sample on the screen.

4. In the Goniometer Manual Control Box

Shift ⟶ F2 ⟶ 2 ⟶ ENTER

This will drive the goniometer to the following position

2-Theta = -30
Omega = -30
Phi = 90?

You can see two screws (Ask any experts(!) to know which of the screw will do what if you have never used a Gonio-Head), adjust them to get the sample to the center of the cross wire. You can adjust the height only in this position.

(Note: By pressing the Enter Key again you can turn the Gonio-Head 180^{o})

5. Now in the Goniometer Manual Control Box

1 ⟶ Enter

This will drive the Goniometer to the following position

2-Theta = -30
Omega = -30
Phi = 0?

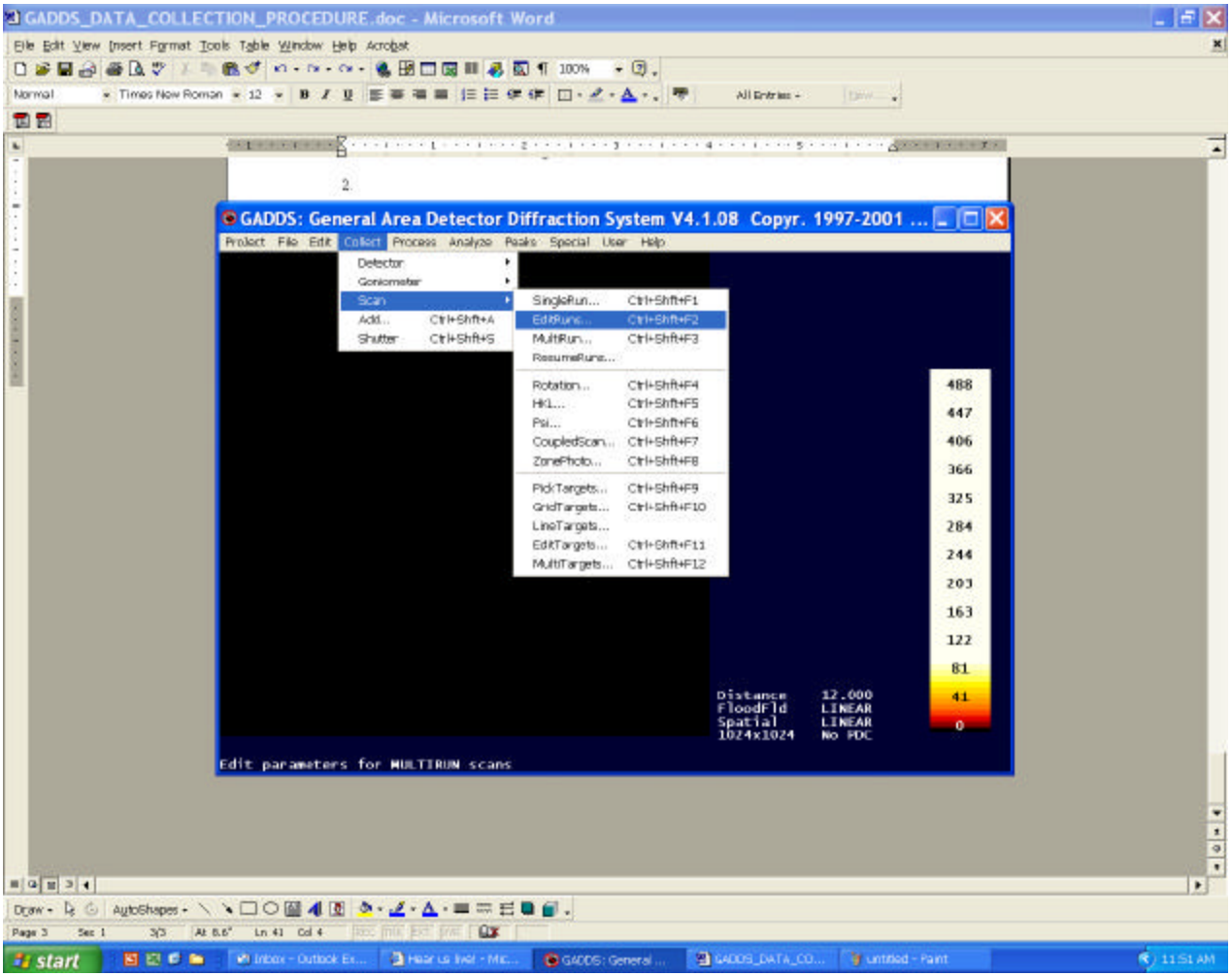
(Note: By pressing the Enter Key again you can turn the Gonio-Head 180^{o})

IV. Starting the Scan

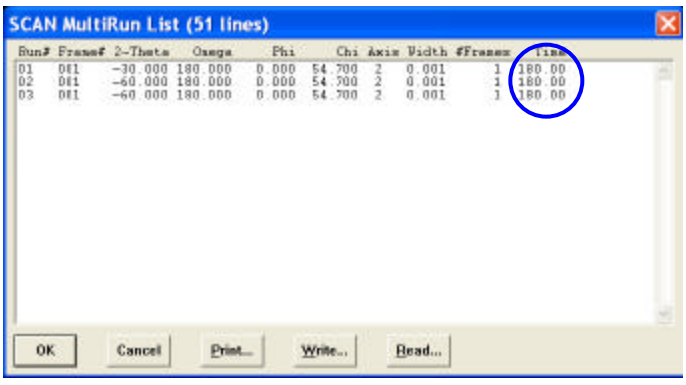
- After adjusting the sample to the center of the crosswire, go back to the GADDS program.

1. Press the Esc once or more to get out of the Manual Mode.

2. Collect ⟶ Scan ⟶ Edit Runs



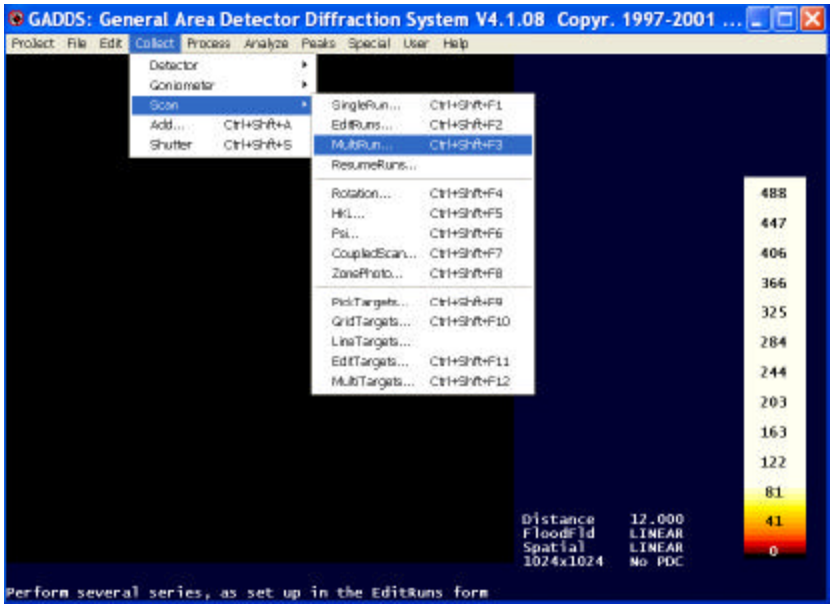
will give you this screen:



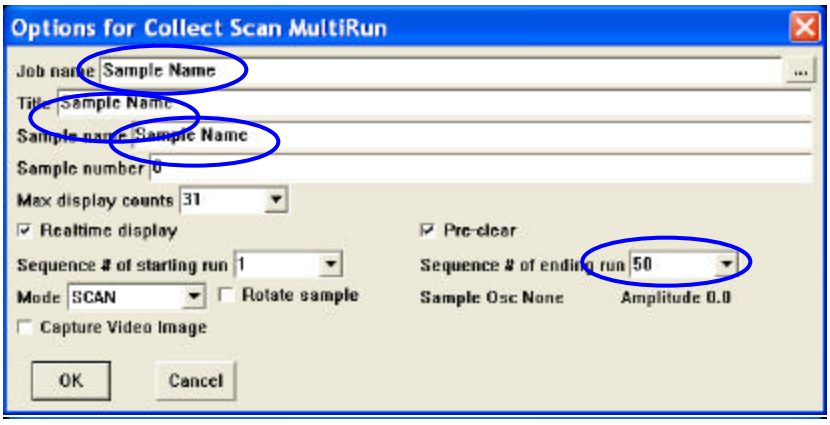
Note1: Set your Run, Frame#, 2-Theta etc, to the values given above to get a powder pattern from 5^{o} Two theta to 90^{o} Two Theta, which is normally sufficient for qualitative analysis.

Note 2: You can change the time from 180 (seconds) to any value between 1 (s) and 2100 (s) depending on the scattering nature of your sample.

3. Collect ⟶ Scan ⟶ Multirun



will give the screen:



You can change the sample names here to what you want to save the frames.
Click OK.

4.

You will start seeing powder rings, in a few seconds, if your sample is a good scatterer. You should see, at least, an image of the beam stop. If you do not, check the following:

- Your X-ray power must be at 40 kV and 40 mA
- Your shutter should be open (small black box in the rear panel frame of the instruement)
- Your sample centering
- If none of the above, catch an expert.

Wait till all the frames are collected.

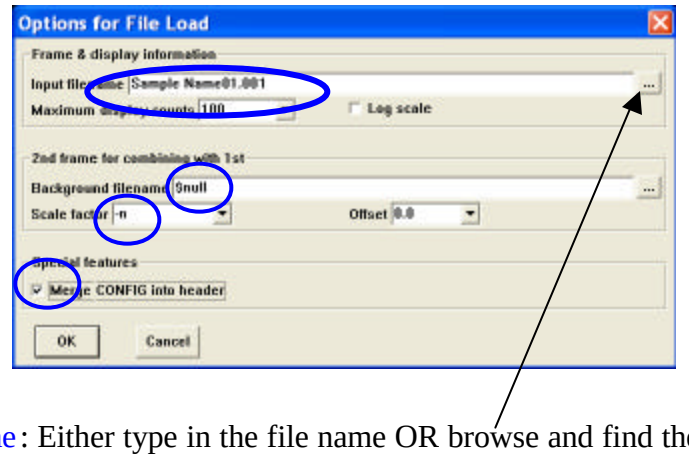
5. The resulting files will be

Sample Name01.001 and Sample Name01.p4p
Sample Name02.001 and Sample Name02.p4p
Sample Name03.001 and Sample Name03.p4p

V. Data Integration:

In the Gadds Program,

1. File ⟶ Load



-Input File Name: Either type in the file name OR browse and find the appropriate file.

-Background File name: $null. (If you are merging two files, give the name of the second file here).

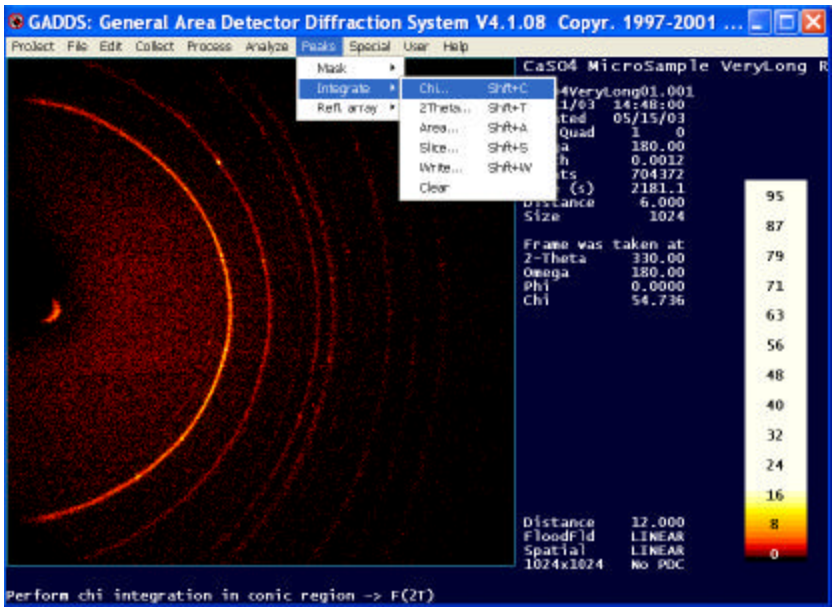
-Scale factor = -N. (If you are merging two files, +N.)

-Click on the Merge CONFIG into header

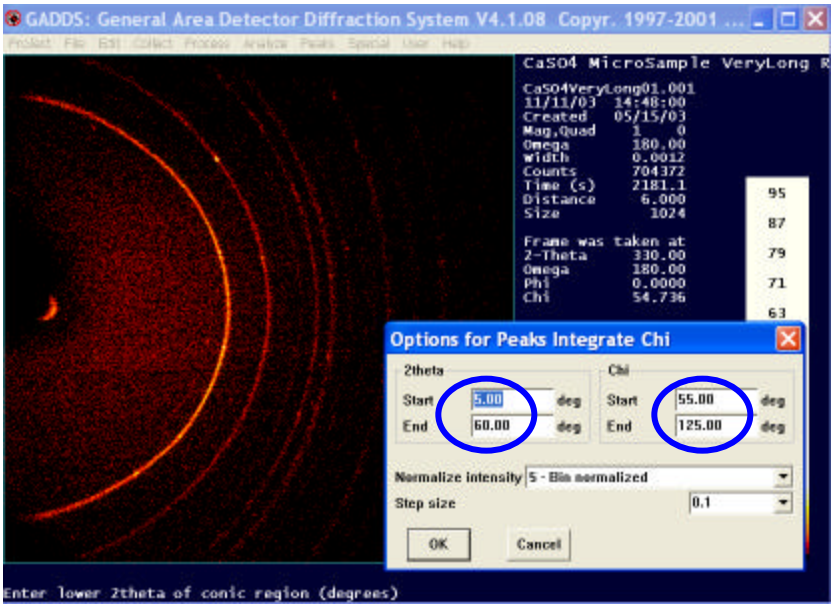
-OK

This will load the Frame with the “Powder Rings”

2. Peaks ⟶ Integrate ⟶ Chi

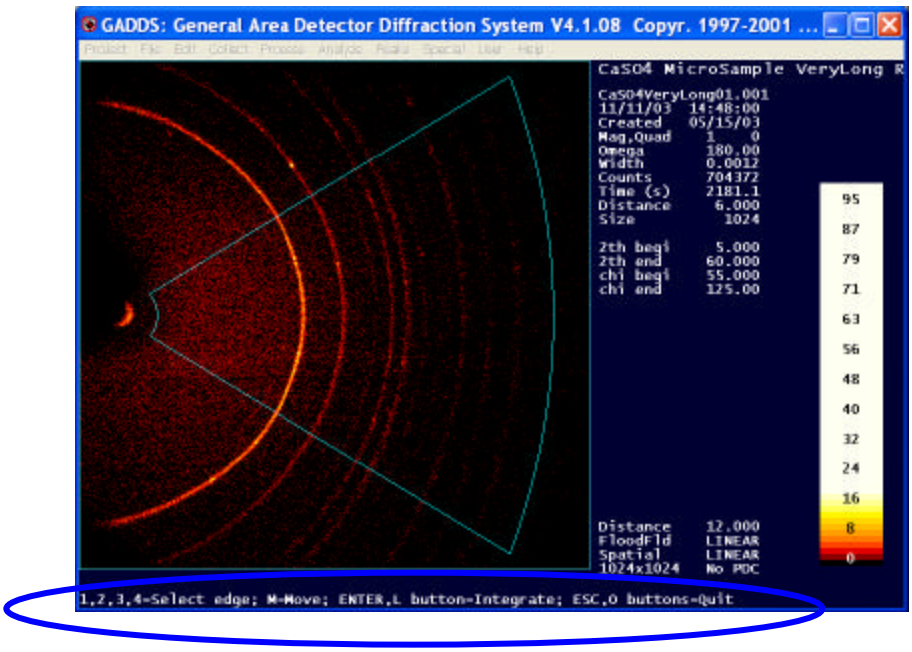


will give you the screen

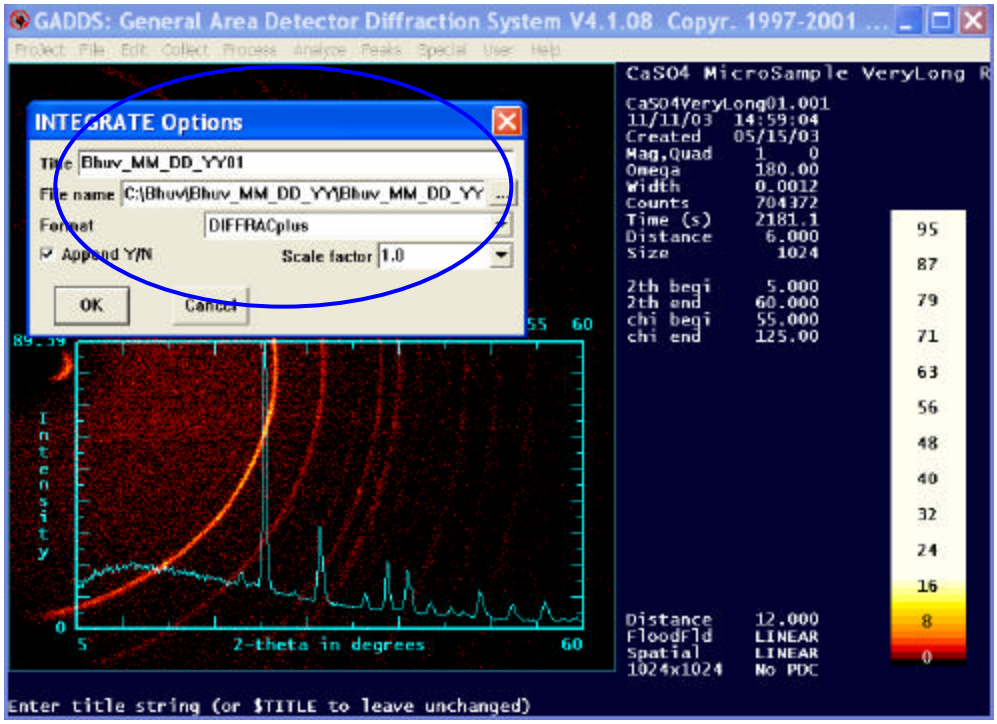


- Change 2-Theta Start and End, Chi Start and End to reasonable values

- Click OK



By selecting 1, 2, 3, or 4 you can move the 4 edges (respectively left, right, bottom and top) and click enter or left mouse button to Integrate.



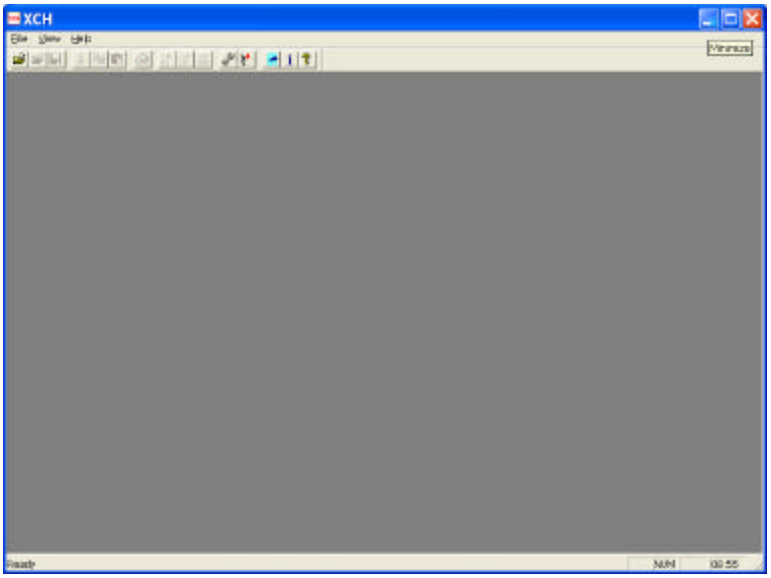
-Change the File Name, Title, if necessary (although, I would not recommend doing this).
-click OK

A file name Bhuv_MM_DD_YY01.Raw will be saved in the mentioned directory.

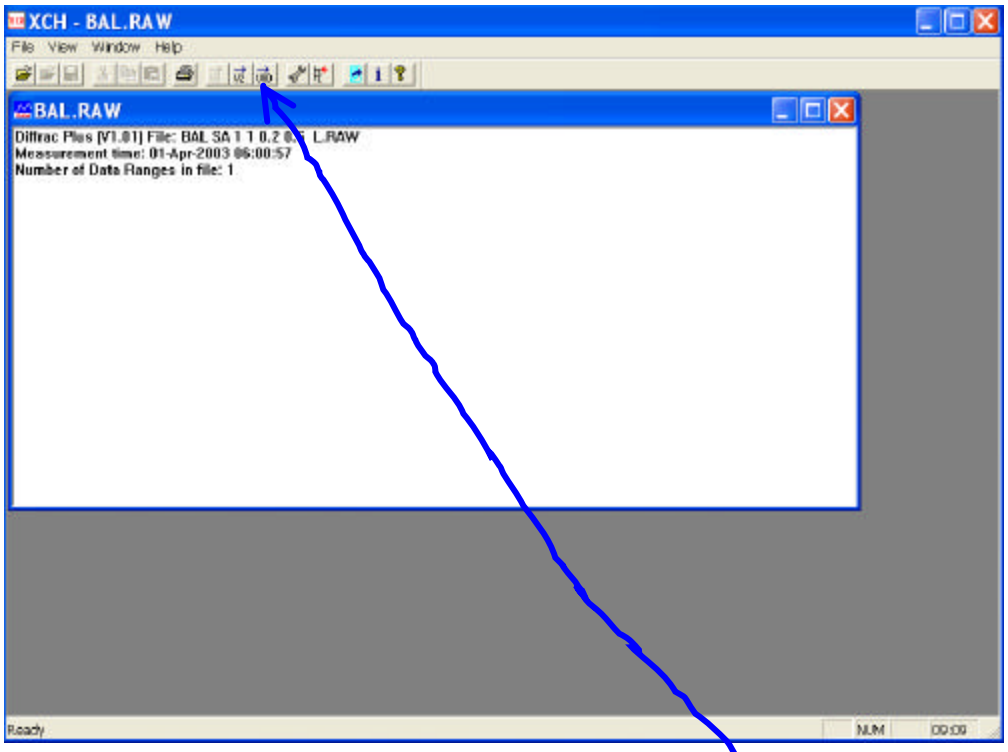
3. Eva or other softwares can be used to work on this powder diffraction file format.

VI. To convert .RAW file to an ASCII (.UXD) file.

Open the Diffrac Files Exchange program.



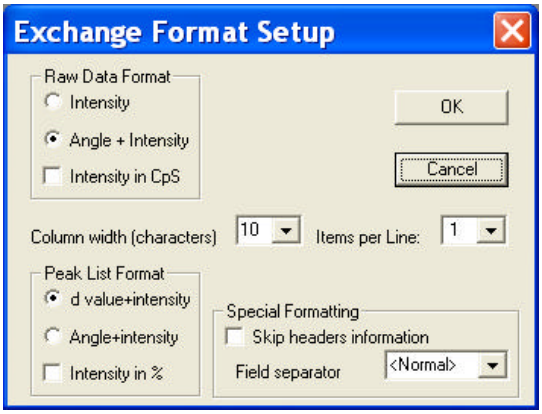
- Open your .RAW (say BAL.RAW) file



- Check the format of the output file:

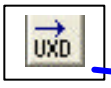
  File ⟶ UXD format

Check if all the values are as given in the following screen.



If the screen you get is not matching to the above, change it accordingly.

- Click OK.

- Now click the icon 

- Select the folder you want to save the ascii file (say, BAL.UXD), and save it.

The UXD file will contain several header lines which you may(!!!) or may not want followed by a two column data. The first column will correspond to Two-theta and the second the corresponding intensities.

Happy Powder Diffraction.

Nattamai Bhuvanesh
X-ray Diffraction Laboratory
Department of Chemistry
Texas A&M University
College Station, TX 77842-3012